Universidad de Córdoba

DEPARTAMENTO DE INGENIERIA RURAL. ÁREA INGENIERIA DE LA CONSTRUCCIÓN

P.D. Ingeniería agraria, alimentaria, forestal y de desarrollo rural sostenible

APLICACIÓN DE RESIDUOS, SUBPRODUCTOS INDUSTRIALES Y NANOMATERIALES PARA LA ESTABILIZACIÓN Y EJECUCIÓN DE CAPAS ESTRUCTURALES DE CARRETERAS

APPLICATION OF WASTE, INDUSTRIAL BY-PRODUCTS AND NANOMATERIALS FOR THE STABILISATION AND EXECUTION OF STRUCTURAL ROAD LAYERS

TESIS DOCTORAL

D. José Luis Díaz López

Para la obtención del

Título de Doctor con Mención Internacional por la Universidad de Córdoba

Director:
Dr. Francisco Agrela Sainz
Dra. Julia Rosales García
Córdoba, noviembre de 2023

TITULO: *Aplicación de residuos, subproductos industriales y nanomateriales para la estabilización y ejecución de capas estructurales de carreteras*

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TÍTULO DE LA TESIS: APLICACIÓN DE RESIDUOS, SUBPRODUCTOS INDUSTRIALES Y NANOMATERIALES PARA LA ESTABILIZACIÓN Y EJECUCIÓN DE CAPAS ESTRUCTURALES DE CARRETERAS

DOCTORANDO: José Luis Díaz López

INFORME RAZONADO DE LOS DIRECTORES DE LA TESIS

El Doctorando José Luis Díaz López ha desarrollado una investigación innovadora y ordenada, centrada en la aplicación de residuos, subproductos y nanomateriales en la estabilización de suelos, y su uso en capas estructurales de carreteras. Ha desarrollado capacidades avanzadas sobre la aplicación de estos materiales sostenibles, y a su vez, ha aprendido técnicas de investigación avanzadas para su uso en esta y otras líneas afines de investigación.

Durante la etapa pre-doctoral, el Doctorando ha cubierto todos los objetivos planteados para esta Tesis Doctoral y se ha ajustado al calendario programado, tanto para desarrollar la línea de investigación propuesta, como para el desarrollo de las actividades de formación complementarias.

La investigación realizada, le ha llevado a obtener un conocimiento técnico preciso de la viabilidad de aplicación de subproductos y materiales reciclados para la ejecución de capas de carreteras. Considerando la necesidad de reutilización de residuos y subproductos para avanzar hacia una economía circular, así como, ha logrado avanzar en el conocimiento de las propiedades técnicas de nuevas infraestructuras de comunicación.

En una primera fase, el Doctorando adquirió un amplio conocimiento de las propiedades físicas, químicas y mineralógicas de los diferentes subproductos y materiales objeto de esta investigación, como son, áridos reciclados mixtos, procedentes de residuos d construcción y demolición, escorias blancas de acería, cenizas de fondo y volantes de biomasa y nanomateriales, así como, la evaluación de la influencia del procesamiento en las plantas de tratamiento de residuos en las características finales de subproductos tales como los áridos reciclados.

Continuando en una segunda fase, en la que este conocimiento previo adquirido, le permitió evaluar la influencia de la aplicación de estos subproductos como materiales estabilizadores de suelos arcillosos expansivos, para la ejecución de subbases de carreteras.

La tercera fase se centró en una parte muy relevante, ya que el Doctorando pudo culminar sus investigaciones con la ejecución a escala real de un tramo de carretera de baja intensidad, mediante la aplicación de nanomateriales en el que se obtuvieron resultados técnicos muy positivos.

La cuarta fase la realizó de manera transversal, y estuvo centrada en la evaluación del impacto ambiental que puede causar la aplicación de subproductos en capas de carreteras, mediante el análisis de lixiviados por diferentes técnicas y el análisis de ciclo de vida.

Durante el periodo pre-doctoral, el Doctorando ha llevado a cabo de forma eficiente y entusiasta las tareas derivadas del plan de investigación de su Tesis Doctoral, además de haber colaborado de forma activa en diversas investigaciones desarrolladas por el Grupo de Investigación. Así mismo, el Doctorando ha asistido a seminarios, conferencias, visitas técnicas y cursos que le han sido de gran utilidad en el proyecto de su Tesis Doctoral. Y ha desarrollado parte de su investigación en un prestigioso centro internacional como es el Instituto Técnico Superior de Lisboa, consiguiendo de esta forma optar al título de Tesis Doctoral con mención internacional.

La labor realizada por el Doctorando se ha desarrollado de forma coherente a lo largo del transcurso de la misma, que ha culminado con la publicación de TRES artículos en revistas indexadas ISI-JCR y UNO en revisión, tres de ellas catalogadas en primer cuartil Q1.

Artículos indexados presentados por el Doctorando:

1. Agrela, F., **Díaz-López, J. L.**, Rosales, J., Cuenca-Moyano, G. M., Cano, H., & Cabrera, M. (2021). *Environmental assessment, mechanical behavior and new leaching impact proposal of mixed recycled aggregates to be used in road construction.* Journal of Cleaner Production, 280, 124362.
2. **Díaz-López, J. L.**, Cabrera, M., Marcobal, J. R., Agrela, F., & Rosales, J. (2021*). Feasibility of using nanosilanes in a new hybrid stabilised soil solution in rural and low-volume roads*. Applied sciences, 11(21), 9780.
3. **Díaz-López, J. L.**, Cabrera, M., Agrela, F., & Julia, R. (2023). *Geotechnical and Engineering Properties of Expansive Clayey Soil*

*Stabilised with Biomass Ash and Nanomaterials for its Application in Structural Road Layer*s. Geomechanics for Energy and the Environment, 36 (100496).

4. **Díaz-López, J.L.**, Rosales, J., Agrela, F., Cabrera, M., Cuenca-Moyano, G.M. (2023). *Evaluation of geotechnical, mineralogical and environmental properties of clayey soil stabilized with different industrial by-products: a comparative study*. Construction and Building Materials; Under Review

Adicionalmente, el Doctorando ha colaborado activamente en investigaciones del Grupo de Investigación que han conllevado a la-co-autoría de CUATRO artículos indexados IS-JCR, UN capítulo de libro editado por Elsevier, UN artículo de divulgación y la participación en OCHO congresos nacionales e internacionales.

Como conclusión, los directores consideran que el Doctorando ha realizado una investigación muy relevante y de actualidad, ya que la ejecución de carreteras requiere cada día más de la aplicación de materiales sostenibles y nuevos materiales, que reduzcan la huella de carbono.

Por todo ello, se autoriza la presentación de la Tesis Doctoral "**Aplicación de residuos, subproductos industriales y nanomateriales para la estabilización y ejecución de capas estructurales de carreteras**".

Córdoba, a 20 de noviembre de 2023

Firma de los directores:

Fdo.: Dr. Francisco Agrela Sainz

Fdo.: Dra. Julia Rosales García

Agradecimientos

"La paciencia es la madre de la ciencia" se acostumbra a decir. En mi caso personal, la paciencia nunca fue una virtud que destacara en mí, pues ya desde pequeño, estando en el colegio, siempre he vivido impacientado por terminar esa etapa, pensando en el siguiente paso. Llegado este momento, con estas primeras líneas, las cuales han sido las últimas que he escrito de mi Tesis Doctoral, es el momento de parar, disfrutar de lo conseguido y pensar en todas las personas que han hecho posible este momento. Por vuestro apoyo, quisiera daros mi más sincero agradecimiento.

En primer lugar, a mi director de Tesis, el Catedrático de Universidad Francisco Agrela Sainz, por darme la oportunidad, la confianza y las palabras adecuadas en los momentos complicados. Se que soy un poco cabezota en algunos momentos, por eso, gracias por haberme guiado hasta este momento.

A mi co-directora, la Dra. Julia Rosales García, por su incansable apoyo en todo momento, ejerciendo de "jefa" cuando había que poner orden en el despacho y laboratorio, directora, guiándome como investigador y amiga, cuidándome cuando lo necesitaba. Eres un ejemplo para mí. Por mostrarme el camino paso a paso hasta aquí.

A mi compañero, mentor y amigo, el Dr. Manuel Cabrera Montenegro, por haberme ensañado tantísimo, por todo su apoyo y cariño y todas las horas que hemos pasado juntos, haciendo este viaje más ameno. Me has ensañado a no rendirme en los malos momentos.

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A todos los Profesores y Profesoras y compañeros y compañeras del Área de Ingeniería de la Construcción de la Universidad de Córdoba, por haberme prestado los medios materiales y humanos necesarios para la realización de esta Tesis Doctoral.

A el Dr. José Ramón Marcobal, por su estrecha colaboración con nuestro equipo y compartir su conocimiento conmigo.

Al Centro de Estudios y Experimentación de Obras Públicas (CEDEX), en especial a José Estarie, María Santana y Rubén Ruiz, por abrirme las puertas de vuestro centro y ayudarme a mejorar como profesional.

Al Instituto Superior Técnico de la Universidad de Lisboa, en especial al Prof. Dr. Jorge de Brito y al Dr. Miguel Bravo. Por acogerme de una manera tan cálida, y acogerme en vuestro laboratorio dándome la oportunidad de internacionalizar mi trabajo. Por todo ello, muito obrigado.

A mis amigos, los cuales no son pocos para ponerlos a todos aquí, uno a uno (cosa que me hace sentir afortunado), pero ellos saben que después de ver cómo nos hacemos mayores y mil momentos y experiencias vividas, compartiendo momentos muy buenos y algunos muy malos, la vida no tendría el mismo color sin ellos. Con vosotros, la vida es "Un Paseo".

A Toñi y Juan Antonio, por vuestro apoyo y vuestro cariño.

A mis titas, titos y primos, también somos muchos por suerte, por acompañarme mientras persigo mis metas.

A mi abuelo Antonio y abuela Inés, seguro que estáis juntos sonriendo, allá donde estéis.

A mi abuela Mari Carmen, Mami, y a mi abuelo José Luis, Papa. Porque en la vida, nadie nos regala nada.

A mi familia, Miguel (Papá), Mari Carmen (Mamá), Tato y Keko. Por haberme enseñado a ser quien soy, dármelo todo y estar siempre a mi lado. Somos todo lo que una familia puede ser. Mis logros son los vuestros.

Y a mi compañera de vida, Lola, mi Mivi. Por acompañarme en este viaje y en todos. Por superar todo lo que nos propongamos juntos. Porque hogar, es donde estemos juntos.

José Luis Díaz López

Córdoba, 2023

Abstract

In recent decades, economic development has brought about an increase in waste generation, as well as a rise in harmful gas emissions and environmental impacts.

Currently, Sustainable Development stands as the primary objective of an advanced society, aiming to reconcile nature preservation with sustained economic growth within the framework of future circular economy models.

In this social, economic, and environmental context, the present Doctoral Thesis emerges with the purpose of evaluating, from both technical and environmental perspectives, the application of recycled materials, waste, by-products, and state-of-the-art nanomaterials for road layers construction. The goal is to reduce the use of conventional construction materials with a high carbon footprint, decrease the need for borrow earth in road construction, and potentially eliminate the transport of soils to landfills in roadworks, all while improving their technical characteristics. This research focuses on the application of mixed recycled aggregates (MRA), biomass bottom ash (BBA), biomass fly ash (BFA), steel slag (SS), and various cutting-edge silica-based nanomaterials.

The research, in its initial phase, begins with the study of the physical, chemical, and mineralogical properties of the aforementioned materials. The objective is to expand the current understanding of these materials and establish the most suitable applications for each material in the various layers that compose a road.

Following the material characterization, the second phase addresses the study of the application of MRA as a material for granular base in roads. This analysis allows evaluating the environmental effects of the selective collection of construction and demolition waste (CDW), facilitating the definition of new restrictive limits for its application and expanding its range of use.

The third phase focuses on the feasibility of using waste, by-products, and nanomaterials as road soil stabilizers. An optimization dosage study demonstrates how the addition of these materials enhances soil properties. Additionally, an innovative study combines nanomaterials with industrial by-products.

The fourth phase involves the construction of a real-scale low-volume road section, applying the knowledge gained during the development of the Doctoral Thesis. This study allows analyzing the evolution over time of the effect of adding these new materials under real conditions.

Finally, the entire study is complemented with a comprehensive life cycle analysis, quantifying the environmental improvements derived from the application of these new materials compared to conventional road construction materials.

The presented methodology and the results obtained during the completion of the Doctoral Thesis demonstrate the feasibility of using new sustainable materials as an alternative for sustainable road construction. This is analyzed from a scientific and rigorous perspective, in harmony with the principles of Sustainable Development.

Resumen

En las últimas décadas, el desarrollo económico ha llevado consigo un aumento en la generación de residuos, así como un incremento en las emisiones de gases nocivos afectando al medio ambiente.

En la actualidad, el Desarrollo Sostenible se establece como el principal objetivo de una sociedad avanzada, donde se busca conciliar el respeto a la naturaleza con un crecimiento económico sostenido, enmarcado en futuros modelos de economía circular.

En este contexto social, económico y ambiental, surge la presente Tesis Doctoral, cuyo propósito es evaluar, desde perspectivas tanto técnica como ambiental, la aplicación de materiales reciclados, residuos, subproductos y nanomateriales de última generación, para la construcción de capas de carreteras. El objetivo es reducir el uso de materiales de construcción convencionales, con una elevada huella de carbono, disminuir la necesidad de tierras de préstamo en la construcción de carreteras, eliminar potencialmente el transporte de suelos a vertederos en obras viales y reutilizar residuos depositados hasta la fecha en vertederos, a la vez que se mejoran sus características técnicas de las carreteras. Esta investigación se centra en la aplicación de áridos reciclados mixtos (ARM), cenizas de fondo de biomasa (CFB), cenizas volantes de biomasa (CVB), escorias blancas de acería (EBA), y varios nanomateriales basados en sílice de última generación.

El trabajo de investigación, en su primera fase, inicia con el estudio de las propiedades físicas, químicas y mineralógicas de los materiales mencionados anteriormente. El objetivo es ampliar el conocimiento actual sobre los mismos y establecer las aplicaciones más adecuadas para cada material en las distintas capas que componen una carretera.

Después de la caracterización de los materiales, la segunda fase aborda el estudio de la aplicación de ARM como material granular para base de carreteras. Este análisis permite evaluar los efectos ambientales de la recogida selectiva de los residuos de construcción y demolición (RCD),

facilitando la definición de nuevos límites restrictivos para su aplicación y ampliando su rango de utilización.

La tercera fase se enfoca en la viabilidad de utilizar residuos, subproductos industriales y nanomateriales como estabilizadores de suelos de carretera. Un estudio de optimización de dosificaciones demuestra cómo la adición de estos materiales mejora las propiedades de los suelos. Además, se realiza un innovador estudio de combinación de nanomateriales con subproductos industriales.

La cuarta fase implica la construcción de un tramo a escala real de carretera de baja intensidad, aplicando el conocimiento adquirido durante el desarrollo de la Tesis Doctoral. Este estudio permite analizar la evolución a lo largo del tiempo del efecto de la adición de estos nuevos materiales en condiciones reales.

Finalmente, todo el estudio se complementa con un análisis de ciclo de vida integral, cuantificando las mejoras ambientales derivadas de la aplicación de estos nuevos materiales en comparación con los materiales de construcción convencionales de carreteras.

La metodología presentada y los resultados obtenidos durante la realización de la Tesis Doctoral demuestran la viabilidad del uso de nuevos materiales a partir de subproductos como alternativa para la construcción sostenible de carreteras. Esto se analiza desde una perspectiva científica y rigurosa, en armonía con los principios del Desarrollo Sostenible.

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Abreviaturas

A	Acidification of Soil and Aater
ADe	Abiotic Depletion of Elements
ADf	Abiotic Depletion of Fossil Fuels
AG	Artificial Gravel
AM	Alternative Mixture
ARM	Árido Reciclado Mixto
AS	Alternative Section
BA	Biomass Ash
BBA	Biomass Bottom Ash
BFA	Biomass Fly Ash
CAH	Calcium Aluminate Hidrated
CASH	Calcium aluminosilicate hidrated
CB	Cenizas de Biomasas
CBR	California Bearing Ratio
CDW	Construction and Demolition Waste
CEM	Cement
CFB	Cenizas de Fondo de Biomasa
CS	Calcium Silicate
CSH	Calcium Silicate Hidrated
CVB	Cenizas Volantes de Biomasa
DMR	DIRECTIVA 2008/98/CE
CS	Clay soil
E	Eutrophication

EEC	European Economic Community
EPA	Environmental Protection Agency
FDW	Falling Weight Deflectometer
FU	Functional Unit
GW	Global Warming
IBP	Industrial By-products
LCA	Life cycle Assessment
LL	Liquid Limit
MDD	Maximum Dry Density
MRA	Mixed Recycled Aggregates
N	Nanomaterial
NA	Natural Aggregates
ODP	Ozone Layer Depletion
OMC	Optimum Moisture Content
OPC	Ordinary Portland Cement
PG-3	Pliego de Prescripciones Técnicas Generales para Obras de Carreteras y Puentes
PI	Plastic Index
PL	Plastic Limit
pMRA	powdered Mixed Recycled Aggregates
POF	Photochemical Oxidation
QL	Quicklime
RA	Recycled Aggregates
RAA	Recycled Asphalt Aggregates
RCA	Recycled Concrete Aggregates
RCD	Residuos de Construcción y Demolición
SEM	Scanning Electron Microscopy

S-EST1	Suelo Seleccionado tipo 1
S-EST2	Suelo Seleccionado tipo 2
Ss	Sandy Soil
SS	Steel Slag
SSBA	Sodium Silicate Based Admixture
SSD	Saturated Surface Dry
TGA	Thermogravimetric analysis
UCS	Unconfined Compressive Strength
UE	Unión Europea
WSS	Water Soluble Sulfate
XRD	X-ray diffraction
XRF	X-Ray Fluorescence

COMPENDIO DE PUBLICACIONES

La presente Tesis Doctoral se ha elaborado como un "compendio de publicaciones", conforme a las directrices establecidas en el artículo 53 del Reglamento 57/2020 que regula los Estudios de Doctorado de la Universidad de Córdoba. El enfoque de esta tesis se centra en la aplicación de residuos, subproductos industriales y nanomateriales para la ejecución y estabilización de capas estructurales de carreteras. Además, se cumplen las condiciones necesarias para que la tesis doctoral pueda optar al título de Doctor con mención internacional, según lo estipulado en el artículo 54 del Reglamento 57/2020.

En este apartado, se presentan las contribuciones científicas derivadas directamente del desarrollo de la tesis doctoral.

- **PUBLICACIÓN 1.**

ENVIRONMENTAL ASSESSMENT, MECHANICAL BEHAVIOR AND NEW LEACHING IMPACT PROPOSAL OF MIXED RECYCLED AGGREGATES TO BE USED IN ROAD CONSTRUCTION.

Authors: Agrela, F., **Díaz-López, J.L.,** Rosales, J., Cuenca-Moyano, G.M., Cano, H., Cabrera, M.

Reference: Journal of Cleaner Production. Vol.: 280. Pag.: 124362. SEPT 2020

Quality indicators: *Impact Factor: 11,016. Q1*

DOI: https://doi.org/10.1016/j.jclepro.2020.124362

Abstract:

Several types of mixed recycled aggregates (MRA) from construction and demolition waste (C&DW) treatment plants in Cordoba and Malaga, Spain, and a sample of natural aggregate (NA) were studied toevaluate the viability of their use in the construction of road layers. The physicochemical properties, mechanical behavior and environmental impact of all samples were determined. The life cycle analysis of road sections manufactured with the materials studied was also determined. All samples of MRA showed a mechanical behavior suitable for use in the formation of road layers. In addition, it was determined that these materials, when they come from C&DW with selective collection at origin, cause less environmental impact than the impact caused by the use of natural aggregates in the formation of road layers.

- **PUBLICACIÓN 2.**

FEASIBILITY OF USING NANOSILANES IN A NEW HYBRID STABILISED SOIL SOLUTION IN RURAL AND LOW-VOLUME ROADS.

Authors: Díaz-López, J.L., Cabrera, M., Marcobal, J.R., Agrela, F., Rosales, J.

Reference: Applied Sciences, Vol.: 11;Pag.: 9780; OCT 2021

Quality indicators: *Impact Factor: 2,921. Q3.*

DOI: https://doi.org/10.3390/app11219780

Abstract:

The application of new materials for soil stabilisation is a growing field of study in recent years. In this work, the effect of two types of silica-based nanomaterials combined with binders (quicklime and cement) are studied to stabilise soils and form structural layers for rural and low volume roads. The physical and chemical properties of the materials have been determined, as well as the mechanical behaviour of the stabilised soil. Three hybrid stabilised soil sections have been designed using a multilayer elastic model, executed at full scale and measuring the evolution of their properties in the medium to short term. The results show that the application of silica-based nanomaterials and two types of binders on the tread layers provide high structural stability and good behaviour of the sections.

- **PUBLICACIÓN 3.**

GEOTECHNICAL AND ENGINEERING PROPERTIES OF EXPANSIVE CLAYEY SOIL STABILIZED WITH BIOMASS ASH AND NANOMATERIALS FOR ITS APPLICATION IN STRUCTURAL ROAD LAYERS.

Authors: Díaz-López, J.L., Cabrera, M., Agrela, F., Rosales, J.

Reference: Geomechanics for Energy and the Environment; Vol.: 36; Pag.: 100496; DEC 2023

Quality indicators: *Impact Factor: 5,000. Q1.*

DOI: https://doi.org/10.1016/j.gete.2023.100496

Abstract:

Clayey soils often pose issues such as swelling, high plasticity, low permeability, and low bearing capacity, particularly in regions with seasonal rainfall. Consequently, soil improvement techniques are necessary for constructing road base layers. Currently, the most used method for road stabilization involves the application of traditional binders such as lime and cement. However, the production of cement and lime has negative environmental impacts and depletes natural resources. In this study, new stabilizing materials based on waste and by-products, as well as a new generation of nanomaterials, are investigated for stabilizing highly plastic and expansive soils. The geotechnical and mechanical properties of soils stabilized with waste materials like biomass ashes from electricity generation, along with small amounts of a silica-based nanotechnological stabilizer, are examined. The obtained results suggest the potential reduction in the use of traditional binders by incorporating by-products, while still maintaining soil properties, and even improving the properties through the application of nano-sized additives.

- **PUBLICACIÓN 4.**

EVALUATION OF GEOTECHNICAL, MINERALOGICAL AND ENVIRONMENTAL PROPERTIES OF CLAYEY SOIL STABILIZED WITH DIFFERENT INDUSTRIAL BY-PRODUCTS: A COMPARATIVE STUDY.

Authors: Díaz-López, J.L., Rosales, J., Agrela, F., Cabrera, M., Cuenca-Moyano, G.M.
Reference: Construction and Building Materials; Under Review
Quality indicators: *Impact Factor: 7,400. Q1*
DOI: Under Review
Abstract:

The utilisation of soil stabilization techniques employing binders like lime or cement enables the use of soils that are classified as disposable, thereby reducing the need for landfills and the consumption of natural resources. However, the production of lime and cement results in significant CO2 emissions. Therefore, the application of industrial by-products (IBP) for stabilizing expansive soils presents an opportunity to minimise the use of traditional binders. This study investigates the geotechnical, mechanical, mineralogical, and environmental properties of four IBP (biomass bottom ash, biomass fly ash, steel slag, and mixed recycled aggregate) combined with a silica-based nanomaterial for road layer applications. The technical feasibility of using IBP is demonstrated, and an environmental assessment through leachate analysis provides insights into their suitability for the intended purpose. Additionally, a life cycle analysis study demonstrates the environmental benefits, including reduced CO_2 emissions and other parameters, resulting from the utilisation of these materials, thereby promoting more sustainable economic models.

Otras publicaciones en revistas científicas indexadas derivadas de la Tesis Doctoral:

USE OF NANOMATERIALS IN THE STABILIZATION OF EXPANSIVE SOILS INTO A ROAD REAL-SCALE APPLICATION.

Authors: *Rosales, J., Agrela, F., Marcobal, J.R.,* ***Díaz-López, J.L.,*** *Cuenca-Moyano, G.M., Caballero, A., Cabrera, M.*

Reference: *Materials*, Vol.: 13; Pag.: 3058; JUL 2020

Quality indicators: *Impact Factor: 3,92. Q2*

DOI: *https://doi.org/*10.3390/ma13143058

Abstract:

Stabilization is a traditional strategy used to improve soils with the main objective of ensuring that this base is compliant with the technical specifications required for the subsequent development of different infrastructures. This study proposes the use of commercial nanomaterials, based on a solution of silicates, to improve the technical characteristics and bearing capacity of the expansive soil. A physical–chemical property study was carried out on the additive nanomaterial. Subsequently, different mixtures of expansive soil, selected soil and artificial gravel with quicklime and commercial nanomaterials were developed to evaluate the improvement obtained by the use of nanomaterials in the technical characteristics of the soil. Compressive strength and the Californian Bearing Ratio index were considerably increased. A full-scale study was carried out in which the nanomaterial product was applied to two different sections of stabilized road compared to a control section. The results obtained showed that the use of nanomaterial led to the possibility of reducing the control section by 30 cm, thus achieving less use of quicklime and a mechanical means for preparing the road section. The use of commercial nanomaterial improved the behavior of the stabilized sub-base layer. Through life cycle assessment, this study has shown that the use of nanomaterials reduces the environmental impact associated with soil stabilization.

ECO-EFFICIENT CEMENT-BASED MATERIALS USING BIOMASS BOTTOM ASH: A REVIEW.

Authors: Cabrera, M., ***Díaz-López, J.L.,*** *Agrela, F., Rosales, J.*

Reference: *Applied Sciences*, Vol.: 10; Pag.: 8026; NOV 2020

Quality indicators: *Impact Factor: 2,921. Q3.*

DOI: *https://doi.org/10.3390/app10228026*

Abstract:

In recent years the use of biomass for electricity generation in thermal and cogeneration plants has increased worldwide because it is an environmentally clean fuel whose impact measured in greenhouse gas emissions is practically zero. However, biomass bottom ash, a waste produced during combustion, has also increased considerably, which has both a negative economic and environmental impact, due to landfill transport and management of this by-product. Although biomass bottom ash has potential characteristics for application in the manufacture of construction materials, its full-scale application is dicult because of the wide range in physicochemical properties, depending on the type of biomass burned, such as wood residue, olive waste, waste paper sludge, cocoa shell, etc., and the type of combustion process in the plant. This study reviews the influence on the physicochemical properties, mechanical behavior, and durability of different cement-based materials, such as mortars, concrete, and cement-treated granular material, manufactured from biomass bottom ash. The previous studies demonstrate the feasibility of substituting natural materials for biomass bottom ash in cement-based materials, presenting adequate mechanical behavior and durability properties to comply with the required technical specifications in different building materials.

ALKALI-ACTIVATED STAINLESS STEEL SLAG AS A CEMENTITIOUS MATERIAL IN THE MANUFACTURE OF SELF-COMPACTING CONCRETE.

Authors: Rosales, J., Agrela, F**., Díaz-López, J.L**., Cabrera, M.

Reference: *Materials*, Vol.: 14; Pag.: 3945; JUL 2021

Quality indicators: *Impact Factor: 3,92. Q1*

DOI: https://doi.org/10.3390/ma14143945

Abstract:

This work develops the manufacture of self-compacting concrete (SCC) with 50% cement reduction. As an alternative binder to cement, the viability of using an alkali-activated combination of stainless steel slag (SSS) and fly ash (FA) has been demonstrated. SSS was processed applying three different treatments. Binders were manufactured mixing 35% SSS with 65% FA, as precursors, and a hydroxide activating solution. This binder was replaced by the 50% cement for the manufacture of SCC. The results obtained show good mechanical properties and durability. The study shows a reduction in the use of cement in the manufacture of SCC reusing two wastes.

EFFECT OF PROCESSED VOLCANIC ASH AS ACTIVE MINERAL ADDITION FOR CEMENT MANUFACTURE.

Authors: *Rosales, J., Rosales, M.,* ***Díaz-López, J.L.,*** *Agrela, F.,* Cabrera, M.,

Reference: *Materials* , Vol.: 15; Pag.: 6035; SEPT 2022

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Abstract:

In the last quarter of 2021, there was a very significant eruption of the Cumbre Vieja volcano on the island of La Palma, belonging to the Canary Islands, Spain. It generated a large amount of pyroclastic volcanic materials, which must be studied for their possible applicability.

This work studies the properties and applicability of the lava and volcanic ash generated in this process. The need for reconstruction of the areas of the island that suffered from this environmental catastrophe is considered in this study from the point of view of the valuation of the waste generated. For this purpose, the possibility of using the fine fraction of ashes and lava as a supplementary cement material (SCM) in the manufacture of cement is investigated. The volcanic material showed a chemical composition and atomic structure suitable for replacing clinker in the manufacture of Portland cement. In this study, the cementing and pozzolanic reaction characteristics of unprocessed volcanic materials and those processed by crushing procedures are analysed. To evaluate the cementitious potential by analysing the mechanical behaviour, a comparison with other types of mineral additions (fly ash, silica fume, and limestone filler) commonly used in cement manufacture or previously studied was carried out. The results of this study show that volcanic materials are feasible to be used in the manufacture of cement, with up to a 22% increase in pozzolanicity from 28 to 90 days, showing the high potential as a long-term supplementary cementitious material in cement manufacturing, though it is necessary to carry out crushing processes that improve their pozzolanic behaviour.

Participación en un Capítulo de Libro editado por Elsevier:

CHAPTER 9. SPECIALIZED CONCRETE MADE OF PROCESSED BIOMASS ASH: LIGHTWEIGHT, SELF-COMPACTING, AND GEOPOLYMERIC CONCRETE.

Authors: Rosales, J., Cabrera, M., López-Alonso, M., **Díaz-López, J.L**., Agrela, F.
Book: The Structural Integrity of Recycled Aggregate Concrete Produced with Fillers and Pozzolans.
ISBN: 978-0-12-824105-6
DOI: *https://doi.org/10.1016/B978-0-12-824105-9.00001-9*

Otra difusión de los resultados obtenidos ha tenido lugar a través de diferentes congresos internacionales y nacionales, jornadas y seminarios como:

1. TECHNICAL AND ENVIROMENTAL PROPERTIES OF RECYCLED MIXED AND CONCRETE AGGREGATES ACCORDING A NEW CLASSIFICATION

Authors: Cabrera, M., Rosales, J., **Díaz-López, J.L**., Cano, H., Agrela, F.
Type of publication: Conference paper
Reference: International congress of construction and demolition waste recycling (C&D waste). Madrid. Madrid. Spain. 30 – 31 october 2019.

2. APLICACIÓN A ESCALA REAL DE CENIZAS DE FONDO DE BIOMASA Y NANOMATERIALES PARA LA ESTABILIZACIÓN DE SUELOS.

Authors: **Díaz-López, J.L.,** Rosales, J.
Type of publication: Conference paper
Reference: 1ST INTERNATIONAL SANTANDER ECO-CONCRETE CONFERENCE. Santander, Cantabria, España. 16 – 17 de diciembre de 2021

3. ACTUACIONES DE LA INGENIERÍA CIVIL EN EL MUNDO RURAL.

Authors: **Díaz-López, J.L**., Agrela, F., Cabrera, M., Rosales, M., Rosales, J.
Type of publication: Revista de divulgación
Reference: Cimbra: Revista del Colegio de Ingenieros Técnicos de Obras Públicas. 421, pp. 31 - 36. Colegio de Ingenieros Técnicos de Obras Públicas, 01/05/2022.

4. ANÁLISIS DE LAS PROPIEDADES GEOMECÁNICAS DE MARGAS GRISES EXPANSIVAS ESTABILIZADAS MEDIANTE CENIZAS DE BIOMASA Y NANOMATERIALES PARA SU USO EN LA AUTOVÍA A-32 POR SU PASO EN VILLACARRILLO (JAÉN).

Authors: **Díaz-López, J.L**., Cabrera, M., Agrela, F., Rosales, M. Rosales, J.
Type of publication: Conference paper
Reference: XI Simposio Nacional de ingeniería Geotécnica. Mieres, Asturias, España. 24 – 27 mayo de 2022.

5. PROPUESTA DE CLASIFICACIÓN DE ÁRIDOS RECICLADOS MIXTOS ACORDE CON SUS CARACTERÍSTICAS TÉCNICAS Y MEDIOAMBIENTAL.

Authors: Cabrera, M., **Díaz-López, J.L**., Agrela, F., Cano, H., Rosales, J.
Type of publication: Conference paper
Reference: VII Congreso internacional de estructuras de la Asociación Española de Ingeniería Estructural. Santander, Cantabria, España. 20 – 22 de junio de 2022.

6. VALORIZACIÓN DE RESIDUOS PROCEDENTES DE LA QUEMA DE BIOMASA DE OLIVO PARA CONSTRUCCIÓN DE CAPAS DE CARRETERAS SOSTENIBLES

Authors: **Díaz-López, J.L.**
Type of publication: Conference paper
Reference: XI Congreso Científico de Investigadores en Formación. Córdoba, Córdoba, España. 4 de mayo de 2023

7. PERFORMANCE OF SELF-COMPACTING CONCRETE FROM A NEW ECO-HYBRID ADDITION BASED ON MIXED RECYCLED AGGREGATES AND BIOMASS BOTTOM ASH.

Authors: **Díaz-López, J.L**., Agrela, F., Cabrera, M., Rosales, J
Type of publication: Conference paper
Reference: II INTERNATIONAL CÓRDOBA ECO-CONCRETE CONFERENCE. Córdoba, Spain. 6-7 July, 2023.

8. ECO-CONCRETE TOWARDS SUSTAINABLE CONSTRUCTION

Authors: Rosales, M., **Diaz-López, J.L**., Agrela, F., Rosales, J.
Type of publication: Conference paper
Reference: II INTERNATIONAL CÓRDOBA ECO-CONCRETE CONFERENCE. Córdoba, Spain. 6-7 July, 2023.

9. CONSTRUCCIÓN DE CARRETERAS CON ESCORIAS DE ACERO INOXIDABLE.

Authors: Rosales, J., Agrela, F., **Diaz-López, J.L**.
Type of publication: Conference paper
Reference: X JORNADAS CÁTEDRA ACERINOX. Algeciras, Spain. March 2023

CAPÍTULO I

ESTADO DEL ARTE

ESTADO DEL ARTE

El crecimiento económico de un país está intrínsecamente ligado a la extensión y calidad de la red de carreteras construida en su territorio. La conectividad intraurbana e interurbana permite enlazar el tejido productivo e industrial de un territorio, ejerciendo una influencia significativa en su desarrollo económico y social.

La construcción de vías de transporte terrestre y el consecuente crecimiento económico e industrial de las zonas conectadas, no obstante, están ineludiblemente vinculados al deterioro del medio ambiente, la emisión de gases nocivos a la atmósfera y la generación de grandes cantidades de residuos industriales, que a menudo son depositados en vertederos.

En este contexto, surge la presente Tesis Doctoral con el propósito de reducir la cantidad de residuos y subproductos industriales analizados (áridos reciclados mixtos, cenizas de fondo de biomasa, cenizas volantes de biomasa y escoria blanca de acería) dispuesta en vertedero. En su lugar, se busca promover su aplicación como material para la construcción de capas de carreteras y la estabilización de suelos geotécnicamente no utilizables, fomentando así un modelo de economía circular y sostenible.

Esta investigación comienza con un estudio sobre el conocimiento actual acerca de la aplicación de estos residuos y subproductos en diversas aplicaciones de la ingeniería civil (bases y subbases de carreteras, fabricación de hormigón, morteros de albañilería, materiales granulares tratados con cemento, etc.) y de los emergentes materiales de construcción de última generación conocidos como "nanomateriales", destinados a su aplicación en obras de carreteras.

La presente investigación muestra un estudio exhaustivo de las propiedades físicas, químicas y mineralógicas de los residuos, subproductos y nanomateriales que se aplicarán en la conformación de capas estructurales de carreteras. Además, amplía el conocimiento sobre la mejora de propiedades mecánicas y geotécnicas de las capas de carreteras desarrolladas mediante la aplicación conjunta de residuos y subproductos procesados, así como nanomateriales, aspecto que hasta ahora ha sido estudiado de manera minoritaria.

Para completar la investigación, se lleva a cabo un estudio de impacto ambiental derivado de los posibles lixiviados producidos por la aplicación de residuos, y se analizan las reducciones de impacto en el medio ambiente mediante un análisis de ciclo de vida.

1.1.- Carreteras y sostenibilidad

Las políticas internacionales actuales tienen como objetivo alcanzar una sociedad moderna, con modelos productivos basados en la innovación, que lleven a un crecimiento sostenible, integrado y continuo.

Para ello, el desarrollo de energías renovables, optimización de modelos energéticamente eficiente y de construcción sostenible es prioritario, estableciendo las líneas de acción, con la creación de nuevos diseños y materiales para la construcción y procesos sostenibles.

Para todo ello, las infraestructuras de movilidad, concretamente las carreteras, juegan un papel decisivo como catalizador de las relaciones económicas en cada área socioeconómica. [1].

La Unión Europea, cifra en 1,5 billones de euros las inversiones necesarias hasta 2030 para la construcción de un Red de Transporte Europea Básica, cuyo objetivo es la conexión de todos los nodos económicos europeos, para garantizar el desarrollo conjunto de la eurozona.

Fig. 1. 1. Red de Transporte Europea [1]

Sin embargo, la construcción y mantenimiento de carreteras, así como el aumento de actividad industrial asociado a la misma, generan una cantidad significativa de residuos que necesitan ser gestionados de manera eficiente y sostenible.

En este contexto nace la presente Tesis Doctoral, en la cual se analizan los efectos de la aplicación de diferentes residuos y subproductos industriales para la ejecución de distintas capas de carreteras, fomentando así un modelo productivo basado en la economía circular.

1.1.1.- Estructura de una carretera

Una carretera se compone de diversas capas superpuestas, cada una con propiedades específicas que contribuyen a la capacidad de carga y resistencia de la vía.

La estructura de una carretera típica consta de varias capas, incluyendo la subrasante, subbase, base y superficie de rodadura. La subrasante es la capa de suelo natural sobre la que se apoya toda la estructura. La subbase, situada encima de la subrasante, cumple la función de distribuir la carga y mejorar la capacidad de drenaje. Por encima de la subbase se encuentra la base, que proporciona estabilidad estructural y contribuye a la resistencia de la carretera. Finalmente, la superficie de rodadura es la capa superior que está en contacto directo con los vehículos y soporta las cargas dinámicas [2].

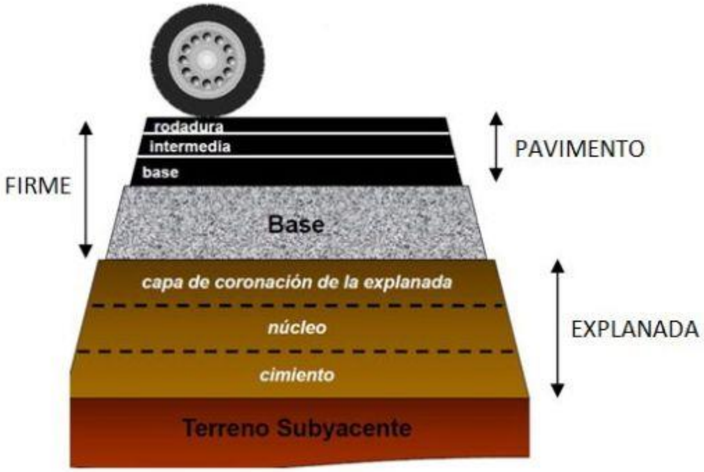


Fig. 1. 2. Sección de una carretera

En la construcción de carreteras, es común que se requieran movimientos de tierra para adecuar el terreno y garantizar la estabilidad y durabilidad de la vía. Estos movimientos pueden implicar la eliminación de suelos marginales y la incorporación de suelos de préstamo. Asimismo, se utilizan técnicas de estabilización de suelos para mejorar las propiedades de los suelos in situ, garantizando una subbase más resistente y duradera.

Las bases de carretera son construidas generalmente mediante la compactación de zahorra artificial. Asimismo, la ejecución de subbases de carreteras mediante la estabilización de suelos se realiza a través de la adición de cal y cemento a los suelos marginales para la mejora de propiedades geotécnicas y mecánicas [3].

La ejecución de bases de zahorra compactada y subbases de suelos estabilizados in situ con cal o cemento, implica un elevado deterioro ambiental, derivado de la extracción de materias primas para la producción de zahorra artificial y fabricación de cal y cemento [4].

Por este motivo, en la presente Tesis Doctoral, se va a analizar la viabilidad de construcción de bases de carretera con áridos reciclados, provenientes de residuos de construcción y demolición como sustituto de zahorra artificial y la ejecución de subbases de carreteras a través de la estabilización de suelos marginales aplicados residuos, subproductos industriales y nanomateriales, como material alternativo a la cal.

La aplicación de estos materiales reciclados supone la reducción de extracción de recursos naturales, de emisiones de gases de efecto invernadero a la atmosfera y el depósito en vertedero de los residuos generados en la industria, fomentando un modelo de economía circular.

En los siguientes apartados, se va a mostrar el estado actual de la técnica de ejecución de las capas de carreteras a analizar en la presente Tesis Doctoral, siendo, capas de zahora compactada y subbases de suelos estabilizados in-situ.

1.1.1.1.- Bases de carretera de zahorra compactada

Las bases de carreteras son una parte fundamental de la infraestructura vial, proporcionando el soporte necesario para garantizar la durabilidad y seguridad de las vías de comunicación. En el contexto de España, donde se ha desarrollado una extensa red de carreteras, es esencial comprender la construcción y el uso de bases de carreteras ejecutadas mediante zahorra compactada [5].

La zahorra artificial es un material granular, de granulometría continua, constituido por partículas total o parcialmente trituradas, en la proporción mínima que se especifique en cada caso y que es utilizado como capa de firme. Es un material ampliamente utilizado en la construcción de carreteras, conocido por su capacidad para proporcionar una base sólida y resistente. la zahorra compactada se ha empleado tradicionalmente en la construcción de carreteras en España, y su uso sigue siendo común en la actualidad [3]

En la ejecución de bases de carreteras con zahorra compactada, es esencial seleccionar materiales de calidad y seguir los procedimientos adecuados. El Pliego de Prescripciones Técnicas Generales (PG-3) [6] en su artículo 510 muestra las características técnicas de las zahorras para ser aplicadas en capas de carreteras, así como el control de calidad a seguir para la correcta ejecución de capas de carretera donde se aplica este material.

Una de las ventajas de la zahorra compactada es su capacidad para adaptarse a diferentes situaciones y condiciones geográficas, la flexibilidad en la selección de materiales y la técnica de compactación permite que las bases de zahorra compactada se utilicen eficazmente en diversas ubicaciones, desde áreas urbanas hasta entornos rurales [2].

En términos de construcción, la aplicación de zahorra compactada generalmente implica la preparación del terreno, la colocación de la zahorra, y su compactación adecuada. Además, se debe prestar atención a las propiedades de los materiales utilizados, como la densidad, la resistencia y la capacidad de drenaje. Estos aspectos son cruciales para asegurar que la base de la carretera cumpla con los requisitos de carga y resistencia [3, 7]

Cabe destacar que en la normativa española, como lo establece la "Instrucción 6.1 IC" [8] se detallan las especificaciones técnicas que deben seguirse en la construcción de bases de carreteras con zahorra compactada. Esto incluye pautas específicas para la selección de materiales, métodos de compactación y evaluación de propiedades para garantizar un rendimiento óptimo.

Además, la normativa establece que la utilización de materiales reciclados como zahorra artificial debe cumplir con ciertas proporciones. Según las directrices de la "Instrucción 6.1 IC," [8] se recomienda que no se exceda el 30% de material reciclado en la composición total de la zahorra artificial. Esta limitación garantiza que se mantengan las propiedades necesarias de compactación y resistencia.

1.1.1.2.- Estabilización de suelos para subbases de carreteras

La estabilización de suelos es un conjunto de procedimientos físicos y/o químicos para la modificación de las propiedades de un suelo, con el fin de que mejore respecto a la resistencia mecánica y a la deformación y que, a su vez, estas propiedades perduren a largo plazo. Este proceso se lleva a cabo en suelos naturales de baja capacidad portante o que sean sensibles al agua, provocando una inestabilidad volumétrica. Mediante la aplicación de estas técnicas se obtiene un material con capacidad para soportar las condiciones climáticas adversas, resistir efectos de la circulación de tránsito de vehículos, controlar el hinchamiento, reducir la plasticidad, permeabilidad y erosión e incrementar la resistencia [9]:

Las técnicas de estabilización se basan en procedimientos físicos y/o químicos centrados en modificar las propiedades de un suelo para mejorarlo en relación con su resistencia, deformación y cambios dimensionales [10, 11].

Algunas de las técnicas más utilizadas para la estabilización de suelos son las siguientes:

Estabilización física. Mejora las propiedades del suelo produciendo cambios físicos. Los métodos de estabilización física se aplican principalmente a suelos granulares. Los principales procedimientos para la estabilización física de un suelo son:

- Mezcla de suelos, necesitando esta técnica de compactación complementaria.
- Uso de geotextiles
- Compactación mediante vibro flotación
- Consolidación previa

Estabilización mecánica. Este método de estabilización se centra en la compactación del suelo, cuyo objetivo es aumentar la resistencia al corte. Los métodos de estabilización mecánica se aplican principalmente a suelos granulares y arenosos.

Estabilización química. Utilización de sustancias químicas que sustituyen iones y produce cambios en la constitución del suelo. El producto debe tener la capacidad de modificar alguna/s propiedad/es del suelo, tales como la resistencia o la permeabilidad. La función esencial de estos productos es la de aglomerar y ligar entre si las partículas finas. Los agentes estabilizantes químicos (por ejemplo, cemento, yeso, cal y otros aditivos alcalinos) se han utilizado comúnmente para la construcción de autopistas, vías férreas y pistas de aeropuertos para mejorar la capacidad de carga, reducir asentamientos, controlar el encogimiento y la hinchazón, y reducir la permeabilidad [12, 13].

Los mecanismos de estabilización química pueden ser resumidos como los siguientes [14]:

- Intercambio de cationes: el sodio, el magnesio y otros cationes son sustituidos por los cationes del calcio cationes del hidróxido de calcio disponible.
- Floculación y aglomeración: la floculación de las partículas de arcilla aumenta el tamaño de grano de las partículas de arcilla aumenta el tamaño de grano efectivo y reduce la plasticidad, aumentando así la resistencia de la matriz.
- Reacción puzolánica: el entorno de alto pH creado por el hidróxido de calcio disponible solubiliza los silicatos y aluminatos en la superficie de la arcilla, que a su vez reaccionan con los iones de calcio para formar productos cementantes, conocidos como geles CSH.
- Cementación por carbonatación: el óxido de calcio reacciona con el dióxido de carbono de la atmósfera para formar precipitados de carbonato de calcio, que cementan las partículas del suelo.

Finalmente, la elección del método de estabilización más adecuado depende del tipo de suelo. A continuación se establecen algunos criterios básicos para la elección del método más efectivo ateniendo a la tipología de suelo a estabilizar [15]:

Suelos granulares gruesos y finos:

- Estabilización mecánica
- Riegos asfálticos
- Suelo/ grava cemento
- Aplicación de cal – cenizas volantes

Arcillas de baja plasticidad:

- Modificación con cal
- Impermeabilizantes químicos
- Compactación y estabilización con cemento

Arcillas de alta plasticidad:

- Estabilización con cal

A continuación, se profundiza en los mecanismos de estabilización química debido a la adición de cal y materiales estabilizadores alternativos.

- Estabilización de suelos con cal

La estabilización de suelos arcillosos expansivos con cal es una técnica comúnmente aplicada y ampliamente estudiada, [14, 16-18] debido a las mejoras de propiedades que implica su uso.

La adición de cal implica dos tipos de acciones sobre los suelos arcillosos:

- Acciones a corto plazo o inmediatas. Mejora de las propiedades geotécnicas del suelo apreciables tras pocos minutos u horas a la aplicación de la cal.
- Acciones a medio/largo plazo. Mejoras producidas tras meses o años de la aplicación de la cal.

Inmediatamente tras la aplicación de la cal, esta se hidrata inmediatamente (es decir, se combina químicamente con el agua) y libera calor. Los suelos se secan, porque el agua presente en el suelo participa en esta reacción y porque el calor generado puede evaporar la humedad adicional.

La cal hidratada producida por estas reacciones iniciales reaccionará posteriormente con las partículas de arcilla. Estas reacciones posteriores producirán lentamente un secado adicional porque reducen la capacidad de retención de humedad. Si se utiliza cal hidratada o lechada de cal hidratada en lugar de cal viva, el secado se produce sólo a través de los cambios químicos en el suelo que reducen su capacidad de retención de agua y aumentan su estabilidad. Tras la mezcla inicial, los iones de calcio (Ca$^{2+)}$ de la cal hidratada migran a la superficie de las partículas de arcilla y desplazan el agua y otros iones.

En esta fase, el índice de plasticidad del suelo disminuye drásticamente, al igual que su tendencia a hincharse y encogerse. El proceso, que se denomina "floculación y aglomeración" generalmente ocurre en cuestión de minutos o unas pocas horas.

Las mejoras en las propiedades geotécnicas del suelo arcilloso estabilizado en esta fase inicial son las siguientes [16]:

- Reducción del índice de plasticidad del suelo. El suelo reduce su plasticidad drásticamente, presentando un aspecto granular y friable. Este cambio en la plasticidad del suelo implica una puesta en obra de mayor simplicidad, facilitando las tareas de extracción, carga, transporte, descarga, extendido, compactación, etc.

- Mayor estabilidad volumétrica. Reducción del hinchamiento asociado a la reducción de la plasticidad.
- Incremento en la resistencia a esfuerzo cortante y mejora de la capacidad de carga.
- Mejora de las propiedades de compactación. La estabilización mediante cal suaviza la curva de compactación del suelo estabilizado con respecto al suelo sin tratar, reduciendo su sensibilidad al agua, facilitando la obtención de la densidad máxima en la fase de ejecución.

Relativo a las mejoras producidas a largo plazo, estas se dan cuando se añaden cantidades adecuadas de cal y agua, el pH del suelo aumenta rápidamente por encima de 10,5, lo que permite que las partículas de arcilla se descompongan. La sílice y la alúmina se liberan y reaccionan con el calcio de la cal para formar hidratos de calcio-silicato (CSH) y calcio-aluminato-hidrato (CAH). Los CSH y CAH son productos cementantes similares a los que se forman en el cemento Portland. Forman la matriz que contribuye a la resistencia de las capas de suelo estabilizadas con cal.

A medida que se forma esta matriz, el suelo se transforma de un material arenoso y granular a una capa dura y relativamente impermeable con una importante capacidad de carga. La matriz formada es permanente, duradera y significativamente impermeable, produciendo una capa estructural que es a la vez fuerte y flexible. Sin embargo, la alteración de la estructura de las partículas se produce lentamente, dependiendo del tipo de arcilla presente, de las condiciones de humedad y temperatura.

Estas reacciones puzolánicas a largo plazo se traducen en un aumento de la capacidad portante del suelo y resistencia mecánica. A su vez, el suelo pierde permeabilidad a medida que cementa. Las mejoras en capacidad portante y reducción de la permeabilidad implican un aumento de la durabilidad de la obra.

- Estabilización de suelos con materiales alternativos

En los últimos años, se han estudiados numerosos materiales alternativos como potencial estabilizar suelos expansivos. A continuación, se hace un breve resumen del estado del arte actual:

Estabilización con sales.

Metodología utilizada principalmente para el control de polvo. Las sales utilizadas mayoritariamente en la estabilización son:

- Cloruro de calcio ($CaCl_2$): El cloruro de calcio disminuye las fuerzas de repulsión de las arcillas, incrementando su cohesión [19].
- Cloruro de sodio (NaCl): Ayuda a mejorar las características mecánicas del suelo, disminuye la resistencia al corte y cambios volumétricos, en condiciones de humedad alta es capaz de absorber hasta 10 veces su propio peso

Estabilización con nanomateriales.

La nanotecnología es una innovación en la técnica de estabilización química de suelos expansivos, debido a la mayor interacción que generan estas nanopartículas, lo cual conlleva una reducción de aditivo para la estabilización y un beneficio medioambiental [20].

Un compuesto químico nanotecnológico es capaz de modificar las características fisicoquímicas del suelo (capilaridad, expansividad y permeabilidad), controlando y reduciendo los efectos producidos por el agua, como la erosión hídrica y el porcentaje de hinchamiento en suelos expansivos. Además, aumenta la resistencia a la compresión simple.

Estabilización con residuos y subproductos

Aunque las técnicas de estabilización con conglomerantes hidráulicos generan una mejora ambiental respecto a otros métodos como la sustitución de suelos marginales por suelos granulares, la producción de cal viva y cemento mediante la calcinación de la cal genera un gran impacto ambiental tanto por las emisiones de CO_2 como por el agotamiento de los recursos naturales [4].

Debido a esto, en los últimos años varios autores han estudiado la posibilidad de aplicar residuos y subproductos de diferentes fuentes industriales como agente estabilizador de suelos, como el fosfoyeso para la formación de terraplenes y taludes [21], la escoria de acería para reducir la expansividad y mejorar la capacidad portante [22], el óxido de magnesio como material alternativo al cemento [23], etc.

Entre todos los tipos de residuos y subproductos, las cenizas de fondo de biomasa y las cenizas volantes de biomasa han sido estudiadas en los últimos años para su aplicación en la ejecución de subbases de carreteras [24] o en la estabilización de suelos [25, 26].

De acuerdo con el estado actual de las técnicas de estabilización de suelos con materiales alternativas, en la presente Tesis Doctoral se propone el uso de Cenizas de Fondo, Cenizas Volantes de Biomasa, Escoria Blanca de Acería como subproductos a aplicar en los procesos de estabilización química debido sus propiedades físicas y químicas, capaces de modificar las propiedades de los suelos. Además, estudia la estabilización de suelos arcillosos expansivos mediante la aplicación de tres nanomateriales base sílice.

Finalmente, dadas las propiedades de ambos materiales, se estudia la combinación de subproductos y nanomateriales como material estabilizador.

1.1.2.- Marco legal de la ejecución de carreteras

Desde el punto de vista normativo es interesante realizar un análisis histórico del conjunto de documentos que, desde los años setenta, han regulado la posibilidad de aplicación y especificaciones exigidas al tratamiento de suelos con cal en el ámbito de las carreteras y conformación de capas de zahorra compactada.

En este análisis es posible encontrar cuatro grandes grupos de normas o disposiciones reguladoras, que se pueden en agrupar en:

a) Las clasificaciones de suelos recogidas en los pliegos generales por cuanto afectan, aunque someramente, a los materiales susceptibles de tratamiento y materiales para zahorras compactadas:

- Pliego de prescripciones técnicas generales para obras de carreteras y puentes (PG-3/75) aprobado por Orden Ministerial de 6 de febrero de 1976 (B.O.E. del 7 de Julio)
- Orden circular 326/00 de la Dirección General de Carreteras, de febrero de 2.000, sobre geotecnia vial en lo referente a materiales para la construcción de explanaciones y drenajes, en cuanto que modifica la clasificación de suelos para terraplenes recogida en el artículo 330 Terraplenes. Fue elevada a rango de Orden Ministerial FOM/1382/2002 el 16 de mayo de 2.002 con su publicación en el B.O.E. el 11 de junio del mismo año.

b) Las instrucciones de firmes, que especifican el posible uso de este tipo de materiales y sus limitaciones:

- Instrucción 6.1.IC 1975 de “Firmes Flexibles”, así como la

Instrucción 6.2.IC 1975 de “Firmes Rígidos” aprobadas por Orden Ministerial de 12 de Marzo de 1976 del Ministerio de Obras Públicas y Urbanismo.

- Instrucción 6.1 y 2-IC sobre secciones de firme, aprobada por Orden Ministerial de 23 de mayo de 1989 (B.O.E. del 30 de junio).
- Orden Circular 10/2002 de la Dirección General de Carreteras del Ministerio de Fomento, sobre Secciones de firme y capas estructurales de firmes.

c) Las especificaciones recogidas en cuanto a características y condiciones a exigir a los materiales, tanto a la cal como al suelo y al producto final del tratamiento:

- Artículo 510 Suelos estabilizados in situ con cal del PG-3/75, Pliego de prescripciones técnicas generales para obras de carreteras y puentes (PG -3/75) aprobado por Orden Ministerial de 6 de febrero de 1976 (BOE del 7 de Julio).
- Orden Circular 297/88T sobre estabilización de suelos in situ y tratamientos superficiales con ligantes hidrocarbonados, de 29 de marzo de 1988, que revisó dicho artículo 510 Suelos estabilizados in situ con cal.
- Orden Circular 10/2002 sobre secciones de firme y capas estructurales de firmes, de la Dirección General de Carreteras del Ministerio de Fomento, que introduce el nuevo artículo 512 Suelos estabilizados in situ que anula el anterior artículo 510 de la Orden Circular 297/88.

d) Las normas que regulan la producción y caracterización del conglomerante, la cal:

- Artículo 200 Cal aérea del PG-3/75, Pliego de prescripciones técnicas generales para obras de carreteras y puentes (PG-3/75) aprobado por Orden Ministerial de 6 de Febrero de 1976 (BOE del 7 de Julio).
- Orden Ministerial de 27 de Diciembre de 1.999 por la que se actualizan determinados artículos del pliego de prescripciones técnicas generales para obras de carreteras y puentes en lo relativo a conglomerantes hidráulicos y ligantes hidrocarbonados (B.O.E. del 22 de enero de 2.000).

En la actualidad, El artículo 510 “zahorras” perteneciente al Pliego de Prescripciones Técnicas Generales, PG-3, aprobado mediante la orden

FOM 2523/2014, regula las características técnicas y condiciones a exigir tanto a los materiales como al producto denominado suelo estabilizado terminado.

El artículo 512 “Suelos estabilizados in situ” perteneciente al Pliego de Prescripciones Técnicas Generales, PG-3, aprobado mediante la orden FOM 2523/2014, regula las características técnicas y condiciones a exigir tanto a los materiales como al producto denominado suelo estabilizado terminado.

Así mismo, la orden FOM/3460/2003, de 28 de noviembre, por la que se aprueba la norma 6.1 IC secciones de firme, de la instrucción de carreteras (BOE de 12 de diciembre de 2003) especifica las condiciones para la aplicación de los suelos estabilizados en capas de carreteras, así como sus limitaciones.

1.2.- Residuos de construcción y demolición

Los Residuos de Construcción y Demolición (RCD), se definen como aquellos “*residuos generados por las actividades de construcción y demolición*” de acuerdo con la Ley 7/2022 De Residuos y Suelos Contaminados Para una Economía Circular.

Sin embargo, el concepto de RCD en el ámbito legal para fomentar su reciclaje y valorización, se remonta a 2008, con en el Real Decreto 105/2008, legislación encargada de la gestión y producción de este residuo. La definición dada es “*cualquier sustancia u objeto que, cumpliendo con la definición de residuo del artículo 3.a) de la Ley 10/1998, de 21 de abril, se genera en una obra de excavación, nueva construcción, reparación, remodelación, rehabilitación y demolición, incluyendo el de obra menor y reparación domiciliaria”*

Se consideran obras de construcción y demolición [27] aquellas actividades de reparación, reforma, nueva construcción o demolición de una construcción.

Las políticas actuales relativas a los RCD, en concordancia con la Ley 7/2022, tienen como objetivo la reducción de la generación de residuos y la reutilización, reciclado y valorización de los ya producidos.

1.2.1.- Producción y gestión de RCD

Analizando la producción de residuos por sector productivo en Europa, en la Figura 1.3., se observa que aproximadamente un tercio de los residuos generados en el continente provienen de actividades relacionadas con el sector de la construcción.

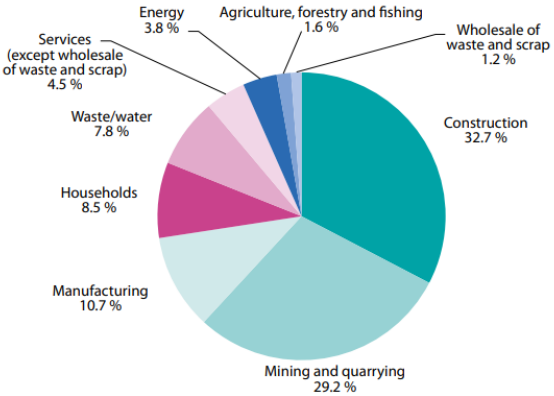


Fig. 1. 3. Producción de residuos por sector productivo [28]

Debido a la alarmante cantidad de residuos generados por el sector de la construcción, la Ley 7/2022 establece que "l*a cantidad de residuos no peligrosos de construcción y demolición destinados a la preparación para la reutilización, el reciclado y otra valorización de materiales, incluidas las operaciones de relleno, con exclusión de los materiales en estado natural definidos en la categoría 17 05 04 de la lista de residuos, deberá alcanzar como mínimo el 70% en peso de los producidos*"

La producción de RCD en España, se mantiene relativamente constante desde 2015, produciendo entre 14 y 15 millones de toneladas al año. Sin embargo, analizando los datos relativos a la valorización de los residuos [29] se observa un gran aumento en la valorización de los RCD entre los años 2015 y 2017, pasando de un 40% de valorización a un 75%, alcanzándose valores cercanos al 90% en el año 2019.

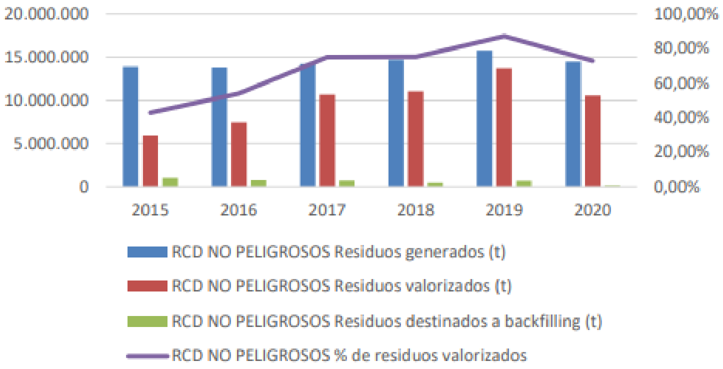


Fig. 1. 4. Producción y valorización RCD en España. Periodo 2015-2020 [29]

Estas cifras son producto de las políticas y legislación española en ámbito de residuos. Actualmente la gestión y los objetivos de valorización y reutilización de los RCD quedan fijados en Plan Estatal Marco de Residuos de Gestión de Residuos (PEMAR) 2023-2035 [27], donde se establecen los objetivos y pautas para conseguir un producto final valorizado de calidad y en cantidades significativas. Las pautas son las siguientes:

Separación de los RCD en fracciones y establecimiento de mecanismos de recogida selectiva.

Fomento de la utilización de RCD mediante la implantación de medidas como la subida de tasa de vertido o establecer obligaciones al promotor o constructor por la incorrecta separación de RCD.

Fomentar la valorización. Establecer un Acuerdo Marco Sectorial para impulsar la utilización de áridos reciclados procedentes de RCD en obras de construcción. Así se propone la inclusión, siempre que sea posible, en los proyectos de construcción de obra pública de un porcentaje mínimo del 5 % de áridos reciclados. Igualmente se aplicará este porcentaje del 5 %, siempre que sea posible, en la obra privada.

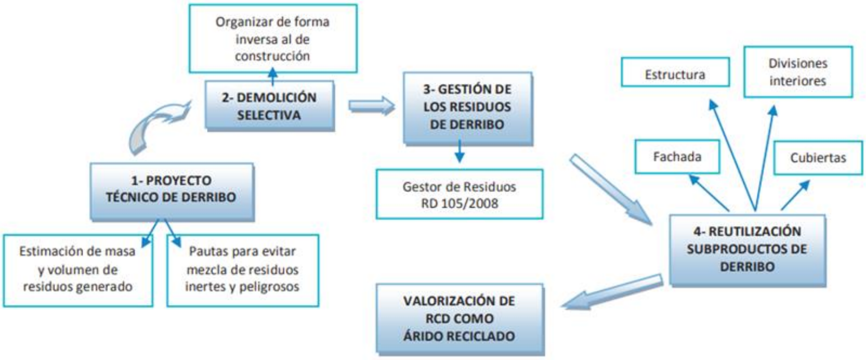


Fig. 1. 5. Esquema general del ciclo de vida de un RCD para su valorización [30]

La Valorización de RCD contempla dos importantes consideraciones:

La valorización de RCD no es la contabilización de las entradas de RCD a Gestores Autorizados, hay que conocer cuántos de estos residuos se reciclan y cuantos son finalmente eliminados en vertedero como rechazos.

La valorización de RCD en rellenos y restauraciones debe realizarse con materiales reciclados procedentes de gestores autorizados, nunca con RCD, (las tierras limpias de excavación para rellenos y restauraciones no están contempladas).

1.2.2.- Composición de los de RCD

La composición de los RCD varía en función del tipo de infraestructuras de que se trate y refleja en sus componentes mayoritarios, siendo estos normalmente productos cerámicos, residuos de hormigón, material pétreo y residuos asfálticos [31]. El tipo y distribución porcentual de las materias primas que utiliza el sector, si bien hay que tener en cuenta que éstas pueden variar de un país a otro en función de la disponibilidad de estos y los hábitos constructivos.

Los materiales minoritarios, entre los que se encuentran residuos de madera, plásticos, vidrio, yeso, papel, etc., dependen en cambio, de un número de factores mucho más amplio como pueden ser el clima del lugar, el poder adquisitivo de la población, los usos dados al edificio etc.

Por otro lado, la composición de las edificaciones varía a lo largo del tiempo y con ello también cambia la composición de los RCD, según sea la; edad del edificio o estructura que es objeto de demolición.

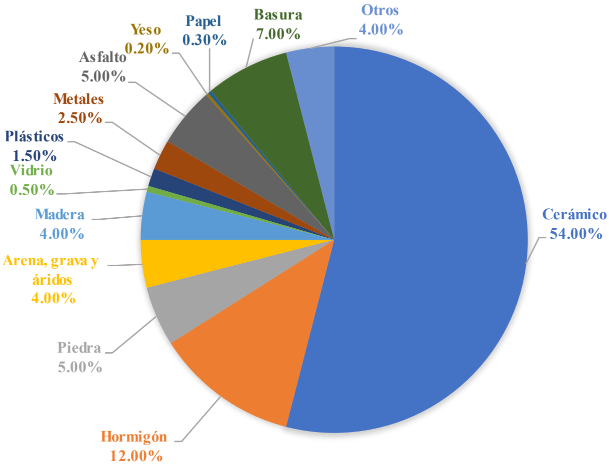


Fig. 1. 6. Composición Residuos de Construcción y Demolición [32]

Como se observa en la Figura 1.6., los residuos cerámicos o mixtos y los residuos de hormigón son los más abundantes en la composición de los RCD.

Los residuos mixtos o cerámicos son producidos generalmente en las operaciones de demolición de estructuras de edificación. De acuerdo con los análisis realizados por los organismos gubernamentales [33], en España aproximadamente el 80% de los residuos producidos son de este tipo, producidos en las demoliciones de edificaciones. Los residuos cerámicos también son producidos como rechazo en la producción de ladrillos, cifrando estos entre un 5-10% la cantidad de ladrillo desechado en los procesos de producción. Si bien la cantidad de este material no es tan significativa, se trata de un residuo muy homogéneo con unas propiedades más estables en el tiempo.

Los residuos de hormigón, sin embargo, proceden principalmente de la demolición de obras de ingeniería civil.

1.2.3.- Tipología de áridos reciclados

Los áridos reciclados son el producto reciclado producido en los centros de valorización y que cumple con los requisitos técnicos para una aplicación determinada.

Los centros de valorización son instalaciones de tratamiento de RCD, cuyo objetivo es seleccionar, clasificar y valorizar las diferentes fracciones que contienen estos residuos, con el objetivo de obtener productos finales aptos para su utilización directa, o residuos cuyo destino será otro tratamiento posterior de valorización o reciclado, y si este no fuera posible, de eliminación en vertedero. Las plantas de producción de áridos reciclados son bastante similares a las plantas de machaqueo de áridos naturales, incluyen machacadoras, cribas y dispositivos de transporte (cintas transportadoras, cangilones, etc.) [30]. Adicionalmente, disponen de equipos para la eliminación de contaminantes y electroimanes para la separación del acero. La planta de tratamiento debe asegurar unas máximas distancias de transporte, es decir, situarse lo más cerca posible del centro de la ciudad donde se originan la mayoría de los residuos de la construcción y donde se da una más amplia demanda de los áridos reciclados.

Tras el tratamiento de los RCD en planta, los áridos reciclados se clasifican en diferentes tipos según su origen y las proporciones de sus componentes:

- **Áridos Reciclados de Hormigón**: Producidos a partir de la trituración y procesamiento de residuos de hormigón.
- **Áridos Reciclados Cerámicos**: Producidos a partir de residuos con una presencia predominante de material cerámico.
- **Áridos Reciclados Mixtos**: Resultan del tratamiento de RCD con diferentes naturalezas.

Si bien esta clasificación cualitativa es aceptada, existe una falta de legislación que regule la definición de los áridos reciclados de acuerdo con su composición y propiedades fisicoquímicas. A continuación se muestran las clasificaciones propuestas por la “Guía Española de Áridos Reciclados” correspondiente al proyecto GEAR [31] y la clasificación propuesta en el reglamento AGRECA de certificación de producto [34].

La clasificación propuesta por la “Guía Española de Áridos Reciclados” en base a la composición está formada por cuatro tipos o categorías de áridos reciclados:

- **Categoría ARH**: Áridos Reciclados de Hormigón: el contenido de hormigón y piedra natural (sin mortero adherido) es del 90% o más en peso. Se suma el contenido de hormigón al de piedra natural.
- **Categoría ARMh**: Áridos Reciclados Mixtos de Hormigón: el

contenido de hormigón y piedra es menor al 90% y el de material cerámico no alcanza el 30%.

- **Categoría ARMc**: Áridos Reciclados Mixtos Cerámicos: el contenido de material cerámico supera el 30%.
- **Categoría ARC**: Áridos Reciclados Cerámicos: el contenido de material cerámico supera el 70%. Se incluyen en esta categoría los AR con asfalto.

Tabla 1. 1. Composición de los áridos reciclados de acuerdo con la clasificación GEAR [31]

Nomenclatura	Nombre	Características
ARH	Áridos Reciclados de hormigón	P+H > 90% C<10% A<5% X<1%
ARMh	Áridos Reciclados Mixtos de Hormigón	P+H > 70% C<30% A<5% X<1%
ARC ARMc	Áridos Reciclados Cerámicos Áridos Reciclados Mixtos Cerámicos	P+H < 30%C <30% A<5% X<1% C<70% A<5% X<1%
ARCARMc	Áridos Reciclados Cerámicos Áridos Reciclados Mixtos Cerámicos	P+H <70% C<0% A<5% X<1%C>% A<5% X<1%
ARMa	Áridos Reciclados Mixtos con Asfalto	5%<A<30% X<1%

P: Piedra; H: Hormigón; C: Cerámico; A: Asfalto; X: Otros

Esta clasificación debe completarse con la determinación del contenido de los otros dos tipos de fracciones en la mezcla, dado que su exceso determina la pérdida de la consideración de árido reciclado:

Contenido de Asfalto: Áridos Reciclados con Asfalto: cuando el árido reciclado contiene entre un 5% y un 30% de materiales bituminosos, más del 30% se considera Mezcla Bituminosa.

Contenido de “impropios”: se considera que con más de un 1% en peso de “impropios”, no puede definirse como árido reciclado, y debe definirse como “Material Inerte”.

Por lo tanto, no tienen la consideración de áridos reciclados los siguientes materiales que, sin embargo, pueden tener otros usos adecuados a sus características técnicas específicas:

Mezclas bituminosas (MB): aquellos materiales inorgánicos previamente utilizados en la construcción con un contenido de mezclas bituminosas superior al 30% en peso.

Por su parte, AGRECA, propone una clasificación basada en la aplicación de los áridos reciclados como zahorra para capas de carreteras, teniendo en cuenta composición y propiedades de los áridos reciclados. Se muestra a continuación la clasificación basada en la composición:

Tabla 1. 2. Composición de los áridos reciclados de acuerdo con la clasificación AGRECA

		ZARHor	**ZARM I**	**ZARM II**	**ZARA**
Composición UNE-EN 933-11	**Rc+Ru+Ra**	-	≥70%	≥70%	≥90%
	Rc+Ra	≥90%	≥55%	≥55%	-
	Rc	-	-	-	-
	Ra	-	-	-	≥50%
	Rb	-	-	-	
	X	<1%	<1%	<2%	<1%
	FL	<1cm^3/kg	<1cm^3/kg	<2cm^3/kg	<1cm^3/kg

Rc: Hormigón; Ru: Piedra; Rb: Cerámico; Ra: Asfalto; X: Otros; FL: Flotantes

- **ZARHor**: Zahorra reciclada de hormigón. Contenido de hormigón reciclado y piedra natural mayor al 90%. Contenido de otros compuesto no flotantes menor al 1%. Contenido en material flotante (madera, plásticos, etc.) menor a 1 cm^3/kg.
- **ZARM I**: Zahorra reciclada mixta tipo I. Contenido de hormigón y piedra natural mayor al 55%. Contenido de otros compuesto no flotantes menor al 1%. Contenido mínimo de mezcla bituminosa 15%. Contenido en material flotante (madera, plásticos, etc.) menor a 1 cm^3/kg.
- **ZARM II**: Zahorra reciclada mixta tipo II. Contenido de hormigón y piedra natural mayor al 55%. Contenido de otros compuesto no flotantes menor al 2%. Contenido mínimo de mezcla bituminosa 15%. Contenido en material flotante (madera, plásticos, etc.) menor a 2 cm^3/kg.

- **ZARA**: Zahorra reciclada asfáltica. Contenido de hormigón reciclado, piedra natural y mezcla bituminosa mayor al 90%. Contenido mínimo de mezcla bituminosa 50%. Contenido de otros compuesto no flotantes menor al 1%. Contenido en material flotante (madera, plásticos, etc.) menor a 1 cm^3/kg.

1.2.4.- Aplicación de áridos reciclados mixtos en obras de carretera e ingeniería civil

En las últimas décadas, se ha producido un avance significativo en las investigaciones relacionadas con el uso de áridos reciclados procedentes de RCD [35, 36]. Los ARM han ganado relevancia como una de las formas más abundantes de áridos reciclados de RCD y han sido objeto de un estudio más detallado en esta Tesis Doctoral. Los ARM presentan propiedades físicas distintas en comparación con los áridos naturales, como una menor densidad y una mayor absorción de agua.

Diversos estudios realizados por diferentes autores [37, 38] coinciden en que los ARM muestran una mayor variabilidad en sus propiedades, incluyendo una menor densidad, mayor absorción y propiedades físicas, como el coeficiente de desgaste de Los Ángeles, menos exigentes en comparación con los áridos naturales. Estas diferencias hacen que, a pesar de las elevadas tasas de generación de ARM, su expansión en aplicaciones técnicamente exigentes represente un desafío.

Entre las aplicaciones más comunes se encuentran de los ARM se encuentran las siguientes:

- **Bases y subbases de carreteras:**

Estudios han demostrado que los ARM aplicados en explanadas y capas estructurales de carreteras rurales pueden ofrecer resultados positivos en cuanto a capacidad de soporte, siempre y cuando se controlen las propiedades limitantes, como el contenido de sales solubles y yeso [39, 40]. Estos hallazgos respaldan la viabilidad de uso de ARM en caminos rurales.

La estabilización de ARM con cemento ha demostrado reducir la susceptibilidad frente al hielo, la permeabilidad y la lixiviación [41]. Varios autores han estudiado el comportamiento de ARM estabilizados con cemento en bases y subbases de carreteras, subrayando la importancia de controlar el contenido de sulfatos.

- Hormigones y morteros

Los áridos reciclados de hormigón han sido ampliamente utilizados en la fabricación de hormigón no estructural, que desempeña una función de mejora de las condiciones de durabilidad en la construcción [37, 42, 43]. La limitación más restrictiva en el uso de ARM en hormigones no estructurales suele ser el contenido de sulfatos.

La normativa española Código Estructural limita el uso de AR en hormigón estructural y establece valores límites para impurezas que pueden afectar la durabilidad y resistencia [44]. La composición química de los ARM, incluyendo altos niveles de sulfatos y cloruros, ha llevado a limitar su uso en hormigón estructural. A pesar de esto, es posible diseñar dosificaciones que incluyan ARM como sustituto del árido grueso convencional dentro de ciertos límites de reemplazo [39].

Los ARM también pueden ser utilizados en la fabricación de morteros, especialmente en morteros de albañilería, donde la sustitución de arena natural por árido reciclado se ha estudiado en profundidad. A pesar de la influencia en las propiedades mecánicas, la adhesión en morteros fabricados con ARM suele ser excelente, aunque se requiere una sobredosificación de cemento para cumplir con las resistencias mínimas [45-47].

1.3.- Residuos y subproductos industriales

La Estrategia Española de Economía Circular, España Circular 2030, del Ministerio de Transición Ecológica y Reto Demográfico (MITERD), sienta las bases para impulsar un nuevo modelo de producción y consumo en el que el valor de productos, materiales y recursos se mantenga en la economía durante el mayor tiempo posible, a la vez que se reduzca al mínimo la generación de residuos y se aprovechen con el mayor alcance posible los que no se pueden evitar.

Por otro lado, dentro de esta Estrategia y sus correspondientes planes de actuación, se incluyen, como una de las relacionadas con el cierre del círculo, las materias primas secundarias.

Las medidas propuestas por el eje de actuación “Materias primas secundarias” van encaminadas a garantizar la protección del medio ambiente y la salud humana, reduciendo el uso de recursos naturales no renovables y reincorporando en el ciclo de producción los materiales contenidos en los residuos como materias primas secundarias.

La derogada Ley 22/2011, de 28 de julio, de residuos y suelos contaminados ya incorporó a nuestro ordenamiento jurídico los conceptos de “subproducto” y “fin de condición de residuo”, que contribuyen a delimitar la aplicación del régimen jurídico de los residuos y a desarrollar el eje de actuación de Materias primas secundarias.

Siguiendo las pautas de esta regulación, y de la Directiva Marco de residuos, la Ley 7/2022, de residuos y suelos contaminados para una economía circular, que entró en vigor en abril del año pasado, recoge en sus artículos 4 y 5 estos conceptos, y aprovecha para regular los procedimientos mediante los cuales pueden aplicarse.

Se muestra a continuación las definiciones dadas por la Ley 7/2022 para los conceptos de residuo, subproducto y condición de fin de residuo.

- **Residuos**

Un residuo es “*cualquier sustancia u objeto que su poseedor deseche o tenga la intención o la obligación de desechar*”.

La gestión de los residuos producidos, su entrega a gestor autorizado o bien, la puesta en disposición de estos a un agente para su posterior gestión es responsabilidad directa del productor.

La consideración de un material como residuo, salvo excepciones, conlleva además las siguientes consideraciones:

i. Limitación para la comercialización y reducción del consumo del material: Esto implica que cuando un material se considera un residuo, puede estar sujeto a restricciones en su comercialización y uso. Esto tiene como objetivo reducir el consumo de materiales que pueden ser dañinos para el medio ambiente o la salud humana.

ii. Pérdida de valor en el mercado asociada al material debido a las limitaciones de uso: Cuando un material se convierte en residuo y está sujeto a restricciones, su valor en el mercado puede disminuir, ya que las limitaciones de uso pueden reducir su demanda.

iii. Obligaciones que debe asumir el productor: En el proceso de gestión de residuos, el productor tiene varias responsabilidades, que incluyen:

La gestión de los residuos ya sea por el propio productor o a través de gestores autorizados, de acuerdo con la categoría asignada al material en el Listado Europeo de Residuos (LER).

- El almacenamiento, mezcla, envasado y etiquetado adecuados del material en cuestión.
- La inscripción en el registro correspondiente de producción de residuos.
- La presentación de una declaración anual de residuos industriales.
- El mantenimiento de registros de aceptación y seguimiento, entre otras obligaciones documentales.
- El cumplimiento de la normativa de traslado de residuos, tanto a nivel nacional como internacional.

Debido a estas responsabilidades y para cumplir con la legislación que promueve la condición de residuo, muchas industrias se esfuerzan en categorizar sus residuos industriales como subproductos. Al hacerlo, buscan evitar las restricciones y limitaciones asociadas con los residuos y, en su lugar, dar un valor adicional a los materiales, lo que puede ser beneficioso tanto desde el punto de vista ambiental como económico.

- **Subproductos**

Una sustancia u objeto resultante de un proceso de producción, cuya finalidad no sea la producción de esa sustancia u objeto, se puede considerar un subproducto cuando se cumplan, simultáneamente, las siguientes condiciones:

1. Que se tenga la seguridad de que la sustancia u objeto va a ser utilizado ulteriormente
2. Que la sustancia u objeto se pueda utilizar directamente sin tener que someterse a una transformación ulterior distinta de la práctica industrial habitual
3. Que la sustancia u objeto se produzca como parte integrante de un proceso de producción
4. Que el uso ulterior cumpla todos los requisitos pertinentes relativos al producto, así como a la protección de la salud humana y del medio ambiente, sin que produzca impactos generales adversos para los mismos

Las Comunidades Autónomas evalúan y autorizan como subproducto las sustancias u objetos que tengan origen en una instalación productora ubicada en su territorio, o en otra comunidad autónoma previo informe favorable de ésta, siempre que se destinen a una actividad o proceso industrial concreto en el territorio de la propia comunidad autónoma. Es muy importante tener en cuenta que estas autorizaciones tienen validez, únicamente, para el uso autorizado del subproducto en la actividad o proceso industrial de destino.

- **Condición de fin de residuo**

Determinados tipos de residuos, que hayan sido sometidos a una operación de valorización, incluido el reciclado, podrán dejar de ser considerados como tales, siempre que se cumplan todas las condiciones siguientes:

1. Que las sustancias, preparados u objetos resultantes deban ser usados para finalidades específicas
2. Que exista un mercado o una demanda para dichas sustancias, preparados u objetos
3. Que las sustancias, preparados u objetos resultantes cumplan los requisitos técnicos para las finalidades específicas, la legislación existente y las normas aplicables a los productos
4. Que el uso de la sustancia, preparado u objeto resultante no genere impactos adversos globales para el medio ambiente o la salud humana

Las Comunidades Autónomas, a petición del gestor de residuos, podrán incluir en la autorización que concedan conforme al artículo 33 de la Ley 7/2022 de residuos (autorización de operaciones de recogida y tratamiento de residuos), que un residuo valorizado en una instalación ubicada en su territorio deja de ser residuo para que sea usado en una actividad o proceso industrial concreto ubicado en esa misma comunidad autónoma, o bien en otra comunidad autónoma previo informe favorable de esta última.

1.3.1.- Generación y gestión de residuos y subproductos industriales

El incremento en la producción de residuos y subproductos industriales, junto con su limitada valorización, constituye una problemática creciente en las sociedades europea y española. En 2020, la UE generó un total de 2.154 millones de toneladas de residuos provenientes de actividades económicas y de los hogares, lo que representa un promedio de 4.815 kg por persona.

En ese mismo año, el 39,9% de los residuos tratados se recicló, mientras que el 12% se destinó a rellenos, y el porcentaje de valorización energética alcanzó un 6,5%. Comparativamente, entre 2004 y 2020, la UE incrementó su tasa de tratamiento de residuos totales del 45,9% al 59,1%, al tiempo que redujo la tasa de eliminación del 54,1% al 40,9%, aunque estos valores aún están lejos de los objetivos propuestos [48].

La acción de reciclar los residuos se convierte en un eslabón crucial para reducir el consumo de recursos primarios, sustituyéndolos por materiales secundarios que ya han tenido al menos un uso previo. A pesar de que Europa aspira a construir una economía completamente circular, resulta interesante analizar cómo están desempeñando los diferentes países lo que respecta a la gestión de residuos y conocer cuántos residuos se generan y reciclan por habitante en Europa, y cuáles son los países europeos líderes en reciclaje.

Para obtener respuestas a estas interrogantes, es crucial considerar las distintas definiciones que nos ayudan a abordar los desafíos relacionados con los residuos en Europa. El tratamiento de residuos se clasifica en dos categorías principales: recuperación y eliminación. La recuperación abarca actividades como el reciclaje, la recuperación de energía y el relleno, que consiste en utilizar los residuos para reacondicionar zonas excavadas, como graveras y minas subterráneas. Por otro lado, la eliminación comprende el vertido y la incineración.

En este contexto, la presente Tesis Doctoral plantea la posible aplicación de subproductos industriales y residuos, reduciendo así la tasa de eliminación, aumentando el porcentaje de valorización y fomentando el desarrollo de un modelo de economía circular.

1.3.2.- Marco legal de la generación de residuos

Para evaluar la viabilidad de utilizar subproductos que previamente se han acumulado en vertederos, es esencial llevar a cabo un análisis exhaustivo del marco legal y las regulaciones promulgadas por las autoridades competentes. En esta sección, se abordará la situación legal y la normativa relacionada con la gestión de residuos y subproductos industriales, con el objetivo de establecer los requisitos fundamentales para la gestión de residuos.

El marco legal europeo para la gestión de residuos es fundamental en la consideración de la viabilidad de aprovechar los subproductos que han sido previamente depositados en vertederos. La Directiva marco de residuos, inicialmente establecida en 1975 en Europa, ha sido complementada a lo largo de los años con diversas directivas adicionales que abordan flujos específicos de residuos. Sin embargo, es importante destacar que la implementación plena de esta directiva se ha visto obstaculizada debido a la falta de prioridad por parte de los Estados miembros de la comunidad, quienes consideran que no existen datos fiables acerca de la calidad de los materiales y su aplicación [49]. En este sentido, es esencial una aplicación más rigurosa de la legislación y el respaldo institucional de la Unión Europea para asegurar un desarrollo sostenible.

Además, se deben tener en cuenta otros factores que pueden provocar daños ambientales, así como aumentos en los costes y riesgos para la salud humana. Esto se relaciona con el incumplimiento de los requisitos impuestos por la Unión Europea, como el uso inadecuado de tecnología para la gestión de residuos en vertederos y otras instalaciones, así como los traslados ilegales de estos materiales.

A continuación, se presenta una visión general de la legislación comunitaria vigente y su estado de aplicación:

Directiva marco sobre residuos (2008/98/EC): La Comisión Europea publicó en 2007 una Comunicación interpretativa sobre los residuos y subproductos, en la que se definieron los términos clave. No obstante, es importante destacar que un material no se clasifica como residuo según el Tribunal de Justicia de la Unión Europea si es posible su reutilización segura, sin necesidad de una transformación previa ni de una solución de continuidad en el proceso productivo [50, 51]. Posteriormente, la Unión Europea presentó la Directiva 2008/98/EC del Parlamento Europeo y del Consejo, en la cual se aborda la gestión de residuos desde su producción hasta su eliminación. Esta directiva se centra en la valorización y el reciclaje, con el objetivo principal de proteger el medio ambiente y la salud humana al prevenir los efectos nocivos derivados del ciclo de producción y gestión de residuos.

Marco Legal en España: En el contexto español, la planificación para la gestión eficiente de los residuos se basa en el Programa Estatal de Prevención de Residuos 2014-2020, el Plan Estatal Marco de Gestión de Residuos (PEMAR 2016-2022) y el Plan Nacional Integrado de Residuos para el período 2017-2019 (PNIR 2017-2019). Estas iniciativas se alinean con la Estrategia 2020 y los objetivos prioritarios del VII Programa de Medio Ambiente de la Unión Europea, en vigencia hasta 2020.

Además, la Ley 22/2011, de 28 de julio, de residuos y suelos contaminados y la Ley 11/1997, de 24 de abril, de envases y residuos de envases, actualmente derogada por la Ley 7/2022, de 8 de abril, de residuos y suelos contaminados para una economía circular complementan la normativa española en materia de residuos. Esta legislación establece reglamentos específicos para la regulación de diversos flujos de residuos, como es el caso de los Residuos de construcción y demolición (Real Decreto 105/2008). Asimismo, las operaciones de valorización y eliminación de residuos se encuentran reguladas por la Orden MAM/304/2002.

A nivel autonómico, cada región cuenta con su legislación específica. Por ejemplo, en Andalucía, se han desarrollado el Plan Integral de Residuos de Andalucía. Hacia una Economía Circular en el Horizonte 2030.

La evolución de todas estas normativas en el ámbito de la gestión de residuos ha impulsado el debate sobre la distinción entre los residuos y las materias primas secundarias. Se plantean cuestiones fundamentales, como cuándo un residuo deja de serlo y se convierte en una materia prima secundaria [49].

En consonancia que lo anteriormente expuesto, en los siguientes apartados se realiza un estudio del estado del conocimiento de los residuos y subproductos utilizados para el desarrollo de la presente Tesis Doctoral. Se analizan los siguientes subproductos:

Cenizas de fondo y Cenizas volantes de biomasa son analizadas. Son un subproducto procedente de la quema de biomasa para la producción de energía. Se considera un sector de vital importancia debido al peso que tiene sobre la producción de energía a nivel estatal y autonómico, y al crecimiento de la tasa de generación de este residuo como motivo del aumento de producción eléctrica.

Escoria blanca de acería. Son un subproducto resultante de la fabricación de acero en acerías de horno de arco eléctrico. Procedentes de un sector vital para la economía española, este subproducto se encuentra actualmente sin una valorización eficiente, a pesar de presentar unas propiedades adecuadas para su aplicación como material estabilizador en capas de carreteras. Sin embargo, es necesario realizar un estudio ambiental detallo para evitar problemas asociados con contaminación por metales pesados presente en su composición.

1.4.- Cenizas procedentes de la quema de biomasa

La producción de energía a partir de fuentes renovables está aumentando en todo el mundo. En Europa, el 39.4% de la producción de energía primaria se produjo a partir de fuentes renovables en 2022 (Figura 1.7.) [52], lo que indica una tendencia hacia modelos energéticos más sostenibles. Entre estas fuentes de recursos renovables para la producción de energía primaria, el uso de biocombustibles, que es cualquier tipo de biomasa susceptible de ser quemada, está en alza debido a que es una energía limpia y está ampliamente aceptado que el balance de emisiones de gases de efecto invernadero es nulo gracias a la renovación de la biomasa, ya que no se rompe el ciclo del carbono.

Fig. 1. 7. Generación neta de electricidad en la UE por tipo de combustible (2022) [52]

Sin embargo, el aumento de la producción de energía a partir de la biomasa genera un incremento de los residuos producidos durante la combustión, las cenizas de biomasa, que sin un tratamiento adecuado acaban depositándose en vertederos, provocando efectos negativos en el medio ambiente.

La biomasa es un residuo orgánico que proviene de diferentes fuentes, como animales, madera y recortes de árboles, procesos agrícolas o industriales y residuos urbanos municipales. Se considera un combustible renovable, ya que su poder calorífico proviene del sol, en una transformación energética reciente, por lo que su combustión no genera un impacto en la huella de carbono (Figura 1.8.) a diferencia de los combustibles fósiles, cuya transformación energética ocurrió hace millones de años [53].

El poder calorífico de estos residuos junto con su producción constante, de naturaleza autóctona y sin dependencia de regiones externas, lo convierten en un combustible muy interesante, aumentando su uso como fuente de producción de energía en las plantas de combustión de todo el mundo [54].

La biomasa es un residuo que proviene de diferentes fuentes, por lo que se hace una clasificación típica según su origen, que es la siguiente:

- Biomasa agrícola: Se incluyen en esta denominación todos los residuos orgánicos y restos vegetales generados por la actividad agrícola, ya sea intensiva, extensiva, cultivos protegidos, etc.
- Biomasa forestal: Los residuos de origen forestal comprenden todos los productos o subproductos resultantes de los aprovechamientos y tratamientos silvícolas que se realizan, bajo criterios técnicos, en las superficies forestales para diferentes aprovechamientos. Provienen de la necesidad de realizar tratamientos silvícolas para el mantenimiento y mejora de los montes y masas forestales mediante talas, podas, limpieza de matorrales, etc. Estos trabajos generan una biomasa leñosa (leñas, ramas y matorrales) que deben ser retirados del monte, pues son un factor de riesgo de grave importancia para la propagación de plagas y de incendios forestales.
- Biomasa Ganadera: los residuos ganaderos son aquellos residuos orgánicos generados por las especies ganaderas en las explotaciones intensivas ganaderas. Se tratan principalmente de la mezcla de deyecciones y la cama de ganado, denominándose comúnmente según la especie de la que proceden en estiércol (ganado vacuno, ovino y equino), purines (ganado porcino) y gallinaza (ganado avícola).
- Biomasa industrial: son aquellos subproductos y desechos de origen orgánico generados por la industria, principalmente la agroalimentaria, forestal, papelera y textil.
- Biomasa urbana: Los residuos sólidos urbanos son aquellos que se originan en los núcleos de población como consecuencia de la actividad habitual y diaria del ser humano. Se clasifican en dos grandes grupos: Fracción Orgánica Residuos Municipales

(FORM) (antes denominados Residuos Sólidos Urbanos (RSU)), y Aguas Residuales Urbanas (ARU).

- Biomasa acuícola/algal: Las microalgas son organismos fotosintéticos que transforman la energía solar en energía química mediante la fotosíntesis. Por tanto, fijan el CO_2 y nitrógeno atmosférico, colaborando al control del efecto invernadero y la lluvia ácida, a los que contribuyen en gran medida los combustibles fósiles.
- Cultivos energéticos: Son especies vegetales cultivadas expresamente para su uso energético. Existen especies cuya aptitud principal es exclusivamente energética (Cynara Carduncululus, Brassica Carinata, Paulownia de corta rotación etc.), y otros cultivos alimentarios tradicionales que pueden ser empleados también para uso energético en su totalidad, como es el caso del cereal y las oleaginosas para ser empleados en la fabricación de biocarburantes.

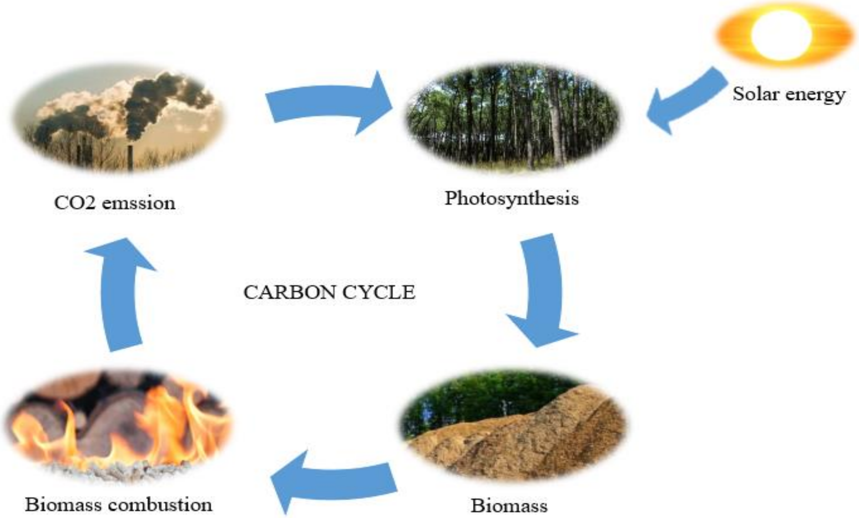


Fig. 1. 8. Ciclo de carbono de la biomasa en combustión [55]

El uso de biomasa en la generación de energía conlleva múltiples ventajas en comparación con los combustibles fósiles convencionales. En primer lugar, la biomasa se deriva de fuentes renovables y es, en gran medida, un subproducto de la actividad humana, lo que garantiza un suministro constante [56]. El aprovechamiento de residuos y subproductos fomenta un enfoque más ecológico en la economía, respaldando la filosofía de "cuna a cuna" (C2C) y promoviendo un modelo de economía circular a nivel local o regional, donde se recoge la biomasa.

Además, las áreas que producen biomasa pueden volverse energéticamente autosuficientes, reduciendo al mismo tiempo el impacto ambiental de los residuos generados por actividades agrícolas o municipales. Este enfoque también incentiva la mejora de los sistemas de gestión de residuos y la creación de empleos locales dedicados a estas tareas, contribuyendo así al desarrollo sostenible de las comunidades.

Focalizando en la región de Andalucía y analizando el potencial de biomasa detectado en esta comunidad, Figura 1.9., se observa que este asciende a 1.477,4 ktep. Si se tiene en cuenta que el consumo de energía primaria en Andalucía en 2021 fue de 17.256,4 ktep significa que el potencial de biomasa representó ese año el 8.52% % de las necesidades energéticas en Andalucía [57]. Para ello, Andalucía cuenta con 18 centrales térmicas de biomasa, con una potencia instalada de 257 MW.

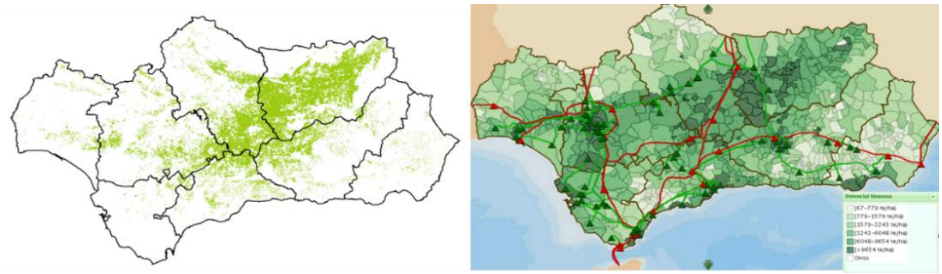


Fig. 1. 9. Cultivos de olivar (izquierda) y potencial de biomasa energética (derecha) en Andalucía [58]

Sin embargo, a pesar de las ventajas mencionadas, el uso de la biomasa como combustible también tiene algunos inconvenientes, ya que la baja densidad de la biomasa en comparación con los combustibles fósiles implica que se necesitan grandes superficies para su almacenamiento y manejo; el principal problema relacionado con la producción de energía a partir de la combustión de biomasa es la gran cantidad de cenizas que se producen en las calderas, que implica un coste extra en la producción y puede generar problemas ambientales sin una gestión adecuada [59].

La presente Tesis Doctoral pretende ampliar el uso y aplicación de las cenizas de biomasa procedentes de la industria del olivar, dado su gran volumen de producción en Andalucía, reduciendo así su depósito en vertedero, con los consecuentes riesgos ambientales que supone, y dando un valor añadido a un residuo con un alto potencial en el sector de la construcción.

1.4.1.- Tipología de cenizas de biomasa

El mayor uso de biomasa como combustible sólido ha implicado un incremento en la producción de residuos en forma de cenizas procedentes de su combustión en plantas de generación de energía térmica y eléctrica. Estos residuos se producen en dos tipologías, Cenizas Volantes y Cenizas de Fondo de Biomasa (CVB y CFB), Figura 1.10.

Las cenizas de fondo de biomasa es un producto parcialmente no quemado y heterogéneo que se recoge del fondo de los hornos. Estas cenizas son un material arenoso-terroso, mezclado con impurezas no combustibles adheridas a la biomasa, como piedras, tierra etc.

Las cenizas volantes de biomasa son la fracción fina e inorgánica que se retiene en los filtros de las chimeneas de las instalaciones de combustión, para evitar su expulsión a la atmósfera [60].

Fig. 1. 10. Ceniza de fondo de biomasa (CFB) (Izquierda) y Ceniza volante de biomasa (CVB) (Derecha)

La cantidad y las propiedades de las cenizas de biomasa (CB) varían dependiendo del tipo de biomasa quemada y de la tecnología de combustión utilizada. No obstante, los elementos más comunes en la CB son típicamente el calcio (Ca), el potasio (K), el silicio (Si), el magnesio (Mg) y el aluminio (Al), además de la presencia de oligoelementos como el manganeso (Mn), el cadmio (Cd), el cromo (Cr), el zinc (Zn), entre otros. [61], En particular, los compuestos más frecuentemente encontrados en la CB de diversas biomasas son el óxido de calcio (CaO) y el dióxido de silicio (SiO_2) [60]

Las cenizas de fondo de biomasa suelen representar la mayor proporción en la producción total de CB, aproximadamente un 60-70%. Sin embargo, las CFB contienen un porcentaje mayor de material no quemado y materia orgánica.

Las cenizas volantes de biomasa pueden contener más contaminantes potenciales debido a la presencia de elementos volátiles y metales pesados [62]. Esta acumulación de contaminantes en grandes cantidades en los vertederos podría plantear riesgos ambientales.

1.4.2.- Aplicación de cenizas de biomasa en obras de carreteras e ingeniería civil

Con el fin de minimizar el impacto ambiental de las cenizas de biomasa, su utilización ha sido ampliamente estudiada en las últimas décadas y existen numerosos trabajos sobre su uso en la aplicación industrial [63]. A continuación, se enumeran los principales usos de las CB:

- Enmienda del suelo y fertilización, aportando nutrientes y elementos compuestos al suelo y reduciendo la acidez gracias a su carácter alcalino.
- Adsorbente de elementos contaminantes como, As, Au, B, Cd, Co, Cr, Cu, F, Hg, Mn, Ni, Pb y Zn, o compuestos, como NH_3, NO_X, PO_4, SOX, fenoles, tolueno, hidrocarburo aromático policlorado, etc.
- Síntesis de minerales y diferentes materiales como cerámicas, vidrios, agregados ligeros, geopolímeros, etc.
- Materiales de construcción y aplicación en ingeniería civil, como la fabricación de materiales enn base de cemento (cemento, mortero de cemento, hormigón) y la estabilización de suelos en la construcción de carreteras.

La aplicación de las cenizas procedentes de la combustión de la biomasa en el ámbito de la construcción queda condicionada por tres aspectos:

- **Cantidad**. La utilización de las cenizas en el ámbito de la construcción necesita asegurar unas cantidades mínimas anuales que garanticen su uso. La cantidad relativa producidas depende de una serie de variables, entre ellas el tipo de caldera, las características de la biomasa empleada y otros factores como el tipo de combustión [64].
- **Composición.** Según Masia et al. [65], en general, los mayores constituyentes de las cenizas de biomasa suelen ser el calcio, potasio, sodio, silicio y fósforo.

- **Aplicabilidad**. La aplicación de las cenizas de biomasa en el ámbito de la construcción quedará condicionada al cumplimiento de las diferentes normas que regulan la calidad de los materiales producidos, entre ellos el Pliego de Condiciones para la Construcción de Carreteras y Puentes [6], el código estructural o la Norma 450-1 sobre cenizas volantes para hormigón, que establece las definiciones, especificaciones y criterios de conformidad que deben de cumplir. En las normas citadas se no admiten el uso de las cenizas procedentes de la combustión de biomasa exclusivamente. No obstante, existen estudios experimentales que han caracterizado este tipo de subproducto y demuestran que es posible su utilización en la producción de cementos [66] por su carácter puzolánico, como sustituto o como retardante del fraguado en lugar del yeso [67] y en la producción de geopolímeros [68]. Han sido aplicadas en la fabricación de morteros [69, 70] y hormigones [55, 59, 71].

A pesar de ello, el grado de conocimiento del comportamiento de las cenizas de biomasa como material de construcción no está aún lo suficientemente contrastado, lo que se evidencia en la carencia de normativa específica. La síntesis de geopolímeros utilizando cenizas de fondo ya ha sido propuesta para la formación de bloques de cemento [72] sin embargo su aplicación no se ha generalizado debido a diferentes problemas intrínsecos con las cenizas.

Se muestran a continuación otros estudios desarrollados en los últimos años acerca de la aplicación de cenizas de biomasa como material de construcción.

- Bases y subbase de carreteras

En estudios recientes, las cenizas de fondo de biomasa han sido consideradas en aplicaciones relacionadas con la construcción. Por ejemplo, Cabrera et al. [60] llevaron a cabo una caracterización exhaustiva de estas cenizas, abordando sus propiedades físicas, químicas y mecánicas. A pesar de que aún contenían un nivel significativo de materia orgánica, los resultados respaldaron su uso en conformidad con las directrices del PG-3, calificándolas como adecuadas para su empleo en núcleos de terraplenes.

Hinojosa et al. [56] se centraron en explorar el potencial uso de las cenizas de fondo de biomasa como material de construcción alternativo, abordando los parámetros químicos que pudieran plantear desafíos en cumplimiento con las regulaciones técnicas requeridas.

Cabrera et al. [41] combinaron árido reciclado mixto con cenizas de fondo de biomasa para bases y subbases de carreteras, mejorando el comportamiento del material al incorporar las cenizas de fondo de biomasa.

- **Hormigones y morteros**

Beltrán et. al [73], comprobaron el rendimiento mecánico de morteros fabricados con cenizas de fondo de biomasa y posteriormente Rosales et al. [70] aplicaron diferentes tratamientos a las cenizas de fondo de biomasa para su utilización en morteros de cemento con resultados muy positivos.

Una de las temáticas más innovadoras que se han investigado ha sido la aplicación de Cenizas de Fondo de Biomasa en la producción de hormigones ligeros [71] y en bloques de mortero [72], los cuales se podría aplicar con fines de aislamiento térmico y acústico, debido a sus condiciones físicas de material poroso, su baja densidad, o su alta capacidad de absorción.

1.5.- Escorias de acería de horno de arco eléctrico

El acero, un material versátil y de excepcionales propiedades mecánicas, ocupa un lugar fundamental en la sociedad moderna. Su aplicación es tan amplia que lo encontramos en sistemas de transporte, infraestructuras críticas, suministro de agua y energía (incluyendo fuentes de energía renovable), maquinaria industrial, viviendas, electrodomésticos y envases. El acero se ha convertido en un elemento esencial de nuestra vida cotidiana, impulsando la economía de varios países desarrollados.

La industria siderúrgica española sobresale como uno de los principales productores de acero en Europa y el mundo, con una producción anual que oscila entre 12 y 14 millones de toneladas [74]. Su compromiso con la sostenibilidad medioambiental se refleja en la gestión responsable de los recursos, la reducción de residuos y el fomento del reciclaje. En las últimas cuatro décadas, ha logrado avances notables: ha reducido sus emisiones de CO_2 en un 75%, disminuido su consumo de agua en un 95% y ha consolidado una producción de acero en la que el 75% proviene de material reciclado. Este último aspecto es especialmente significativo ya que el acero es altamente reciclable, conservando su calidad en cada ciclo. De hecho, hoy en día se reciclan componentes de acero que fueron manufacturados hace más de un siglo, lo que subraya su sostenibilidad y eficiencia.

En el ámbito de la producción de acero, existen dos enfoques predominantes. Por un lado, la siderurgia integral, que emplea principalmente mineral de hierro como materia prima, complementando con aproximadamente un 20% de chatarra para regular la temperatura del proceso. Por otro lado, los hornos de arco eléctrico, que utilizan exclusivamente chatarra como materia prima. Estos últimos han ganado popularidad gracias a la disminución de los costos de producción, la eficiencia en la duración del proceso y la ausencia de emisiones de gases de combustión. En España, alrededor del 70% de la producción de acero se realiza en hornos de arco eléctrico [75].

La distribución geográfica de estos hornos en España es diversa, con una siderurgia integral y 21 aceras de horno eléctrico de arco (Figura 1.11.). Aunque la mayoría se ubica en la región de la Cornisa Cantábrica, en particular en Asturias y el País Vasco, la industria siderúrgica está presente en once de las Comunidades Autónomas de España, respaldando la economía y el empleo en todo el país [74].

Fig. 1. 11. Localización de las acerías de horno de arco eléctrico en España [76]

Sin embargo, la producción de acero conlleva la producción de residuos, denominados "Escorias". Debido a la importancia estratégica de la industria de la acería de horno eléctrico, en la presente Tesis Doctoral se plantea la aplicación de los residuos producidos como material estabilizador en capas de carreteras.

1.5.1.- Tipología de escorias procedentes de horno de arco eléctrico

Escoria de acería es el nombre que se asigna al material fundido formado por reacciones químicas entre la materia prima, los materiales añadidos al horno y las impurezas oxidadas durante el refinado del metal [77].

Las escorias de acero generalmente se clasifican según el tipo de horno en el que se producen, tal y como se muestra en la Figura 1.12. Las propiedades de la escoria dependen del tipo de proceso utilizado para producir el acero y de las condiciones de enfriamiento de la escoria y el proceso de valorización.

El proceso de fabricación del acero, tanto común como especial, en las acerías de horno de arco eléctrico se compone de dos etapas: una primera denominada metalurgia primaria o fusión, donde se produce la fusión de las materias primas que se realiza en hornos de arco eléctrico, y la segunda, denominada metalurgia secundaria o afino del baño fundido, que se inicia en el horno eléctrico y finaliza en el horno cuchara.

Fig. 1. 12. Principales subproductos generados en la industria siderúrgica [22]

Las escorias de acería son el residuo resultante de la fundición de materiales en la metalurgia primaria. Debido al gran volumen de producción, en la presente Tesis Doctoral, se van a analizar las escorias producidas en la fabricación de acero mediante hornos de arco eléctrico.

La principal materia prima empleada para la fabricación de acero en horno de arco eléctrico es la chatarra de hierro dulce o acero. Como elementos auxiliares se pueden cargar también pequeñas cantidades de fundición, de mineral de hierro y de ferroaleaciones.

La etapa de fusión incluye una serie de fases como la oxidación, dirigida a eliminar impurezas de manganeso y silicio, la defosforación y la formación de escoria espumante en la que se acumulan todas las impurezas. Al final de todas estas fases se extraen las **escorias negras**.

La etapa de afino incluye la desoxidación, que permite eliminar los óxidos metálicos del baño, la desulfuración y la descarburación del acero. El líquido fundido procedente del horno eléctrico se alimenta al horno-cuchara, se cubre con una escoria que se denomina escoria blanca y se agita continuamente con el soplado de gas inerte, normalmente argón. La escoria blanca permite la reducción de los óxidos metálicos presentes en el baño, durante la denominada fase de desoxidación. Paralelamente se realiza la desulfuración del líquido fundido, que se produce por simple contacto con la cal existente en la **escoria blanca (EBA)**.

En el año 2018, se generaron cerca de 1,2 millones de toneladas de escorias negras y unas 230 mil toneladas de escorias blancas.

Fig. 1. 13. Escorias de acero (De izquierda a derecha: Escoria Negra y Escoria Blanca)

Sobre el uso de las escorias negras, el uso mayoritario, un 49% del total, es en construcción de carreteras (uso como árido), mientras que un 5% se dedicó a otras aplicaciones ligadas y un 18% a aplicaciones no ligadas. Aproximadamente un 4% fue destinado a vertedero. Sobre el uso de las escorias blancas, el único uso admitido, materia prima en la fabricación del Clinker, demanda gran parte de la producción, sin embargo, indicar que una parte importante es depositada en vertedero [75].

De las dos tipologías de escorias producidas, en la presente Tesis Doctoral se van a aplicar la escoria blanca producida por la empresa Siderúrgica Sevilla, ya que al contener una granulometría compuesta principalmente de partículas finas su potencial de valorización como material para bases de carreteras es limitado, depositándose mayoritariamente en vertedero, sin embargo, debido a sus propiedades físicas y químicas se presenta como un potencial material estabilizador de suelos.

1.5.2.- Aplicación de escoria acería en obras de carreteras e ingeniería civil

Las escorias de acero han sido ampliamente estudiadas, sus propiedades fisicoquímicas son conocidas y existen numerosos estudios en los que se demuestra su posibilidad de aplicación, debido a eso, existen diferentes estudios y aplicaciones de las Escorias de Siderurgia.

Según las estadísticas publicadas por Euroslag [78], en el año 2016 el uso de las escorias de acería fue de 25,8 millones de toneladas, de las cuales, el 49,3% se utilizó en la construcción de carreteras, 16,4% en uso metalúrgico, 4,7% producción de cemento y 2,4% en la ingeniería hidráulica como aplicaciones de ingeniería civil, el restante en otros usos.

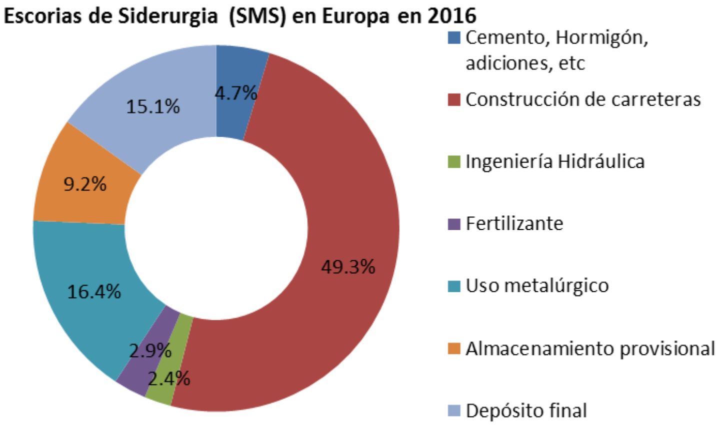


Fig. 1. 14. Aplicaciones de Escorias de Acero año 2016 [78]

Así, desde el punto de vista económico para los productores de acero, la consideración de la escoria como subproducto en lugar de residuo conlleva, además del ahorro del canon de vertido, poder comercializar directamente la escoria, producto generado paralelamente junto al acero por motivos inherentes de la producción de este último.

- Bases y subbases de carreteras

Es bien conocida la aplicación de las escorias negras de acería como material en la construcción de carreteras, tanto en subbases, bases o capas de rodadura. Como hemos observado al comienzo de este estudio, el 48% de las escorias de acería son utilizadas en la construcción de carreteras.

Debido a la alta resistencia a la fricción y a la abrasión, la escoria de acero está siendo más utilizada en carreteras, intersecciones y áreas de estacionamiento donde se requiere una alta resistencia al desgaste [79].

La utilización de la escoria blanca en este tipo de aplicaciones ha sido penalizada por su gran cantidad de finos. En el trabajo desarrollado por Izco et al. [80], se contempla la posibilidad de valorización de la mezcla de ambas escorias, que son ampliamente utilizadas para la construcción de pistas forestales, ya que presentan unas propiedades muy ventajosas respecto de las que posee cada tipo de escoria empleada individualmente.

Vázquez [81], describe la experiencia belga con el uso de escorias de acero inoxidable como agregado en el asfalto para la construcción de camino.

Otros estudios desarrollados por Wu et al. [82] demostraron la viabilidad de utilización de la escoria de acero como agregado en mezclas de asfalto.

- **Hormigones y morteros**

A su vez, existen varios estudios [83, 84] basados en la sustitución parcial o total del cemento y/o la arena por escorias de acería en los morteros. El estudio realizado por Manso et al. [84], se centra en la comparación de propiedades físicas y aspectos de durabilidad entre los morteros convencionales y aquellos con una sustitución de cemento y la fracción fina de arena por la escoria blanca. El porcentaje en peso de la escoria blanca en los morteros frescos está alrededor del 22%.

Por otro lado, los estudios realizados por Sheen et al. [85] demostraron la posibilidad de sustitución en una tasa de reemplazo de hasta el 30% de cemento por escorias de acero inoxidable con diferentes tratamientos de finura obteniéndose mayores resistencias a compresión que los morteros fabricados con el 100% de cemento.

Referente a la fabricación de hormigón a partir de escorias de acería, Moosberg-Bustnes [86] realizó un estudio sobre la viabilidad del uso de diferentes escorias de acería en el hormigón. Así, el trabajo recoge las investigaciones del uso de la fracción fina de la escoria desintegrada procedente de la fabricación de acero inoxidable procedente de descarbonización con oxígeno de argón (AOD) como árido de fracción fina en el hormigón, junto a los efectos de la molienda húmeda realizado sobre AOD y escoria de Horno de Arco Eléctrico (EAF) para usar como árido fino en el hormigón y por último estudia el tratamiento de fusión más granulación de la escoria blanca, AOD y EAF para utilizarlos como aditivo en el hormigón. La escoria de acería de horno eléctrico puede ser utilizada para sustituir arena en hormigón y se pudo observar que no existe riesgo ambiental asociado con su utilización.

Otros estudios llevados a cabo por Sheen et al. [87] demostraron la posibilidad de sustitución del 100% de la fracción fina de áridos naturales por escoria de acero inoxidable con un tratamiento de oxidación, separación magnética y tamizado. Una sustitución del 100% produce mejores propiedades de hormigón endurecido, como la resistencia a la compresión, la resistencia superficial y la velocidad de ultrasonido.

Pellegrino & Gaddo [88]demostraron que la escoria de horno arco eléctrico es adecuada para reemplazar los agregados naturales tradicionales en conglomerados, incluso en porcentajes altos y para tamaños medianos (hasta 2-4 mm de tamaño). Al igual que demostró Kortbaoui et al. [89] años antes en su estudio.

De acuerdo con los estudios previos, el potencial cementante de las EBA está demostrado. Esta capacidad cementante es el punto de partida para aplicarlo como material estabilizador de suelos arcillosos expansivos, ya que indica la posibilidad de mejoras en las propiedades mecánicas. Sin embargo, es necesario mantener un control ambiental debido al potencial contaminante de este subproducto.

1.6.- Nanomateriales

Los nanomateriales, en el contexto de obras de carreteras, se refieren a materiales cuyas estructuras y características físicas se encuentran a escala nanométrica. Los nanómetros son una unidad de medida extremadamente pequeña, equivalente a una milmillonésima parte de un metro (10^{-9} metros) [90]. Los nanomateriales pueden tener una amplia gama de aplicaciones en la construcción de carreteras debido a sus propiedades únicas y su capacidad para mejorar la resistencia, durabilidad y sostenibilidad de la infraestructura vial.

La nanotecnología constituye una innovación incremental en el desarrollo de estabilizantes químicos, al combinar las propiedades de las formulaciones convencionales con la capacidad de interacción a nivel molecular que tienen las nanopartículas incorporadas otorgando una mejora de propiedades geotécnicas al terreno.

Los nanomateriales pueden originarse a partir de diversos procesos, siendo uno de los métodos más comunes la nanosíntesis.

La nanosíntesis implica la creación de materiales a nivel nanométrico a partir de materiales más grandes. También se pueden encontrar nanomateriales de origen natural, como ciertos minerales y arcillas que tienen estructuras nanométricas.

1.6.1.- Tipos de nanomateriales

Existen varias tipologías de nanomateriales, cada uno con propiedades únicas que pueden ser aprovechadas en obras de carreteras. Algunos ejemplos incluyen [91]:

Nanopartículas metálicas: Pequeñas partículas de metales como el oro, la plata o el cobre, que se utilizan en aplicaciones como la mejora de la conductividad eléctrica en señalización vial.

Nanotubos de carbono: Tubos de carbono con diámetros nanométricos que pueden mejorar la resistencia y durabilidad del asfalto y el hormigón en carreteras.

Nanopartículas de sílice: Utilizadas para mejorar la resistencia al desgaste y la vida útil de las carreteras y como agentes de sellado en pavimentos.

Nanofibras poliméricas: Refuerzan la estabilidad y resistencia de los materiales utilizados en la construcción de carreteras.

El presente apartado se va a centrar en los nanomateriales base sílice, siendo estos los más interesantes para su aplicación las tareas de estabilización de suelos para carreteras.

1.6.1.1. Nanosílice

En los últimos años, las nanopartículas han atraído un considerable interés científico para muchas aplicaciones de ingeniería civil. Los tipos de nanopartículas que se usan más comúnmente en compuestos cementosos son SiO_2, TiO_2, Al_2O_3 y nanotubos de carbono [92]. De todas las nanopartículas introducidas, el nano-SiO_2 desempeña el papel más importante. Las nanopartículas de SiO_2 exhiben una alta actividad puzolánica debido a la alta cantidad de SiO_2 amorfo puro [93].

Los nanomateriales que se utilizan comúnmente para mejorar las propiedades geotécnicas son nano-sílice. Influyen en las propiedades de consolidación, permeabilidad, resistencia y propiedades de índice de plasticidad. La nano-sílice está compuesta de partículas finas de SiO_2 que están conectadas por enlaces covalentes y de hidrógeno que forman partículas más grandes. La ventaja más importante de la nano-sílice sobre la sílice, es su área de superficie específica más grande que causa más interacción entre las partículas.

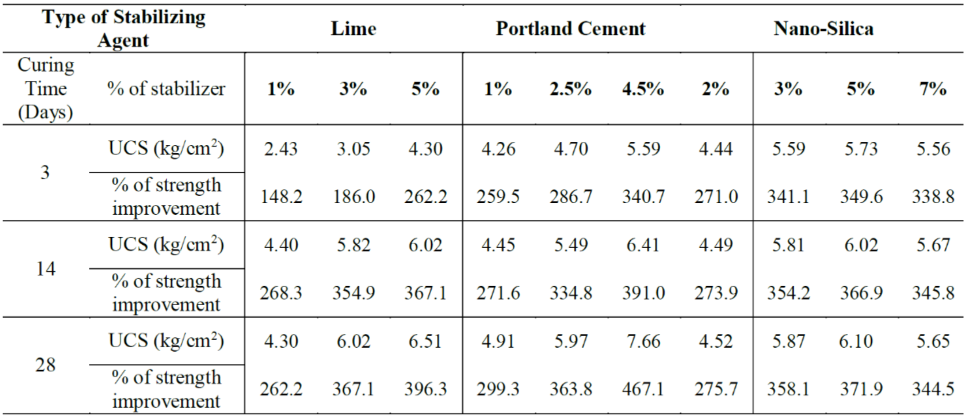
Las nanopartículas interactúan muy activamente con otros constituyentes del suelo (incluida la fase líquida, los cationes, la materia orgánica y minerales de arcilla) debido a su área de superficie específica excepcionalmente alta y las superficies reactivas con cargas, por lo que tienen una profunda influencia en la microestructura y las propiedades físicas, químicas y de ingeniería de los suelos, incluso cuando representan solo una fracción muy pequeña en peso de la materia [94].

El uso de partículas de nano-SiO_2 lleva a una modificación de las propiedades de las mezclas, debido a que estas partículas son el resultado de una reacción química entre SiO_2 y $Ca\ (OH)_2$ durante la hidratación del cemento o de la cal [95].

El uso de nano-SiO_2 para la estabilización de suelo, según diferentes estudios [96] muestran un aumento de la resistencia a compresión no confinada del suelo en relación con el uso de otros estabilizadores tradicionales como la cal o el cemento, tal y como se muestra en la tabla 1.3.

Además del aumento de resistencia, la adición de nano-SiO_2 produce una disminución del índice plástico, un aumento del contenido de humedad óptima del suelo y una ligera disminución de la densidad seca máxima. Se puede afirmar que la aplicación de nano-SiO_2 puede acelerar la estabilización del suelo a ciertos niveles [97].

Tabla 1. 3. Resistencia a compresión no confinada de suelo estabilizado [93]



Type of Stabilizing Agent		Lime			Portland Cement			Nano-Silica			
Curing Time (Days)	% of stabilizer	1%	3%	5%	1%	2.5%	4.5%	2%	3%	5%	7%
3	UCS (kg/cm^2)	2.43	3.05	4.30	4.26	4.70	5.59	4.44	5.59	5.73	5.56
	% of strength improvement	148.2	186.0	262.2	259.5	286.7	340.7	271.0	341.1	349.6	338.8
14	UCS (kg/cm^2)	4.40	5.82	6.02	4.45	5.49	6.41	4.49	5.81	6.02	5.67
	% of strength improvement	268.3	354.9	367.1	271.6	334.8	391.0	273.9	354.2	366.9	345.8
28	UCS (kg/cm^2)	4.30	6.02	6.51	4.91	5.97	7.66	4.52	5.87	6.10	5.65
	% of strength improvement	262.2	367.1	396.3	299.3	363.8	467.1	275.7	358.1	371.9	344.5

1.6.2.- Aplicación de nanomateriales en obras de carreteras e ingeniería civil

Uno de los usos más prometedores de los nanomateriales en obras de carreteras es su aplicación como material estabilizador de suelos. Los suelos estabilizados con nanomateriales muestran una mayor capacidad de soporte, resistencia a la erosión y durabilidad en comparación con los suelos convencionales. Esto puede llevar a la construcción de carreteras más sólidas y de larga duración.

En los últimos años se han realizado diversos estudios de aplicación de nanomateriales en obras de carreteras, por ejemplo, las siguientes:

Kulanthaivel et al. [98] estudiaron el efecto del uso de Nano-SiO2 sintetizado mediante un proceso sol-gel junto con cemento para estabilizar suelos arcillosos, concluyendo que una adición del 7% de nano-SiO2 mejora la resistencia a la compresión no confinada y reduce la permeabilidad en el rango de 0,01976 cm/seg a 0,01198 cm/seg del suelo, lo que concuerda con estudios anteriores realizados por Choobbasti et al. [99].

Ghasabkolaei et al. [100] y Bahmani et al. [97] estudiaron la estabilización de suelos con cementos y nanosilano con dosis inferiores al 1% en peso de Nano-SiO_2, observando un aumento de la resistencia a la compresión no confinada en todas las mezclas con aditivos nanoparticulados y una elevada tasa de formación de hidrato de silicato cálcico (CSH), lo que implica una mejora de las propiedades del suelo.

Además de estudios de laboratorio, se encuentran algunas experiencias reales de aplicación de nanomateriales en carreteras.

Autovía A-382 de Jerez de la Frontera a Arcos de la Frontera. Tramo III: del P.K. 13+000 al 20+000 (Cádiz) [101].

La propuesta implementada consistió en cambiar los 45 cm de zahorra artificial por el mismo espesor de un suelo estabilizado con cal y con el polímero GB (nombre comercial del polímero proporcionado por Green Road Solutions) como aditivo complementario. Adicionalmente, se proponía otra solución alternativa con los mismos espesores, pero substituyendo la zahorra por un suelo estabilizado sólo con cal, para comparar de manera directa la mejora que los polímeros aportaban a la estabilización.

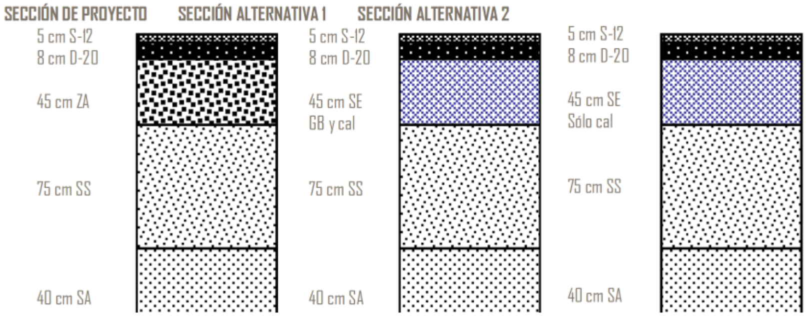


Fig. 1. 15. Sección de firme de proyecto y Soluciones Alternativas [101]

El uso de polímero GB en combinación con la cal llevó a un aumento del valor de resistencia a compresión y menor hinchamiento, proporcionó un aglomerado más estable a nivel de deflexiones, viéndose mínimamente afectado por el cambio estacional. Y a largo plazo cabe esperar la aparición de fisuras en el tramo estabilizado con cal (SC) por la reología asociada a los ligantes puzolánicos, mientras que el tramo estabilizado con cal y polímero (SCP) debería quedar libre de este fenómeno.

Pista de aterrizaje de helicóptero utilizando una aplicación de polímero de infusión pesada sobre granito descompuesto y materiales de suelo in situ. Casar de Cáceres, 2010

La empresa Terratech utilizó la estabilización con polímeros para construir una pista fuerte capaz de soportar la erosión eólica y la abrasión de la superficie causada por los helicópteros de aterrizaje.

Se usaron 500 polímeros para desarrollar la capacidad de carga y la unión de alta resistencia entre las partículas finas y del firme. El suelo existente se molió y se mezcló con la solución de polímero en una mezcla homogénea y luego se compactó en su lugar. Una capa de sellado final se aplicó a mano con un sistema de bomba presurizada y una manguera contra incendios. La mezcla de suelo existente y material granulado de curso bien graduado creó un acabado de superficie suave para la plataforma de aterrizaje.

La aplicación de nanomateriales en la construcción de carreteras ha demostrado ser una innovación revolucionaria con el potencial de mejorar significativamente la resistencia, durabilidad y sostenibilidad de la infraestructura vial. En particular, el uso de nano-SiO_2 ha destacado debido a su capacidad para interactuar activamente con los suelos, lo que resulta en mejoras en la resistencia y otras propiedades geotécnicas.

Esta implementación no solo acelera la estabilización del suelo, sino que también reduce el índice plástico y aumenta el contenido de humedad óptima, lo que lo convierte en un estabilizador de suelos eficaz.

En la presente Tesis Doctoral, se aborda las técnicas de estabilización de suelos con materiales alternativos mediante la combinación de nanomateriales con subproductos, una temática prácticamente inexplorada en la estabilización de suelos, la cual ofrece una solución prometedora para abordar los desafíos de la construcción de carreteras de manera más eficiente y sostenible.

1.7.- Evaluación ambiental para una construcción sostenible

La creación de nuevos modelos productivos basados en la sostenibilidad tiene como objetivo el alcanzar un crecimiento económico sostenido, que garantice el bienestar de la población y la protección del medio ambiente.

En este contexto, la cuantificación del impacto ambiental asociado al desarrollo de nuevos materiales, construcción de nuevas vías a partir de materiales reciclados y afección ambiental derivado de la explotación de las construcciones es un aspecto crucial.

En el presente apartado, se exponen los métodos de cuantificación de impacto ambiental aplicados durante el desarrollo de la presente Tesis Doctoral, concretamente, se cuantifica el potencial contaminador de los residuos y subproductos industriales en relación con los metales pesados lixiviados al entrar en contacto con el agua, así como la inmovilización de estos en los procesos de ejecución de capas de carreteras.

Además, se aplica la metodología de Análisis de Ciclo de Vida, para cuantificar en términos de, por ejemplo, emisiones de gases de efecto invernadero, los procesos industriales de valorización y puesta en obra de los materiales reciclados en comparación con materiales convencionales, pudiendo valorar las posibles mejoras ambientales mediante valores discretos, y no en términos cualitativos.

1.7.1.- Análisis de lixiviados

La lixiviación es un proceso por el cual se liberan sustancias químicas presentes en los materiales, tales como metales pesados, a partir de la infiltración de agua a través de estos materiales. Este fenómeno puede tener impactos significativos en el medio ambiente, contaminando suelos y aguas subterráneas [102].

El proceso de lixiviación es influenciado por diversos factores físicos, tales como el tamaño de las partículas expuestas, el tiempo, las condiciones del flujo lixiviante, la temperatura, la porosidad, la forma geométrica y el tamaño de los materiales, la permeabilidad de la matriz, las condiciones hidrogeológicas, entre otros. Asimismo, factores químicos como el pH del material, el equilibrio o control cinético de la liberación, la formación de complejos inorgánicos u orgánicos, y las condiciones redox del material desempeñan un papel fundamental. Además, en lo que respecta al comportamiento frente a la lixiviación, se requiere una distinción según el tipo de material y/o la infraestructura a la que pertenecerá: materiales monolíticos (como aquellos basados en cemento, hormigón, ladrillos, o materiales recubiertos) y materiales granulares (tales como áridos, cenizas, escorias, etc.). En los materiales monolíticos, el proceso de lixiviación está controlado por la difusión, mientras que en los materiales granulares, la liberación está dominada por mecanismos de percolación [102]. La realización de análisis de lixiviación en proyectos de construcción de carreteras es esencial para evaluar el riesgo de contaminación y tomar medidas preventivas.

Los ensayos de lixiviación se destacan como la mejor opción para simular en entornos de laboratorio los procesos físico-químicos que ocurren bajo las condiciones de exposición que experimentarán las capas de carretera construidas con áridos reciclados o suelos estabilizados con subproductos en situaciones reales [103]. Estos ensayos pueden clasificarse en varios tipos, que permiten una comprensión más profunda y precisa de los efectos de la lixiviación en los materiales utilizados en obras viales.

- Test de conformidad (EN 12457-4: 2003): Esta prueba evalúa la lixiviación de los materiales y permite determinar su idoneidad para ser admitidos en vertederos. Evalúa la lixiviación básica y rápida de una o dos etapas en estado granular.
- Test de percolación (EN 14405:2017): Similar a la prueba de conformidad, esta prueba evalúa la lixiviación en condiciones que simulan la percolación natural en un vertedero.
- Test de Tanque o de difusión: consiste en una prueba de lixiviación para materiales monolíticos. Consiste en reproducir en el laboratorio el mecanismo de liberación que rige la liberación en materiales sólidos: la difusión superficial de especies químicas expresada en mg/kg (CEN/TS 15863:2012).

Por lo tanto, realizar un estudio exhaustivo del proceso de lixiviación y la liberación de contaminantes es esencial. Estos resultados sirven como indicadores de sostenibilidad, lo que es fundamental para demostrar la viabilidad de reutilizar subproductos industriales en proyectos de ingeniería civil. Esto se refleja en la inclusión de pruebas de lixiviación, como la prueba de conformidad y el test de percolación, en la presente Tesis Doctoral. Los resultados de estas pruebas juegan un papel decisivo en la toma de decisiones sobre la utilización de dichos materiales.

Dado que numerosos factores influyen en el proceso de lixiviación, la elección del tipo de prueba es esencial. Omitir estos factores puede dar lugar a resultados que no se pueden extrapolar a situaciones del mundo real. Por lo tanto, es imperativo definir adecuadamente el escenario de lixiviación.

Es importante destacar que las pruebas de lixiviación se llevan a cabo en una amplia variedad de materiales para diversos propósitos, como regulación, gestión de residuos, evaluación de impacto ambiental y fines científicos. Debido a la creciente preocupación por el impacto ambiental de las actividades humanas, las pruebas de lixiviación se han convertido en una parte fundamental de la gestión de residuos y subproductos. Además, es relevante mencionar que, a lo largo de los años, los investigadores han observado similitudes en el comportamiento de los materiales frente a la lixiviación, incluso cuando tienen diferentes naturalezas o composiciones. [104].

La Decisión del Consejo Europeo [105], de 19 de diciembre de 2002, por la que se establecen los criterios y procedimientos de admisión de residuos en los vertederos con arreglo al artículo 16 y al anexo II de la Directiva 1999/31/CEE, se distinguen tres tipos de vertederos: vertederos para residuos inertes, para no peligrosos y para residuos peligrosos.

Para la admisión de residuos en los vertederos hay que realizar una primera caracterización básica sobre el residuo (liberación de elementos peligrosos al lixiviado). La concentración de las especies químicas críticas desde un punto de vista ambiental, se miden sobre el lixiviado obtenido mediante una prueba o test de conformidad. En base a los resultados obtenidos por dicho ensayo, se obtendrá el nivel de liberación del material y, por tanto, se determinará la clase de vertedero en el que el residuo se considera admisible.

En cuanto a las especies químicas reguladas por la normativa vigente por ser considerados potencialmente peligrosos para el medio ambiente, son las siguientes: As, Ba, Cd, Cr, Cu, Hg, Mo, Ni, Pb, Sb, Se, Zn, sulfatos, cloruros y fluoruros. Así pues, dicha normativa indica la concentración máxima (expresada en mg/kg para el test de conformidad y en mg/l para el test de percolación) de cada uno de esos elementos, para cada uno de los tres tipos de vertederos: los de residuos inertes, los de no peligrosos y los de peligrosos. Estos niveles se calcularán, en términos de liberación total, para las proporciones entre líquido y sólido (L/S) de 10 l/kg y de 0.1 l/kg y una vez medidos los niveles liberados en el lixiviado de cada material, se compararán con los valores límite de concentración que se indican en la Tabla 1.4. para poder clasificar el material según su potencial contaminante.

Tabla 1. 4. Clasificación de peligrosidad de los residuos en función de la cantidad lixiviado para admisión a vertedero, de acuerdo con los test de conformidad (L/S=10l/kg) y test de percolación (L/S=0.1l/kg)

Elementos	***Residuo Inerte***		***Residuo No Peligroso***		***Residuo Peligroso***	
	Test de conformidad (mg/kg)	*Test de percolación (mg/l)*	*Test de conformidad (mg/kg)*	*Test de percolación (mg/l)*	*Test de conformidad (mg/kg)*	*Test de percolación (mg/l)*
Cr	0.5	0.1	10	2.5	70	15
Ni	0.4	0.12	10	3	40	12
Cu	2	0.6	50	30	100	60
Zn	4	1.2	50	15	200	60
As	0.5	0.06	2	0.3	25	3
Se	0.1	0.04	0.5	0.2	7	3
Mo	0.5	0.2	10	3.5	30	10
Cd	0.04	0.02	1	0.3	5	1.7
Sb	0.06	0.1	0.7	0.15	5	1
Ba	20	4	100	20	300	60
Hg	0.01	0.002	0.2	0.03	2	0.3
Pb	0.5	0.15	10	3	50	15

Ante la carencia de normativa y procedimiento de evaluación ambiental a nivel nacional, se recurran a las metodologías aprobadas por la Comisión Europea (v.g. test de cumplimiento UNE-EN 12457-3)

1.7.2.- Análisis de ciclo de vida

El Análisis de Ciclo de Vida (ACV) es un enfoque objetivo que nos permite evaluar las implicaciones ambientales asociadas a un producto, proceso o actividad. Su objetivo es identificar y cuantificar tanto el consumo de recursos, como la energía, y las emisiones al entorno, con el propósito de determinar el impacto resultante de estos recursos y emisiones. El ACV desempeña un papel fundamental en la evaluación y la implementación de estrategias de mejora ambiental [106].

Este enfoque abarca todo el ciclo de vida de un producto, proceso o actividad. Esto implica considerar todas las etapas, desde la extracción y el procesamiento de materias primas, la producción, el transporte y la distribución, hasta su uso, posibles fases de reutilización y mantenimiento, el reciclaje y, por último, la disposición final. El ACV es una herramienta crucial en la evaluación ambiental de obras de carretera. Se trata de un enfoque sistemático que evalúa los impactos ambientales asociados con un producto, proceso o actividad a lo largo de su ciclo de vida completo, desde la extracción de materias primas hasta su disposición final.

En el caso de las carreteras, el ACV permite evaluar los impactos ambientales relacionados con la producción de materiales, construcción, mantenimiento y eventual demolición de la infraestructura. Estos impactos pueden abarcar aspectos como el consumo de energía, las emisiones de gases de efecto invernadero, la generación de residuos y la degradación del suelo.

El ACV se basa en varias etapas, que incluyen la definición de los objetivos y alcance del estudio, la recopilación de datos pertinentes, la evaluación de impacto y la interpretación de resultados. Los datos recolectados pueden incluir información sobre el consumo de energía, emisiones de gases contaminantes, uso de recursos naturales, entre otros.

Al integrar el ACV en la evaluación ambiental de una obra de carretera, se pueden identificar áreas clave para mejorar la sostenibilidad del proyecto. Esto incluye la optimización de materiales y procesos para reducir la huella ambiental en todas las etapas de vida de la carretera.

1.7.2.1.-Evolución histórica y marco normativo del ACV

Los orígenes del ACV se remontan a la década de los años 70 del siglo pasado. En ese momento, Hunt et al. [107] presentaron los resultados de su estudio en forma de perfiles de recursos y emisiones, aunque todavía no se realizó un análisis cuantitativo de los impactos ambientales asociados a estos. Los primeros métodos para evaluar los impactos ambientales en el contexto del ACV se publicaron a principios de la década de 1990, destacando metodologías como Swiss Ecoscarcity (o Ecopoints) [108] y la metodología CML 1992 [109].

En ese mismo período, en 1993, se inició el proceso de estandarización para el ACV bajo los auspicios de la Organización Internacional de Normalización (ISO). Este proceso culminó en 2006 con la publicación de las normas ISO 14040 y ISO 14044, que constituyen el marco normativo vigente. Paralelamente, se llevaron a cabo proyectos específicos en varios países para desarrollar metodologías destinadas a evaluar los impactos del ciclo de vida. Estas metodologías incluyen:

1. Métodos midpoint, como EDIP y CML 2002 [110].
2. Métodos endpoint, como Ecoindicator 99 y EPS [111]
3. Métodos que combinan enfoques de midpoint y endpoint, como IMPACT 2002+ [112], LIME [113], y ReCiPe [111].

En la actualidad, las normas ISO 14040 y ISO 14044, junto con la norma ISO 15804, establecen el marco normativo para evaluar y cuantificar los impactos ambientales de materiales y productos de construcción. [114, 115].

El ACV puede abordar la etapa de producto (desde la obtención de materias primas hasta la fabricación), conocida como "de la cuna a la puerta" Alternativamente, puede considerar todo el ciclo de vida de un producto según los límites del sistema (etapa de producto, construcción, uso y fin de vida), denominándose "de la cuna a la tumba" [116, 117].

1.7.2.2.- Metodología del Análisis de Ciclo de Vida (ACV)

La metodología del ACV comprende varias etapas clave:

Definición de objetivos y alcance: Se establecen los objetivos del estudio y se determinan los límites del sistema, identificando las etapas del ciclo de vida que se incluirán.

- **Análisis de inventario:** Se recopilan datos sobre las entradas y salidas de cada etapa del ciclo de vida de la carretera, como el consumo de energía, emisiones de gases contaminantes, uso de recursos naturales y generación de residuos.
- **Evaluación de impacto:** Se evalúan los impactos ambientales asociados con los datos recopilados, utilizando diferentes indicadores de impacto, como el potencial de calentamiento global, la acidificación, la eutrofización, entre otros.
- **Interpretación de resultados:** Se interpretan los resultados para identificar las etapas del ciclo de vida que contribuyen significativamente a los impactos ambientales y para proponer mejoras.

En el contexto de las obras de carretera, el ACV se aplica para evaluar y comparar diferentes opciones de diseño, materiales y técnicas constructivas. Esto incluye la evaluación de:

- **Materiales de construcción**: Comparación de materiales utilizados en la construcción de la carretera, teniendo en cuenta su extracción, procesamiento, transporte y disposición final.
- **Técnicas de construcción**: Evaluación de técnicas constructivas para minimizar el consumo de energía, las emisiones y los residuos generados durante la construcción.
- **Mantenimiento y vida útil**: Consideración de la durabilidad y los requisitos de mantenimiento de la carretera a lo largo de su vida útil.

En la presente Tesis Doctoral, aplica la metodología de ACV para cuantificar la reducción de afección al medio derivada de la aplicación de residuos y subproductos, centrándose en las etapas de producción de materiales y procesos constructivos y puesta en obra.

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CAPÍTULO II

OBJETIVOS

OBJETIVOS

El **objetivo principal,** que aborda la presente Tesis Doctoral, es el estudio de la posible aplicación de residuos y subproductos industriales, tales como áridos reciclados provenientes de residuos de construcción y demolición, cenizas volantes y de fondo provenientes de la quema de biomasa y escoria blanca de acería, procedente de la industria productora de acero y nanomateriales de última generación, para su aplicación en la construcción de capas de carreteras para vías de baja intensidad.

Para la consecución del objetivo principal planteado, es necesario discretizar el objetivo principal en objetivos secundarios que garanticen la correcta evolución y desarrollo de la Tesis Doctoral. Los objetivos secundarios planteados son los siguientes:

Objetivo secundario 1. Estudio de las propiedades físicas, químicas y mineralógicas de los materiales implicados en la investigación. Se caracteriza en profundidad los residuos, subproductos, nanomateriales y suelos a estudiar, analizando así la influencia de cada uno de los parámetros en el desempeño estructural de las capas de carretera ejecutadas. Se realizan ensayos de caracterización física (granulometría, densidad y absorción, coeficiente de los ángeles, etc.), caracterización química (composición elemental, contenido en materia orgánica, contenido en sulfatos, etc.) y caracterización mineralógica (difracción de rayos X y microscopía SEM).

Objetivo secundario 2. Evaluación de los efectos de los distintos tratamientos en planta aplicados a los áridos reciclados mixtos para su aplicación como zahorra compactada en capa de carreteras.

Objetivo secundario 3. Análisis de las propiedades geotécnicas y mecánicas de suelos estabilizados mediante la aplicación de residuos, subproductos y/o nanomateriales. Se realiza una campaña experimental en laboratorio que incluye ensayos geotécnicos (límites de plasticidad, hinchamiento libre, potencial porcentual de colapso, ensayos en célula triaxial) y ensayos de comportamiento mecánico (propiedades de compactación, índice CBR y resistencia a compresión simple).

Objetivo secundario 4. Estudiar las posibles consecuencias medioambientales que puede conllevar la aplicación de residuos y subproductos industriales en capas de carreteras.

Para ello se realizan ensayos de lixiviación, tanto de los residuos y subproductos como de los suelos estabilizados, desarrollos para la determinación de la posible liberación de metales pesados en un medio lluvioso.

Se realizan diferentes ensayos de lixiviación:

- Test de Conformidad: ensayo de lixiviación básico en el cual el material se encuentra en estado granular

- Test de Columna o de Percolación: ensayo para materiales granulares con la diferencia que en este caso el ensayo tiene capacidad de analizar el comportamiento similar al producido en condiciones naturales de precipitación.

Objetivo secundario 5. Análisis de ciclo de vida de la de la ejecución bases de carretera con áridos reciclados mixtos y subbases de carreteras a partir de suelos estabilizados in situ con residuos, subproductos y/o nanomateriales. Permitiendo así cuantificar las mejoras ambientales, medida en términos de reducción de emisiones de CO_2 (entre otros parámetros) debidas a la aplicación de estos materiales frente a materiales convencionales.

Objetivo secundario 6. Construcción y auscultación de un tramo a escala real de carretera, para ser usado como vía de baja intensidad, aplicando una solución novedosa de doble estabilización de suelos, aplicando nanomateriales. El tramo se ha ejecutado aplicando métodos y maquinaria convencional de construcción de carreteras. Además, una campaña de auscultación a medio y largo plazo es llevada a cabo para analizar el desempeño estructural del tramo ejecutado a lo largo del tiempo, expuesto a condiciones ambientales y de tráfico reales.

CAPÍTULO III

METODOLOGÍA

III

METODOLOGÍA

El Capítulo I tiene como objetivo contextualizar el estado actual técnico, ambiental y social en el marco de la ingeniería civil y la generación y gestión de residuos y subproductos a nivel industrial. Como resultado del estudio mostrado en el Capítulo I, se establecen las bases para el desarrollo procedimental de la presente Tesis Doctoral, el cual se muestra de modo esquemático en la Figura 3.1.

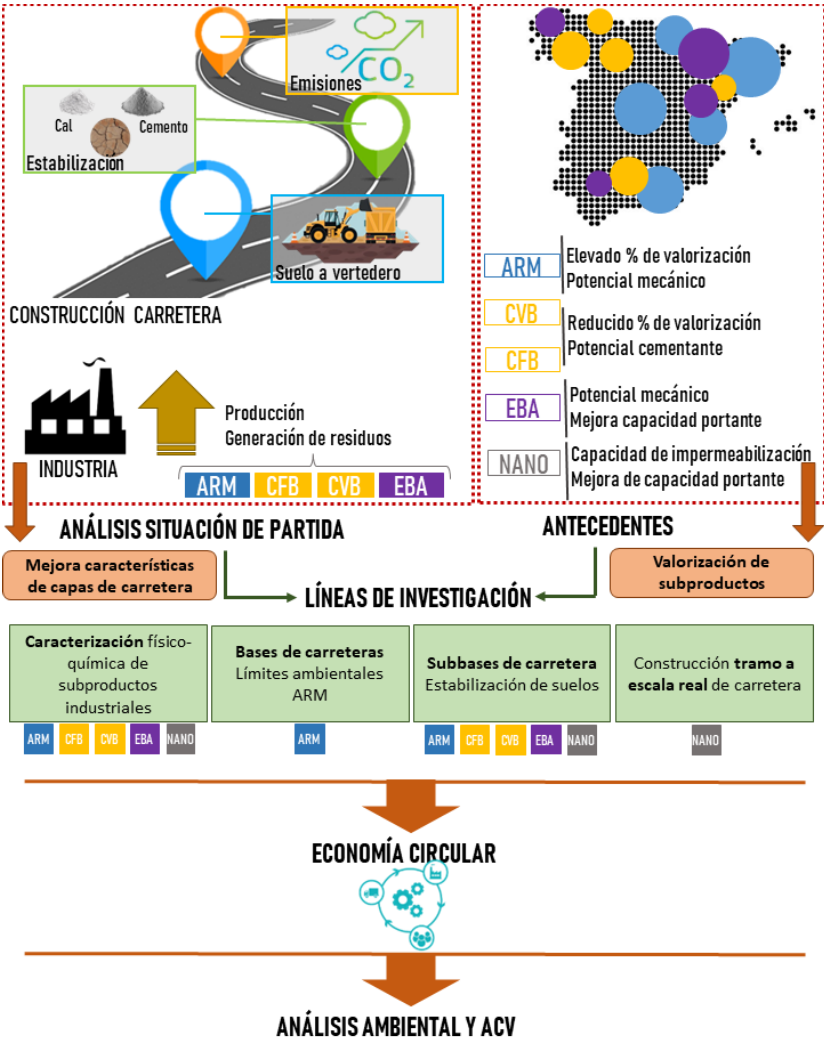


Fig. 3. 1. Esquema procedimental de la Tesis Doctoral

Tal y como se muestra en la Figura 3.1, tras analizar la situación actual relativa a la construcción de carreteras y generación y gestión de residuos y subproductos industriales y los antecedentes técnicos y ambientales, se establecen las Líneas de Investigación a seguir durante el desarrollo de la presente Tesis Doctoral.

En primer lugar, se establecen cuatro líneas de investigación orientadas a fomentar la economía circular en la construcción de carreteras. Estas líneas de investigación son:

- Conocimiento profundo de las propiedades físicas, químicas y mineralógicas de los subproductos industriales mediante ensayos de caracterización.
- Conocimiento de la influencia del procesamiento en planta de los áridos reciclados en su impacto ambiental en la ejecución de bases de carreteras.
- Estudio de los subproductos industriales y combinación de subproductos y nanomateriales como nuevos materiales estabilizadores de suelos arcillosos expansivos, para la ejecución de subbases de carreteras.
- Ejecución de un tramo a escala real de carretera mediante la aplicación de nanomateriales. Validación del estado del conocimiento en condiciones ambientales reales.

Tras finalizar el estudio de las cuatro líneas de investigación principales, se obtiene un conocimiento técnico preciso de la viabilidad de aplicación de subproductos y materiales reciclados para la ejecución de capas de carreteras.

Sin embargo, de acuerdo con lo mostrado en el Capítulo I, la necesidad de cuantificar los impactos ambientales derivados de la construcción de carreteras implica una línea de investigación transversal dedicada a aplicar la metodología de Análisis de Ciclo de Vida, determinando así las afecciones ambientales o reducciones de estas debidas a la sustitución de materiales convenciones por subproductos industriales. El estudio engloba desde la generación de los subproductos hasta su puesta en obra y finalización de la capa de carretera ejecutada con medios mecánicos actuales.

Tras el establecimiento de las líneas de investigación, se expone a continuación la metodología seguida durante el desarrollo de la tesis doctoral, la cual permite alcanzar los objetivos planteados en el Capítulo II.

La metodología seguida se compone de tres fases, las cuales se desarrollan durante la realización de la Tesis Doctoral. Las fases metodológicas se muestran en la figura 3.2.

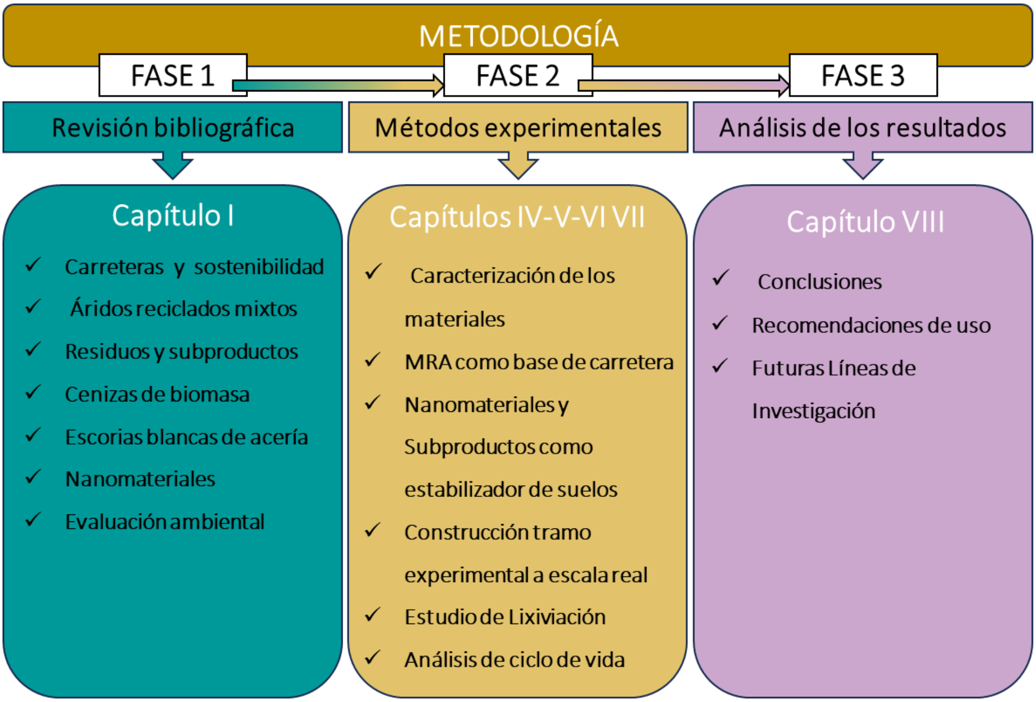


Fig. 3. 2. Esquema metodológico de la investigación

- **Fase 1. Revisión bibliográfica.**

Esta fase inicial consiste en la realización de un estudio preliminar acerca del grado de desarrollo e investigación en la aplicación de subproductos industriales y nanomateriales para su aplicación en capas de carreteras. Este estudio preliminar se realiza mediante una búsqueda bibliográfica intensiva, que tiene como resultado la redacción del Capítulo I. El conocimiento del estado del arte actual y la finalización de la fase uno permite el establecimiento de los objetivos a cumplir durante el desarrollo de la Tesis Doctoral, así como la metodología de ésta.

- **Fase 2. Métodos experimentales.**

Esta fase se centra el desarrollo experimental a realizar durante la Tesis Doctoral. Esta fase se divide en las siguientes etapas experimentales (EX).

- **EX.1**. Caracterización avanzada de los materiales involucrados en la investigación. Se realiza una caracterización física, química, mineralógica y ambiental mediante análisis de lixiviados de los residuos, subproductos y nanomateriales, con objeto de determinar su idoneidad para su aplicación en distintas capas de carreteras. Con la realización de esta Etapa Experimental se pretende alcanzar el Objetivo Secundario 1. Los resultados obtenidos para cada material se muestran en las siguientes publicaciones:

  ARM: "*Environmental assessment, mechanical behavior and new leaching impact proposal of mixed recycled aggregates to be used in road construction*"

  Nanomateriales y Subproductos (CFB, CVB y EBA): "*Feasibility of using nanosilanes in a new hybrid stabilised soil solution in rural and low-volume roads*"

  "*Geotechnical and engineering properties of expansive clayey soil stabilized with biomass ash and nanomaterials for its application in structural road layers*"

  "*Evaluation of geotechnical, mineralogical and environmental properties of clayey soil stabilized with different industrial by-products: a comparative study*"

- **EX.2.** Estudio de las propiedades de distintos ARM producidos con diferentes tratamientos en planta para su aplicación como base de carretera. De acuerdo con los resultados obtenidos en la EX.1. y evaluando las propiedades mecánicas de cada de tipo de árido, se proponen nuevos límites de aplicación para ARM, ampliando su posible aplicación como capa de carretera. Mediante el desarrollo de esta Etapa Experimental se busca alcanzar el Objetivo Secundario 2. Los resultados obtenidos se muestran en la publicación "*Environmental assessment, mechanical behavior and new leaching impact proposal of mixed recycled aggregates to be used in road construction*"

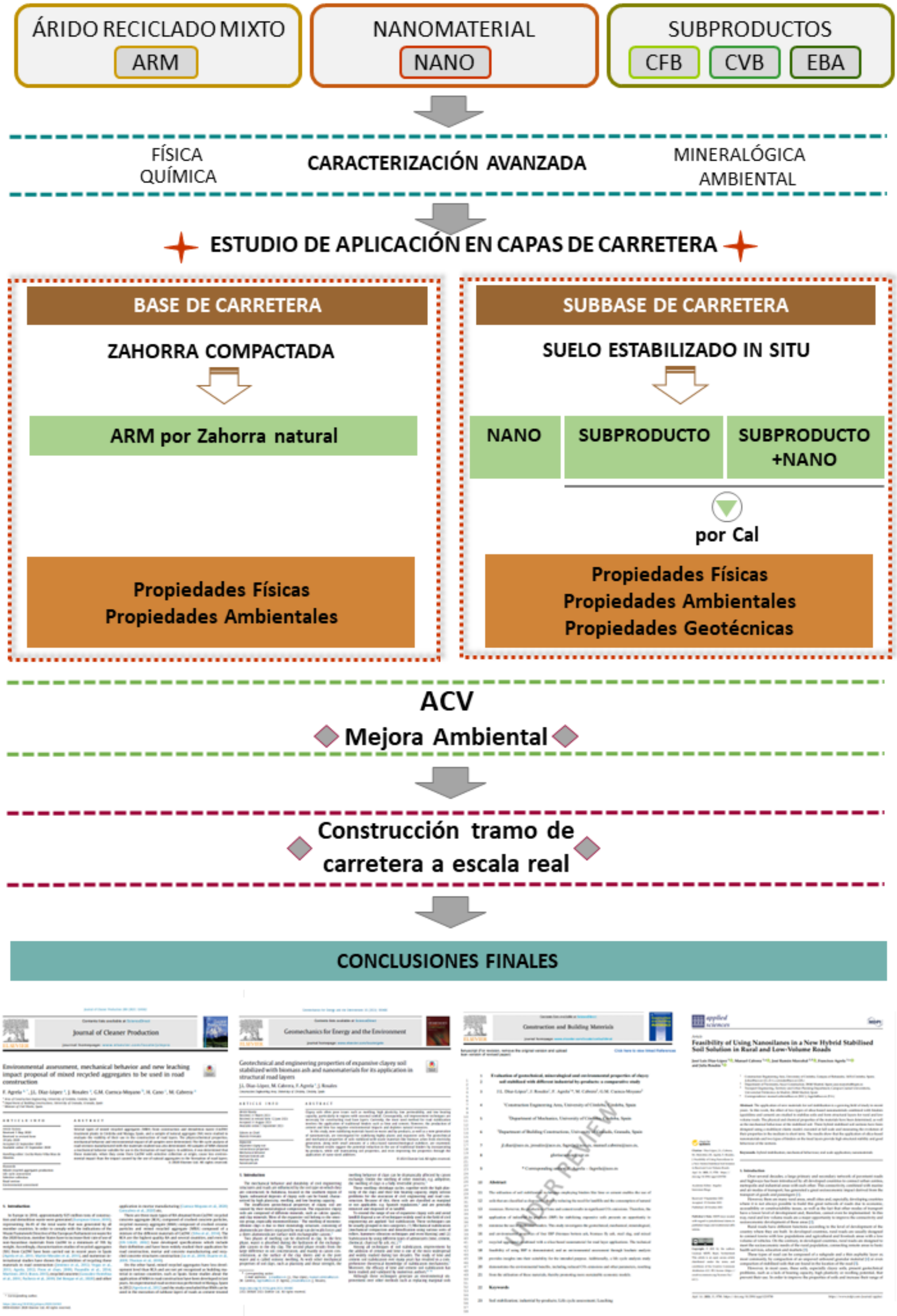


Fig. 3. 3. Esquema desarrollo experimental de la investigación realizada

- **EX.3.** Estudio del potencial estabilizador de los residuos, subproductos y nanomateriales analizados para su aplicación en arcillas expansivas. Al igual que en la Etapa Experimental anterior, considerando las propiedades físicas y químicas de los subproductos analizados se desarrolla un estudio para determinar las propiedades geotécnicas, mecánicas y mineralógicas de diferentes mezclas de los subproductos analizados con un suelo arcilloso expansivo. Además, se analiza la influencia de la combinación de nanomateriales de última generación con estos subproductos. El desarrollo de esta Etapa Experimental permite alcanzar Objetivo Secundario 3. Los resultados obtenidos se muestran en las siguientes publicaciones:

  "*Feasibility of using nanosilanes in a new hybrid stabilised soil solution in rural and low-volume roads*"

  "*Geotechnical and engineering properties of expansive clayey soil stabilized with biomass ash and nanomaterials for its application in structural road layers*"

  "*Evaluation of geotechnical, mineralogical and environmental properties of clayey soil stabilized with different industrial by-products: a comparative study*"

- **EX.4.** Estudio del impacto ambiental y análisis de ciclo de vida. En este bloque se estudia el impacto medioambiental que causa la aplicación de los subproductos industriales mediante el análisis de lixiviados de metales pesados. Además, se cuantifica la reducción de emisiones de CO_2 al medioambiente derivado de la reducción de aplicación de materiales convencionales a favor de materiales reciclados. Con este bloque se pretenden conseguir los Objetivos Secundarios 4 y 5. Los resultados obtenidos se muestran en cada una de las publicaciones en función al material alternativo analizado.

  ARM: "*Environmental assessment, mechanical behavior and new leaching impact proposal of mixed recycled aggregates to be used in road construction*"

  Nanomateriales y Subproductos (CFB, CVB y EBA): "*Evaluation of geotechnical, mineralogical and environmental properties of clayey soil stabilized with different industrial by-products: a comparative study*"

- **EX.5.** Ejecución de un tramo a escala real de carretera para su uso como vía de baja intensidad. De acuerdo con el estado del arte actual y los resultados obtenidos en la EX.1, se evalúa la influencia de la aplicación de nanomateriales en la estabilización de suelos expansivos. Se realizan estudios en laboratorio y una implementación a escala real con la construcción de un tramo de carretera. El desarrollo de esta Etapa Experimental tiene como meta la consecución de los Objetivos Secundarios 3 y 6. Los resultados obtenidos se muestran en la siguiente publicación "*Feasibility of using nanosilanes in a new hybrid stabilised soil solution in rural and low-volume roads*"

- **Fase 3. Análisis de los resultados.**

A partir del conocimiento adquirido en la Fase 1 y a la luz de los resultados obtenidos en la Fase 2, se consigue tener una visión clara acerca de la características técnicas y ambientales de aplicar materiales reciclados, subproductos industriales y nanomateriales para la conformación de capas de carreteras, permitiendo extraer así conclusiones que permiten establecer pautas y recomendaciones de uso.

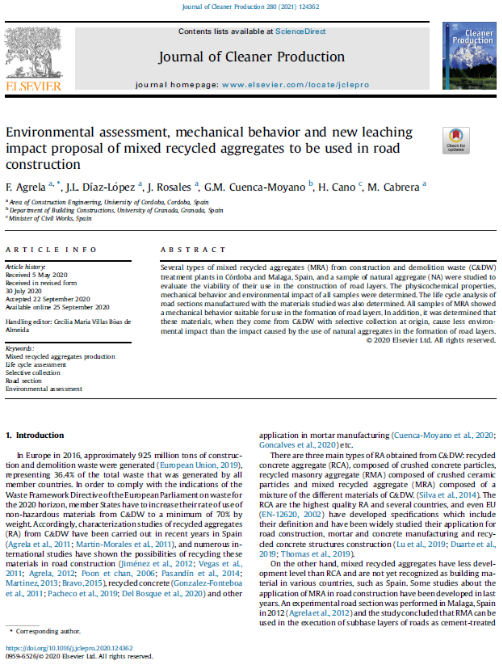
Estas conclusiones, pautas y recomendaciones de uso para la aplicación de residuos, subproductos industriales y nanomateriales en capas de carreteras fueron publicadas en diversos medios de difusión científica, como artículos en revistas científicas indexadas, publicaciones en congresos nacionales e internacionales, simposios, seminarios etc.

Esta difusión de resultados permite un avance en la materia para toda la comunidad científica, fomentado así el uso de estas nuevas tecnologías, avanzando hacia un futuro sostenible basado en modelos de economía circular.

CAPÍTULO IV

ENVIRONMENTAL ASSESSMENT, MECHANICAL BEHAVIOR AND NEW LEACHING IMPACT PROPOSAL OF MIXED RECYCLED AGGREGATES TO BE USED IN ROAD CONSTRUCTION

Agrela, F., Díaz-López, J.L., Rosales, J., Cuenca-Moyano, G.M., Cano, H., Cabrera, M.



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Environmental assessment, mechanical behavior and new leaching impact proposal of mixed recycled aggregates to be used in road construction

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ABSTRACT

Several types of mixed recycled aggregates (MRA) from construction and demolition waste (C&DW) treatment plants in Córdoba and Malaga, Spain, and a sample of natural aggregate (NA) were studied to evaluate the viability of their use in the construction of road layers. The physicochemical properties, mechanical behavior and environmental impact of all samples were determined. The life cycle analysis of road sections manufactured with the materials studied was also determined. All samples of MRA showed a mechanical behavior suitable for use in the formation of road layers. In addition, it was determined that these materials, when they come from C&DW with selective collection at origin, cause less environmental impact than the impact caused by the use of natural aggregates in the formation of road layers.

1. Introduction

In Europe in 2016, approximately 925 million tons of construction and demolition waste were generated (European Union, 2019), representing 36.4% of the total waste that was generated by all member countries. In order to comply with the indications of the Waste Framework Directive of the European Parliament on waste for the 2020 horizon, member States have to increase their rate of use of non-hazardous materials from C&DW to a minimum of 70% by weight. Accordingly, characterization studies of recycled aggregates (RA) from C&DW have been carried out in recent years in Spain (Agrela et al., 2011; Martin-Morales et al., 2011), and numerous international studies have shown the possibilities of recycling these materials in road construction (Jiménez et al., 2012; Vegas et al., 2011; Agrela, 2012; Poon et chan, 2006; Pasandín et al., 2014; Martinez, 2013; Bravo, 2015), recycled concrete (Gonzalez-Fonteboa et al., 2011; Pacheco et al., 2019; Del Bosque et al., 2020) and other application in mortar manufacturing (Cuenca-Moyano et al., 2020; Goncalves et al., 2020) etc.

There are three main types of RA obtained from C&DW: recycled concrete aggregate (RCA), composed of crushed concrete particles, recycled masonry aggregate (RMA) composed of crushed ceramic particles and mixed recycled aggregate (MRA) composed of a mixture of the different materials of C&DW. (Silva et al., 2014). The RCA are the highest quality RA and several countries, and even EU (EN-12620, 2002) have developed specifications which include their definition and have been widely studied their application for road construction, mortar and concrete manufacturing and recycled concrete structures construction (Lu et al., 2019; Duarte et al., 2019; Thomas et al., 2019).

On the other hand, mixed recycled aggregates have less development level than RCA and are not yet recognized as building material in various countries, such as Spain. Some studies about the application of MRA in road construction have been developed in last years. An experimental road section was performed in Malaga, Spain in 2012 (Agrela et al., 2012) and the study concluded that RMA can be used in the execution of subbase layers of roads as cement-treated

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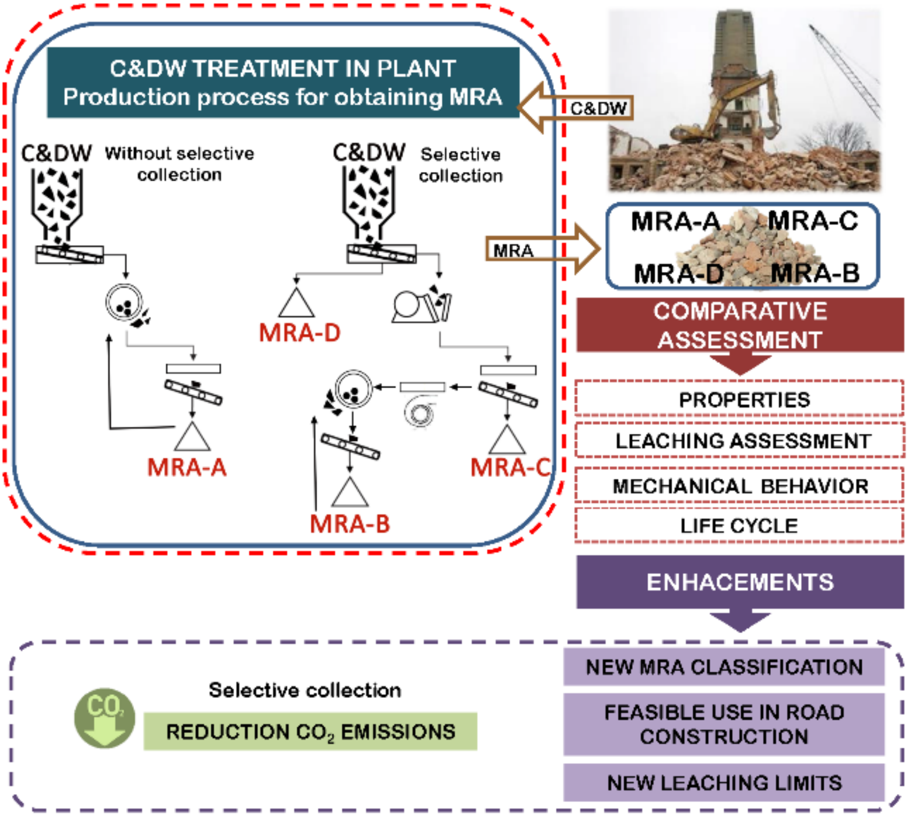
IV

ENVIRONMENTAL ASSESSMENT, MECHANICAL BEHAVIOR AND NEW LEACHING IMPACT PROPOSAL OF MIXED RECYCLED AGGREGATES TO BE USED IN ROAD CONSTRUCTION

Agrela, F., Díaz-López, J. L., Rosales, J., Cuenca-Moyano, G. M., Cano, H., & Cabrera, M. (2021). Environmental assessment, mechanical behavior and new leaching impact proposal of mixed recycled aggregates to be used in road construction. Journal of Cleaner Production, 280, 124362.



Abstract

Several types of mixed recycled aggregates (MRA) from construction and demolition waste (CDW) treatment plants in Córdoba and Malaga, Spain, and a sample of natural aggregate (NA) were studied to evaluate the viability of their use in the construction of road layers. The physicochemical properties, mechanical behavior and environmental impact of all samples were determined. The life cycle analysis of road sections manufactured with the materials studied was also determined. All samples of MRA showed a mechanical behavior suitable for use in the formation of road layers. In addition, it was determined that these materials, when they come from CDW with selective collection at origin, cause less environmental impact than the impact caused by the use of natural aggregates in the formation of road layers.

Keywords:

Mixed recycled aggregates production; Life cycle assessment; Selective collection; Road section; Environmental assessment.

4.1.- Introduction

In Europe in 2016, approximately 925 million tons of construction and demolition waste (CDW) were generated (European Union, 2019) representing 36.4% of the total waste that was generated by all member countries. In order to comply with the indications of the Waste Framework Directive of the European Parliament on waste for the 2020 horizon, member States have to increase their rate of use of recycled and treated materials from CDW to a minimum of 70% by weight.

Accordingly, studies of the properties of recycled aggregates from CDW have been carried out in recent years in Spain (Agrela et al., 2011, Martin-Morales et al., 2011,Barbudo et al., 2012a), and numerous international studies have shown the possibilities of recycling these materials in road construction (Jiménez et al., 2012, Vegas et al., 2011, Agrela, 2012; Poon and Chan, 2006; Pasandín et al., 2014, Martinez, 2013, Bravo, 2015), recycled concrete (Gonzalez-Fonteboa et al., 2011, Pacheco et al., 2019, Del Bosque et al., 2020) and other application in mortar manufacturing (Cuenca-Moyano et al., 2020, Goncalves et al., 2020).

Silva et al. (2014) classified the RA from CDW into four types according to their properties and composition. Two main types of RA were recycled concrete aggregates (RCA), mainly composed of crushed concrete particles, and mixed recycled aggregates (MRA) composed of a mixture of the different materials of CDW, crushed ceramic and asphalt particles, combined with concrete particles.

RCA present more suitable properties for its application than the remaining different types of RA. (Silva et al., 2014). The EU have developed specifications which include their definition and have widely studied their application for road construction, mortar and concrete manufacturing and recycled concrete structures construction (Lu et al., 2019, Duarte et al., 2019, Thomas et al., 2019).

Currently, studies about MRA application are less developed than RCA and implementation is scarce. Some studies about the application of MRA in road construction have been developed in recent years, such as the one carried out in an experimental road section in Malaga, Spain in 2012 (Agrela et al., 2012). This study concluded that MRA can be used in the execution of sub-base layers of roads as cement-treated materials in an amount of 3% by dry mass. Del Rey et al (2016) developed a laboratory study of cement-treated MRA in a size fraction of 0/8 mm in order to probe the use as sub-base layers for low-traffic, obtaining positives results.

Other studies have analyzed the properties of MRA in different applications, such as the analysis of the functional and structural parameters of paved roads (Tavira et al., 2018) or analysis of the most important physical properties and mechanical behavior of MRA in the geotechnical applications and construction of unpaved roads (Cardoso et al., 2016)

From an environmental point of view it is very important to characterize the leachate components of RA, in order to ensure safety in the use of these recycled materials (Van der Sloot et al., 2002). Leaching tests are focused on determining pollutant concentrations that are limited by European directive 2003/33 / EEC of landfill admission due to there being no specific regulation for the application of recycled materials in road layers. Different uses of MRA involve different problems derived from leaching, thus, manufacture of concrete is limited by sulfate concentrations (Martin-Morales et al., 2011) or road layer execution that is limited by heavy metal concentrations (Cabrera et al., 2016). Galvín et al (2014) analyzed four samples of MRA for use on roads, concluding that all samples can be classified as non-hazardous waste that is caused by high sulfate concentrations due to a composition rich in gypsum and ceramic materials.

Several studies have shown that the use of RA from CDW as a replacement for NA reduced the environmental impact of emissions generated during concrete manufacturing (Yazdanbakhsh et al., 2018) and masonry mortars (Cuenca-Moyano et al., 2019). Similarly, Mroueh et al. (2000) analyzed alternative road sections using different wastes such as crushed concrete waste, and the results showed impact reduction due to the use of RA instead of NA. Butera et al. (2015) evaluated the use of RA from CDW in road sub-base, and concluded that the impacts were less than those generated by depositing in landfill. In this sense, the use of life cycle assessment (LCA) in the construction sector can provide the development of strategies within the framework of the circular economy, which encourage the efficient use of resources by reducing the high environmental impact associated with the sector (European Commission, 2014a, b; European Parliament and Council, 2008).

In this study, physicochemical properties and the mechanical behavior of MRA were studied in order to determine its potential uses in road construction. In addition, environmental aspects were studied by the leaching analysis of the materials ensuring compliance with European landfill regulations and establishing new limits of inert material for execution of roads layers. The environmental impact of MRA and NA production and application were determined through LCA, that allow establishing the environmental potential for the use of recycled materials. This study shows that the use of MRA is a feasible and sustainable solution for road construction.

4.1.1.- Previous classification proposal of recycled aggregates

A large number of countries have now published regulations in order to increase the amount of RA from CDW in building and road construction (Goncalves et al., 2010). These regulations are adapted to the typical construction materials in each area, resulting in an RA with significant differences between its characteristics (De Brito et al., 2019a).

De Brito, Agrela and Silva (2019) presented a proposal for the international classification of six types of RA that can be used for road construction in order to simply their use. In this classification proposal, RA are classified according to their main composition and a series of physicochemical properties (composition, minimum density, water absorption, Los Angeles abrasion test and water-soluble sulfate) are determined through international standardized tests.

The classification (Table 4.1) shows six types of RA. The first type (RCA) is the highest quality that could be obtained, composed mainly of concrete particles and natural aggregates, 90-85% by mass minimum (RCA-I and RCA-II respectively).

Table 4. 1. Classification of recycled aggregates according their physicochemical properties (De Brito, Agrela and Silva, 2019)

Type of RA proposed	Composition				Minimum density (SSD)	Water absorption (%)	Los Angeles (%)	Acid-soluble sulphate (%)
	Rc+Ru (%)	Rb (%)	Ra (%)	Others (%)				
RCA-I	> 90	<10	<5	<1	2200	<6	<35	<0.7
RCA-II	> 85	<15	<10	<3	2100	<8	<37	<0.8
MRA-I	>70	<30	<10	<5	1900	<8	<40	<0.8
MRA-II	>60	<40	<20	<8	1800	<12	<45	<1.0
MRA-III	>40	<60	<30	<15	1650	<15	<50	<1.2
RAA	< 50	<10	> 50	<3	2000	<8	<40	<0.8

**In RAA these values have been changed, because in our opinion they are more coherent with the production or this type of Recycled Aggregates*

Rc: concrete and natural aggregates with adhered mortar; Ru: particles of natural materials (rocks, gravels, etc.); Rb: particles of ceramic bricks, tiles, calcium silicate masonry units, etc.; Ra: bituminous mixture particles; Others: wood, plastic, plaster, aluminum, etc.

The MRA is divided into three types (I, II and III) depending on the amounts of concrete particles, natural aggregate and rubble masonry in it. This is the most common type of RA obtained from CDW recycling plants, since these are all types of waste with varied and difficult steps to separate their composition (De Brito et al., 2019b). Finally, The RAA (Recycled Asphalt Aggregate) type is obtained from the demolition of road surface layers and is composed mainly of bituminous materials, 50 % by mass minimum.

The main composition with the properties shown in Table 4.1 classifies the type of aggregate recycled after CDW treatment in the plant. SSD density and water absorption determine the compaction properties of RA. Finally, the organic matter content and acid-soluble sulfates determine both the use of RA in the manufacture of cement-based materials and they present environmental limitations due to leaching problems.

4.2.- Materials

In this section, the production processes and physicochemical properties of MRA and artificial gravel (AG), that is a control material to establish a comparative between recycled and natural aggregates, are studied.

4.2.1.- Mixed Recycled Aggregates

Four MRA have been studied, named MRA-A, MRA-B, MRA-C and MRA-D. MRA-A. The material was produced at the plant of the Arecosur Company, Malaga. At that plant, a short treatment (Fig. 4.1) is carried out on the CDW in order to retain the particle size fraction 0/40 mm. Screening and crushing processes are applied only to CDW. The characteristics of the aggregates obtained in this process are similar to MRA-I (Table 4.1). After CDW first crushing process, 60% of MRA-A is obtained, the remaining 40% is introduced again into the crushing by impact mill.

MRA-A's high quality is due to the fact that the CDW comes from selective collection at origin, thus only clean materials are received at the plant, consisting of natural aggregate, concrete, masonry, asphalt and minimum percentages of other components.

Selective collection implies obtaining a higher quality material with less processing, which results in less predictable environment impact, as was shown in previous studies (Iriarte et al, 2009) in which life cycle assessment (LCA) was carried out in different municipal waste.

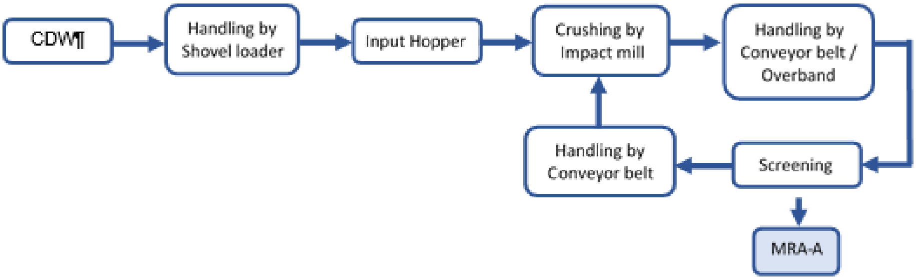


Fig. 4. 1. Production processes to obtain MRA in the treatment plant of Arecosur company

MRA-B, MRA-C, MRA-D were manufactured in the treatment plant of Gecorsa company Córdoba. These three materials do not come from the selective collection of CDW before being processed in the recycling plant, so the aggregates' production process is more complex than MRA-A. From this production process (Fig. 4.2), MRA-I, MRA-II and MRA-III (Table 4.1) are theoretically obtained. As shown in Fig. 4.2, MRA-D is obtained directly in the pre-screening process. The rest of the material retained in the sieve is crushed using a jaw-crusher and screened, through which the MRA-C is obtained. Then, the material retained in the sieves is crushed and screened, resulting in MRA-B. As there are practically no rejections, 100% of the CDW is recycled. From 1t of CDW that is treated, 0.3t of MRA-D, 0.3t of MRA-C and 0.28t of MRA-B are obtained; the remaining 0.12t are reintroduced again for crushing.

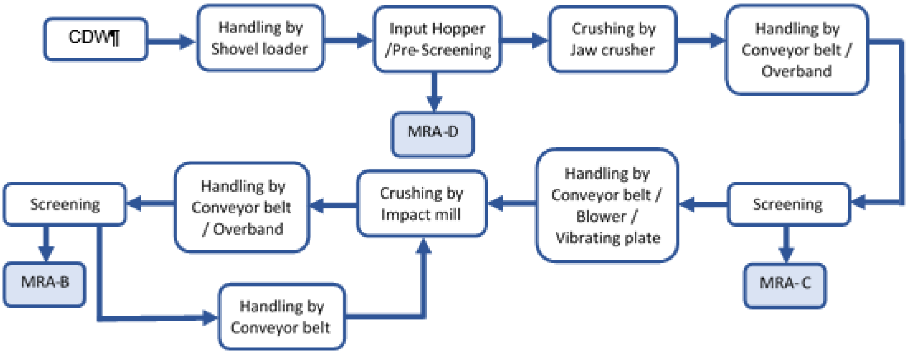


Fig. 4. 2. Production processes to obtain mixed recycled aggregates in the treatment plant of Gecorsa company

The composition of the MRA was determined by UNE-EN 12620:2003+A1:2009 standard, the following values were obtained for each material (Table 4.2).

Table 4. 2. Composition of mixed recycled aggregates

Composition	*Rc+Ru* (%)	*Rb* (%)	*Ra* (%)	*Gypsum* (%)	*Others (No gypsum) (%)*	*Assigned Classification type*
MRA-A	73.17	25.29	0.01	0.47	1.06	**MRA-I**
MRA-B	83.76	11.78	0.28	2.78	1.4	**MRA-I**
MRA-C	71.77	22.09	0.68	5.40	0.06	**MRA-II**
MRA-D	64.89	15.22	5.59	9.25	5.05	**MRA-III**

Table 4.3 shows the information concerning the properties tested in the laboratory: density and water absorption (UNE-EN 1097-6), plasticity index (UNE 103-103 and UNE 103-104), Los Angeles abrasion test (EN 1097-2), water-soluble and acid-soluble sulfate contents (UNE-EN 1744-1) and content of organic material (UNE 103204).

Table 4. 3. Physical and chemical properties of mixed recycled aggregates

PROPERTIES	*AG*	*MRA-A*	*MRA-B*	*MRA-C*	*MRA-D*	*TEST METHOD*
Water-soluble sulfate content (%SO_3)	<0.01	0.29	0.85	1.05	2.27	**UNE-EN 1744-1**
Acid-soluble sulfate content (%SO_3)	<0.01	0.31	0.97	1.28	2.9	**UNE-EN 1744-1**
Water- soluble salts (%)	<0.1	0.95	1.47	2.45	3.22	**UNE-EN 1744-1**
Organic material (%)	0.3	0.17	0.33	0.93	1.07	**UNE 103204**
Density-SSD (Mg/m^3)						**UNE-EN 1097 - 6**
0.063-4 mm	2.67	2.33	2.17	2.18	2.14	
4-31.5 mm	2.71	2.24	2.27	2.30	2.26.	
Water absorption (%)						**UNE-EN 1097 - 6**
0.063-4 mm	2.5	5.83	8.30	9.90	11.10	
4-31.5 mm	1.6	7.50	7.40	8.90	10.10	
Average	2.1	7.12	7.74	9.49	10.61	
Plasticity	Non plastic	Non plastic	Non plastic	Non plastic	Non plastic	**UNE 103103/UNE 103104**
Los Angeles abrasion	29	33	34	36	40	**UNEEN 1097 - 2**
Assigned classification type	---	**MRA-I**	**MRA-II**	**MRA-III**	**unclassifiable**	

The results of physicochemical properties (Table 4.3) show that AG has better properties than all MRA samples. Comparing the four samples of MRA, it can be observed that the acid-soluble sulfates content is the most restrictive parameter and which determines the MRA classification. That value decreases from MRA-A to MRA-D; MRA-A presented very low sulfates values and MRA-D very high values. As for the other properties, it can be observed that the four samples have acceptable values for their application in civil works.

Particle size distribution was analyzed in accordance to Spanish Standard UNE-EN 933-1. Fig. 4.3 shows the particle size distribution curves.

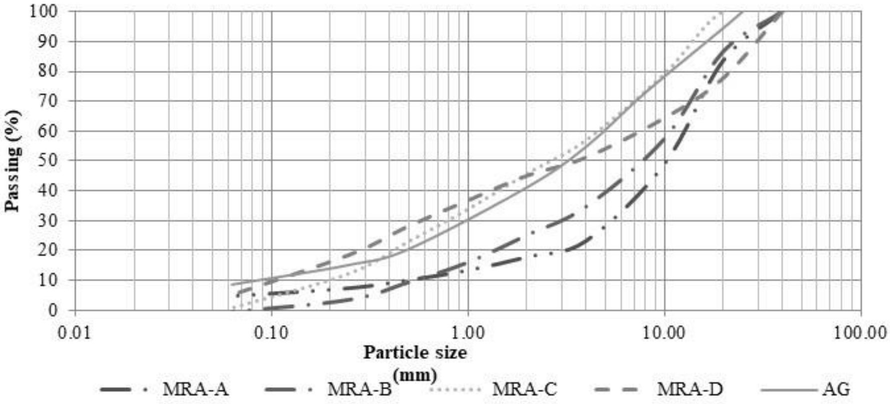


Fig. 4. 3. Particle size distribution

The particle size distribution curves of all materials are continuous, indicating more opportunities for interactions between particles and the ability to obtain a greater degree of compaction. The percentage of fine fraction (<0.063 mm) was less than 4.5%.

According to the results obtained in tests carried out on the four recycled aggregates and the parameters indicated in Table 4.1, the final classification of each material is shown in the following table (Table 4.4):

Table 4. 4. Theoretical and final classification of mixed recycled aggregates

RA	*Theoretical classification*	*Final classification*	*Reason of final classification*
MRA-A	MRA-I	MRA-I	
MRA-B	MRA-I	MRA-II	Excess sulfates limit
MRA-C	MRA-II	MRA-III	Excess sulfates limit
MRA-D	MRA-III	unclassifiable	Excess sulfates limit

4.2.2.- Artificial Gravel

AG was produced in Cerro Muriano’s quarry, located 17 km from the city of Cordoba. This quarry produces granite stone and the extraction is done by blasting (Fig. 4.4). The extracted material is transported by truck to the processing area, where after several crushing and screening processes, AG is obtained. Thus, 60% of AG is obtained from granite stone in the production process, and the remaining 40% is reintroduced into primary crushing for the production of AG.

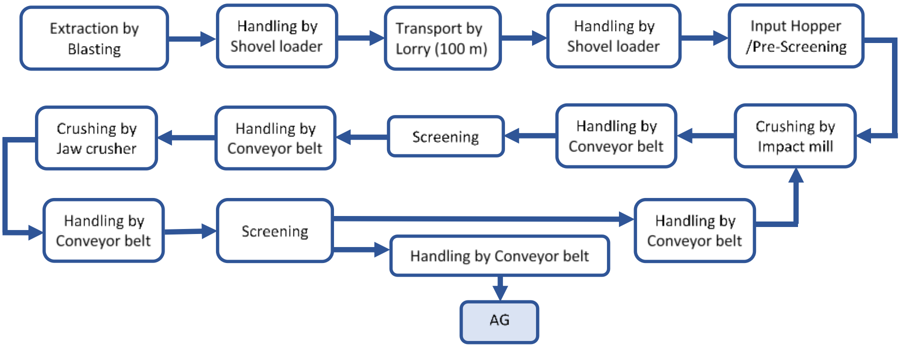


Fig. 4. 4. Production processes to obtain artificial gravel (AG)

Physicochemical properties of AG are shown in Table 4.3. AG did not have any content of sulfate and soluble salts and organic matter.

AG presented a continuous 0/20 mm fraction particle size distribution, shown in Fig. 4.3, which allows a better interaction of the particles in the formation of road layers treated with cement.

4.3.- Experimental methods and results

4.3.1.- Mechanical behavior

This section shows the results of the compaction study carried out on the materials using the modified Proctor test and bearing capacity results measured by the CBR index are shown.

4.3.1.1.- Modified Proctor (UNE 103501:1994)

The Modified Proctor test is a geotechnical laboratory test used to determine soil compaction properties, specifically to determine the optimum water content at which soil can reach its maximum dry density.

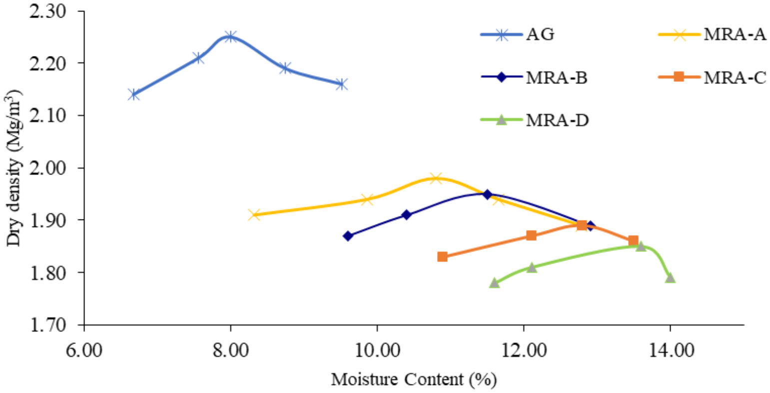


Fig. 4. 5. Moisture – density relationship

The moisture-density curves are indicative of the density sensitivity with respect to variations in the moisture content of each material (Paige-Green P, 2003). Materials with flat curves can tolerate a higher variation in moisture content without compromising much of the achieved density from compaction. However, materials with sharp curves are very sensitive to moisture changes, making it necessary to ensure that moisture content for these materials is close to the optimum value during compaction. The dry density – moisture relationship for the four MRA and the AG are shown in Fig. 4.5. In addition, maximum dry density and optimal moisture content are shown in Table 4.5.

Table 4. 5. Dry Maximum density and Optimal Moisture Content of artificial gravel and mixed recycled aggregates

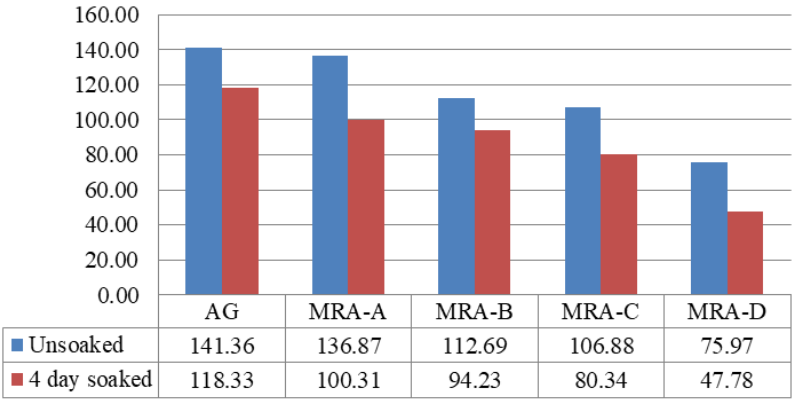
	AG	MRA-A	MRA-B	MRA-C	MRA-D
Dry Maximum Density (Mg/m^3)	2.25	1.98	1.95	1.89	1.85
Optimal Moisture Content (%)	8.1	10.8	11.5	12.8	13.6

4.3.1.2.- California Bearing Ratio (CBR) (UNE 103502:1995)

The California Bearing Ratio (CBR) test is a strength test that compares the bearing capacity of a material with that of a well-graded crushed stone It is primarily intended for, but not limited to, evaluating the strength of cohesive materials having maximum particle sizes less than 20 mm.

The basic CBR test involves applying one ratio of force per unit area required to penetrate a soil mass with standard circular piston at the rate of 1.25 mm/min. to that required for the corresponding penetration of a standard material

The CBR test was carried out in both unsoaked and 4-day soaked conditions, and the results are summarized in Fig. 4.6.



	AG	MRA-A	MRA-B	MRA-C	MRA-D
🟦 Unsoaked	141.36	136.87	112.69	106.88	75.97
🟥 4 day soaked	118.33	100.31	94.23	80.34	47.78

Fig. 4. 6. Results from the CBR index test

4.3.2.- Environmental assessment process

In this section, environmental assessment process is carried out through the analysis of the leachates generated by the MRA and the AG. For this purpose, compliance testing and percolation testing are carried out.

4.3.2.1.- Compliance test UNE EN 12457- 4: 2004

Compliance test was conducted to check whether all four MRA satisfy European regulations. To classify these materials according to the EU

Landfill Directive, not only heavy metals but also inorganic anions were measured (sulfates content). The UNE-EN 12457-3 procedure consists of a two-step batch leaching test and is performed on materials with particle-size dimensions of less than 10 mm.

Table 4. 6. pH, Temperature and conductivity of mixed recycle aggregates

RA	*pH*	*Temperature (°C)*	*Conductivity (µS/cm)*
MRA-A	11.14	20.3	1074
MRA-B	10.6	20.2	1966
MRA-C	10.7	20.3	2350
MRA-D	10.5	20.4	1465

Galvín et al. (2012) studied the main factors that affected the release of pollutants by recycled aggregates, reporting that the most relevant are: the solid liquid ratio, the contact time of the water with the material, and the pH value (being this last factor the most influential of the three in the release of polluting elements).

The results obtained from the compliance test are shown in Table 4.7. The acceptance criteria used are also indicated. Based on the results of compliance testing, the materials are classified as non-hazardous material.

Table 4. 7. Leached concentration of artificial gravel and mixed recycled aggregates by UNE-EN 12457-4 and acceptance criteria (EU Landfill Directive 2003/33/EC) (mg/kg)

	AG	*MRA-A*	*MRA-B*	*MRA-C*	*MRA-D*	*Inert Limit*	*Non-hazardous limit*
Cr	0.01489	0.0651	0.142	0.091	0.0054	< 0.5	<10
Ni	0.00767	0.0055	0.0212	0.0062	0.0237	< 0.4	<10
Cu	0.0041	0.0577	0.2831	0	0.0162	< 2	< 50
Zn	0.02612	0.0236	0.0583	0.0311	0.0697	< 4	< 50
As	0.01522	0.0521	0.0135	0.0105	0.0258	< 0.5	< 2
Se	0	0.0041	0.0177	0.0011	0.0218	< 0.1	< 0.5
Mo	0	0.0306	0.1405	0.0547	0.0792	< 0.5	< 10
Cd	0.00022	0	0	0	0	< 0.04	< 1
Sb	0.00139	0.0145	0.0237	0.0175	0.018	< 0.06	< 0.7
Ba	0.04019	0.2155	0.3799	0.1934	0.3144	< 20	< 100
Hg	0	0	0	0	0	< 0.01	< 0.2
Pb	0.00022	0	0	0.0053	0	< 0.5	< 10
SO_4	110	2564	8229	9675	12201	<6000*	<20000

*The actually sulfate content limit is 1000 mg/kg, but even if these values are exceeded, concentrations below 6000 mg/kg with a L/S ratio = 10 l/kg may be considered inert provided that the percolation values in column C_0 with a L/S ratio = 0.1 l/kg are less than 1500 mg /l.

4.3.2.2.- Percolation Test CEN/TS 14405

The Column LeachingTest (CEN/TS 14405) describes a procedure to determine the leachability of inorganic components from solid earthy and stone materials and waste as a function of the value of L/S. The method involves passing demineralized water upward through a vertical column of particulate material (4 mm or smaller).

This test allows the quantification of the retention in the matrix, simulating the release progress of a contaminant during the second life-cycle of the material (Van der Sloot et al., 1996). The total test duration is approximately 21 days. However, laboratory results do not translate directly to field conditions because of factors such as temperature, channelling, degree and duration of contact with water, ageing effects (carbonation) and others (Heasman et al., 1997) and (Van der Sloot, 2000).

Due to the intended application of the materials in civil engineering works (located outdoors and subjected closely to environmental phenomena), it is necessary that the laboratory study for leaching characterization simulates the effect of rain episodes percolating through the granular material and takes place in engineering applications in which this type of material has proven to be suitable and feasible (Galvin et al., 2014).

The results obtained from the performance of the percolation leach test are shown in Table 4.8 and the acceptance criteria used is also indicated.

Table 4. 8. concentration of artificial gravel and mixed recycled aggregates by CEN/TS 14405:2004and acceptance criteria (EU Landfill Directive 2003/33/EC) (mg/l)

	MRA-A	*MRA-B*	*MRA-C*	*MRA-D*	*Inert Limit*	*Non-hazardous limit*
Cr	0.036198	0.029735	0.00544	0.009213	<0.1	< 2.5
Ni	0.004682	0.003479	0.020466	0.033791	<0.12	<3
Cu	0.018887	0.033913	0.111085	0.313888	<0.6	< 30
Zn	0.002552	0.06129	0.332584	0.55472	<1.2	< 15
As	0.003088	0.006362	0.017133	0.02552	<0.06	< 0.3
Se	0.01342	0.011317	0.007815	0.013882	<0.04	< 0.2
Mo	0.158797	0.156013	0.093504	0.164493	<0.2	< 3.5
Cd	0	0	0.000134	0.000306	<0.02	<0.3
Sb	0.002769	0.006072	0.00701	0.010568	<0.1	<0.15
Ba	0.050327	0.04818	0.077593	0.111165	<4	< 20
Hg	0.000014	0	0	0.000073	<0.002	<0.03
Pb	0.010805	0.006381	0.009228	0.013752	<0.15	< 3
SO_4	1175.6	1591.1	1601.2	1958.42	<1500	<8500

4.3.3.- Life cycle assessment

In this section, the LCA was applied to the production processes of the aggregates studied (AG, MRA-A, MRA-B, MRA-C and MRA-D). In addition, the use of these aggregates as unbound granular sub-base layer was evaluated using LCA.

4.3.3.1. Methodology

Life cycle assessment (LCA) allows quantifying the environmental impacts of a product, process or system throughout its life cycle. According to ISO 14040 (2006) and ISO 14044 (2006), the LCA application is based on four stages: definition of the goal and scope, inventory analysis, life cycle impact assessment and interpretation of results. In this LCA study the first two stages are included in this section, while the remaining two are collected in “Life cycle impact assessment” and “Discussion” sections.

5.3.3.1.1. Goal and scope definition

This LCA study was developed to determine and compare the environmental impact associated with the production of AG and the MRA studied (MRA-A, MRA-B, MRA-C and MRA-D). In addition, in order to determine the environmental loads derived from the use of these aggregates as unbound granular sub-base layer, the LCA of the construction of four road sections was carried out. In this case, MRA-D was not used due to the high sulfate content in its composition.

The functional unit (FU) was 1km long of a road section of 8m wide, with a thickness of 25 cm for the sections executed with AG, MRA-A and MRA-B, and a thickness of 30 cm for the MRA-C section, and for the aggregate production it was considered 1t.

Fig. 4.7 shows system boundaries for AG road section (Fig. 4.7a) and for MRA road section (Fig. 4.7b) whose characteristics are listed in Table 4.9. The system boundaries include raw material production (aggregates and water), transport and road section construction; that is, the LCA is limited from cradle to gate. In details, the system boundaries include the following steps:

- The aggregate production, according to the defined processes related in paragraphs 3.1 and 3.2 for the MRA (Fig. 4.1 and Fig. 4.2), and AG (Fig. 4.3);
- The avoided production of AG when MRA is used, since the use of MRA from recycling CDW involves the incorporation of waste as secondary raw materials, which avoids the consumption of natural resources. The replacement of AG by MRA was performed in the ratio 1: 1 by volume;
- Transportation of aggregates from their place of production to the road construction site;
- The supply of tap water and its transport to the road construction site;
- The construction of road section, that is the unbound granular sub-base layer, through the activities of extended, compacted and irrigation. The preparation of the support base was not part of the study.

The LCA was performed using SimaPro 8.0.2. The impact assessment was carried out according to the categories recommended by EN 15804 + A1 (CEN 2013) regarding the sustainability of construction sites for construction products and services. These impact categories are: abiotic depletion of elements (ADe), abiotic depletion of fossil fuels (ADf), global warming (GW), ozone layer depletion (ODP), photochemical oxidation (POF), acidification of soil and water (A) and eutrophication (E). For these categories, the characterization factors of the CML-IA method were used as established in EN 15804 + A1 (CEN 2013).

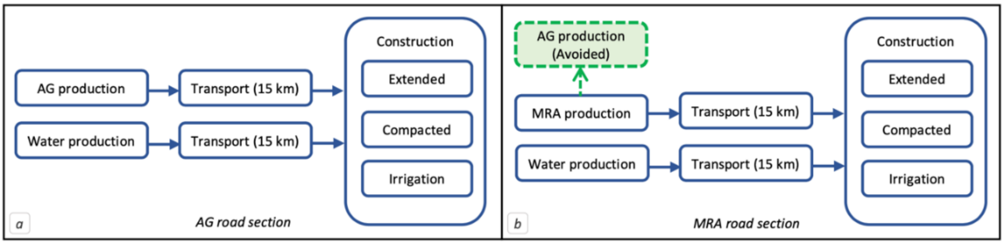


Fig. 4. 7. System boundaries

Table 4. 9. Characteristics of road sections and dosages per functional unit (1 km)

Road section	Aggregates (t)				Water (t)	Thickness	Proctor Density	Moisture
	AG	MRA-A	MRA-B	MRA-C		(m)	(t/m^3)	(%)
AG	4500	-	-	-	360	0.25	2.25	8.1
MRA-A	-	3960	-	-	427.68	0.25	1.98	10.8
MRA-B	-	-	3990	-	448.5	0.25	1.95	11.5
MRA-C	-	-	-	4536	580.608	0.30	1.89	12.8

The data collected during the inventory phase were loaded into the SimaPro 8.0.2 software (PRé Consultants 2014) and processed using the CML-IA method (Guinée et al. 2002). Initially, the environmental loads generated during the production of AG and recycled aggregates (MRA-A, MRA-B, MRA-C and MRA-D) were determined, in order to identify the processes that contribute to greater measure of impact. Subsequently, for the GW category four road sections were evaluated in which the aggregates studied were used in the construction of unbound granular sub-base layer.

4.3.3.2. Inventory

In this stage, a detailed collection of the inputs (raw materials and energy) and outputs (products, coproducts, waste, and emissions to air, soil and water) for all the processes of the system were taken into account. Primary data were site-specific and collected through interviews with producers related to average production. Ecoinvent v.3.01 database (allocation) (Ecoinvent, 2014) was used as secondary data for generic materials, energy and transport. Data quality requirements, including temporal, geographical and technological representativeness were guaranteed due to the modification of the Ecoinvent processes data according to the information provided by the producers and technology implemented in Spain.

The characteristics of the aggregates production equipment and the construction of the road section are listed in Table 4.10.

Table 4. 10. Features of the equipment

Process		Equipment	Amount	Power (kW)	Production (t/h)	Diesel consumption (MJ/t)	Distance (km)
AG							
	Handling	Shovel loader	2	-	30.518	-	-
	Transport	Lorry 28 t	1	-	-	-	0.1
	Handling	Conveyor belt, 15 m	3	8	112.61	-	-
		Conveyor belt, 25 m	2	20	168.92	-	-
	Screening	Vibrating screen	3	18.5	225	-	-
	Crushing	Impact mill	1	125.12	400	-	-
		Jaw crusher	1	206.08	400	-	-
MRA-A							
	Handling	Shovel loader	1	-	30.518	-	-
	Crushing	Impact mill	1	75	250	-	-
	Handling	Conveyor belt, 5 m	3	4	106.8	-	-
		Overband	1	3.68	108.91	-	-
	Screening	Vibrating screen	1	22.08	250	-	-
MRA-B/C/D							
	Handling	Shovel loader	1	-	30.518	-	-
	Screening	Vibrating screen	3	22.08	250	-	-
		Vibrating plate	1	3	80	-	-
	Crushing	Jaw crusher	1	160	325	-	-
		Impact mill	1	75	250	-	-
	Handling	Conveyor belt, 5 m	3	4	108.9	-	-
		Conveyor belt, 10 m	1	7.36	108.9	-	-
		Conveyor belt, 15 m	1	7.36	148.51	-	-
		Overband	2	3.68	108.91	-	-
		Blower	1	14	144.74	-	-
Road section construction	Aggregates distribution and transport	Shovelloader	1	-	-	5.551	-
		Lorry	1	-	-	-	17
	Water distribution and transport	Lorry	1	-	-	-	0.1
	Extended	Shovel loader	1	-	-	5.551	-
		Motor grader	1	-	-	1.806	-
	Compacted	Compactor	1	-	-	1.87	-
	Irrigation	Irrigation tank truck	1	-	-	33.034	-

4.3.3.3.- Life cycle impact assessment

First, the evaluation of the life cycle impact assessment of the aggregates (AG, MRA-A, MRA-B, MRA-C and MRA-D) is included, and subsequently, for a theoretical real-scale application of the aggregates in the construction of road sections.

4.3.3.3.1. Characterization results of aggregates

Table 4.11 lists the results of characterization for the production of 1tof aggregates, as well as the percentage of variation (in brackets) of the MRA with respect to AG. Also, the results in relation to the highest value are shown comparatively in Fig. 4.8. The highest impact values in all categories were generated in the production of AG. In contrast, the lowest impact values were found in the production of MRA-D, which with respect to AG decreased from 83.3% for the ODP category to 95.9% for category E. Comparatively, the variation in impact between AG and MRA were higher than 85% in categories E, A and ADe, and higher than 32% in categories GW, ADf, ODP and POF. Mainly, these impacts were caused during the blasting extraction process as well as the consumption of natural resources (granite) for the production of AG.

With respect to the production of MRA, MRA-B generated the highest impact values, followed by MRA-C, MRA-A and MRA-D, which could be due to the higher number of processes involved in its production.

Table 4. 11. Characterization results of aggregates (per 1t)

Impact category	Units	AG	MRA-A	(Δ%)	MRA-B	(Δ%)	MRA-C	(Δ%)	MRA-D	(Δ%)
ADe	kg Sb eq.	5.51E-06	4.65E-07	-91.6	8.52E-07	-84.6	5.00E-07	-90.9	2.98E-07	-94.6
ADf	MJ	3.16E+01	1.01E+01	-68.1	1.78E+01	-43.7	1.21E+01	-61.6	4.45E+00	-85.9
GW	kg CO_2 eq.	2.57E+00	7.10E-01	-72.4	1.25E+00	-51.5	8.55E-01	-66.7	3.20E-01	-87.6
ODP	kg CFC-11 eq.	1.39E-07	5.04E-08	-63.8	9.47E-08	-32.1	6.22E-08	-55.4	2.32E-08	-83.3
POF	kg C_2H_4 eq.	8.98E-04	1.73E-04	-80.8	3.04E-04	-66.1	2.02E-04	-77.5	6.35E-05	-92.9
A	kg SO_2 eq.	5.13E-02	4.59E-03	-91.1	7.96E-03	-84.5	5.54E-03	-89.2	2.18E-03	-95.8
E	kg PO_4 eq.	1.25E-02	1.11E-03	-91.1	1.88E-03	-84.9	1.32E-03	-89.4	5.05E-04	-95.9

The results highlighted in Table 4.11 show the MRA with an adequate mechanical behavior and leaching impact along with a significant decrease in the impact in the categories analyzed with respect to AG.

Likewise, the contribution to the impact of aggregate production processes is shown in Fig. 4.9. For the production of AG (Fig. 4.9a), the processes that most contributed to the impact were extraction by blasting in the categories ADe (56.9%), POF (48.3%), A (75.7%) and E (78.7%), and crushing for ADf (49.6%), GW (43.2%) and ODP (43%). In categories A and E, the highest values were produced by air emissions of Nitrogen oxides and Ammonia during the blasting extraction process.

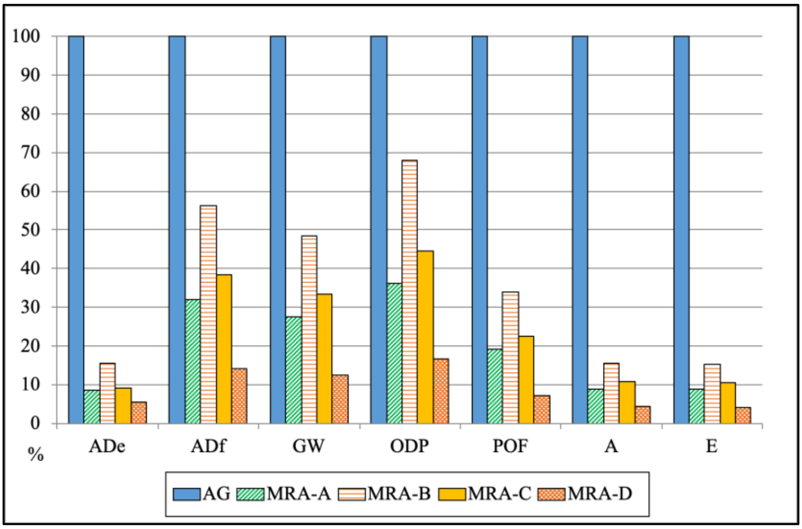


Fig. 4. 8. Comparative chart of aggregates impact assessment**.**

Regarding the production of MRA-A, the impacts were generated mainly by the crushing process, with contributions that ranged between 35% for the ADe category, and 68% for the POF category, due to air emissions from Sulfur dioxide and Carbon monoxide, fossil. The second major impact was caused by the handling by shovel loader process in all categories, except for ADe; for this category, the second largest contribution was due to the handling by conveyor belt process.

Similarly, in the production of MRA-B and MRA-C, the greatest contribution to impact in all categories was due to the crushing process, with percentages similar to those indicated for MRA-A. In addition, the second largest impact was also caused by handling by shovel loader in all categories, except for ADe, whose second largest contribution was due to hopper/pre-screening.

Finally, in the production of MRA-D, handling by shovel loader caused the greatest contribution to impact in all categories, except for ADe, with contributions ranging from 74.9% for ODP and 86.7% for category E, such as consequence of the air emissions of Nitrogen oxides and the water emissions of Phosphate. For the ADe category, the biggest contribution was from the hopper/pre-screening process due to the use of raw materials (Copper, Chromium and Tin) for the manufacture of the equipment.

Fig. 4. 9. Contribution analysis of the production processes of AG (a), MRA-A (b), MRA-B (c), MRA-C (d) and MRA-D (e).

4.3.3.3.2. Characterization results of road sections

The results obtained in the GW category corresponding to 1km of road section, as well as the contribution of the processes to the impact, are shown in Fig. 4.10 (left column and right column). The highest CO_2 emission value was 33.04t for AG road section, followed by 14.02t for MRA-C road section, 12.52t for MRA-B road section and finally, 10.69t for MRA-A road section with the lower value (Fig. 4.10, left column). By processes (Fig. 4.10, right column), the greatest contribution to impact in all road sections occurred during the transport of aggregates from their place of production to the construction site of the section (15 km) with values around 52-67.9%. Then, aggregate production generated variable impacts of between 20% when MRA-B was used and 35% when AG was used. Construction activities caused between 12.5% and 18.6%, while water production and transportation generated impacts around 0.7% and 0.1%, respectively. However, the use of MRA as a replacement for AG avoided its production, so that the impact on the GW category was reduced by 11.56t of CO_2, this being the environmental benefit generated.

The main substances emitted into the air during the transport process were Carbon dioxide, Dinitrogen monoxide and Methane, fossil. On the other hand, by avoiding the production of AG, air emissions of substances such as Dinitrogen monoxide and Methane, biogenic produced during crushing and blasting processes were reduced.

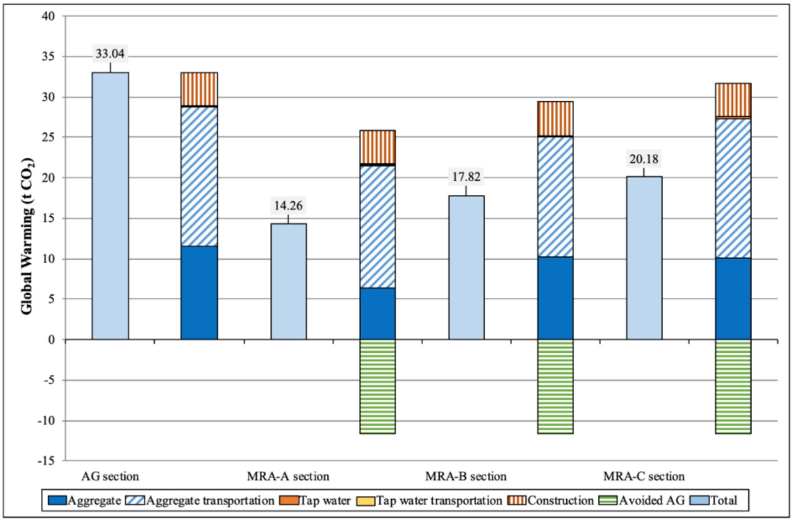


Fig. 4. 10. CO_2 emissions generated by section (left column) and process contribution (right column).

4.4.- Discussion

4.4.1.- Discussion of the results of mechanical behavior

Curves shape and values obtained in Modified Proctor test differ considerably between MRA and AG and the values also vary between the four MRA. The AG had the highest dry density value and the lowest optimum moisture content value. The different maximum dry density and optimum moisture content between both types of materials is mainly due to the physical properties, since MRA have compositions with percentages of ceramic and plaster material, materials with a low density and a high porosity (Poon and Chan, 2006). Conversely, AG is composed of granite particles, which had a high density. Also the different values of dry density and humidity between the MRA are due to their composition and particle size distribution, which is due to high values in ceramic particles content and a high percentage of fine fraction.

The lower density of MRA than AG means a material reduction for the execution of a 1 m3of road sub-base, which translates into a lower consumption of natural resources and a reduction of the environmental impact. On the other hand, high optimum humidity values imply greater water additions to obtain the maximum dry densities.

Analyzing CBR results obtained, MRA-A sample presented the highest value of the four MRA in unsoaked conditions (136.87%) followed by the MRA-B sample (112.69%) whereashe MRA-D sample showed the lowest bearing capacity (75.97%). The measured values are consistent with the data obtained by previous authors (Jiménez et al., 2012) which characterized recycled aggregates to be applied in civil infrastructures. From the comparison between CBR data unsoaked and after 4 day soaked it can be concluded that the influence of the soaked period was worse on the CBR values for all cases.

The bearing capacity measured by the CBR index is directly related to the MRA composition, obtaining the most positive results in the samples that presented the highest percentage of natural material and concrete, associating the CBR drop with higher percentages of ceramic material, gypsum and other components. Furthermore, according to the results obtained, it can be observed that there is a relationship between different properties and CBR index (Fig. 4.11).

The relationship between the degree of compaction and the bearing capacity of the RA (Fig. 4.11a) was observed in previous studies (Poon and Chan, 2006), this being a determining parameter in the execution of road layers. On the other hand, the decrease in bearing capacity with the increase in water absorption (Fig. 4.11b) could be due to the higher content of ceramic materials, gypsum, etc., associated with the increase in water absorption (Agrela et al., 2011). The same occurs with the fall of CBR with the increase in water-soluble sulfates (Fig. 4.11c), which could be due to the presence of gypsum related (Barbudo et al., 2012b).

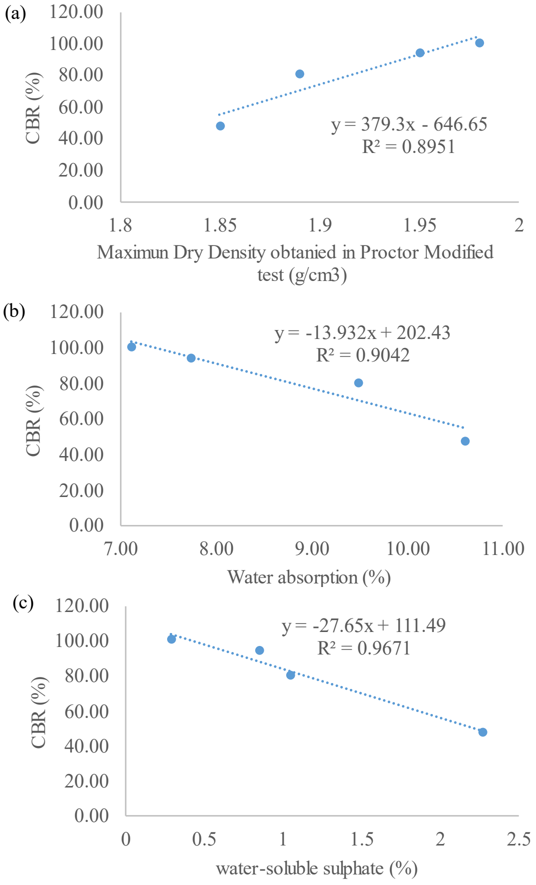


Fig. 4. 11. Correlation between CBR vs. Maximum Dry Density (a), CBR vs. Water Absorption (b) and CBR vs. Water-Soluble Sulfates (c)

The quality of the MRA as useful material for the execution of roads bases and sub-bases is demonstrated. All the samples tested exceeded the lower limit of 20% of CBR imposed by the Spanish regulations for the execution of linear works (PG-3, 2002), which confirmed that MRA-I and MRA-II type with a composition by weight of at least 40-50% Ru + Rc and with a total of 85-90% of Ru + Rc + Rb + Ra are suitable for use in road bases and sub-bases, respectively. Finally, analyzing CBR values of MRA-C and MRA-D samples, it can be observed that MRA from CDW without selective collection and with short processing, which is not as complex as the production of MRA-B, could be used for road construction. However, its application is not recommended due to the possible environmental issues caused by sulfates leaching due to the gypsum content (Barbudo et al., 2012b).

4.4.2.- Discussion of leaching results

According to the results obtained in compliance test (Table 4.7), it can be observed that the AG was classified as an inert material because it comes from a natural stone material, whereby the low contaminant level of this material is demonstrated without the need to perform the percolation test. Analyzing Fig. 4.12, none of the four MRA samples exceed the limits of inert material in heavy metal concentration; in fact the concentrations of all elements in the four samples were less than 40% of the inert limit. However, all four samples exceed inert levels in sulfates concentration, being classified as non-hazardous waste according to the compliance test.

A relationship is observed between the selective collection at the origin of CDW, which reduces gypsum content and other components, and the concentration of leached sulfates, since the concentration of MRA-A sulfates is 80% lower than the concentration of MRA-B, despite the fact that it is the most complex MRA-B manufacturing process.

Analyzing Table 4.8 and Fig. 4.13, the results obtained in the compliance test to the MRA-B, MRA-C and MRA-D are checked, with the exception of MRA-A which is classified as inert. In addition, as the sulfate levels in the compliance test do not exceed 6000 mg / kg. it is concluded that it is the only recycled aggregate classified as inert. Moreover, it can be observed that the sulfate concentrations obtained in the compliance test are between 10-60% over the non-hazardous limit, whereas the concentrations obtained in the percolation test are in no case higher than 25% of the non-hazardous limit. These results indicate that the limits imposed by the regulations are more restrictive for the compliance test, limiting the possible use of mixed recycled aggregates.

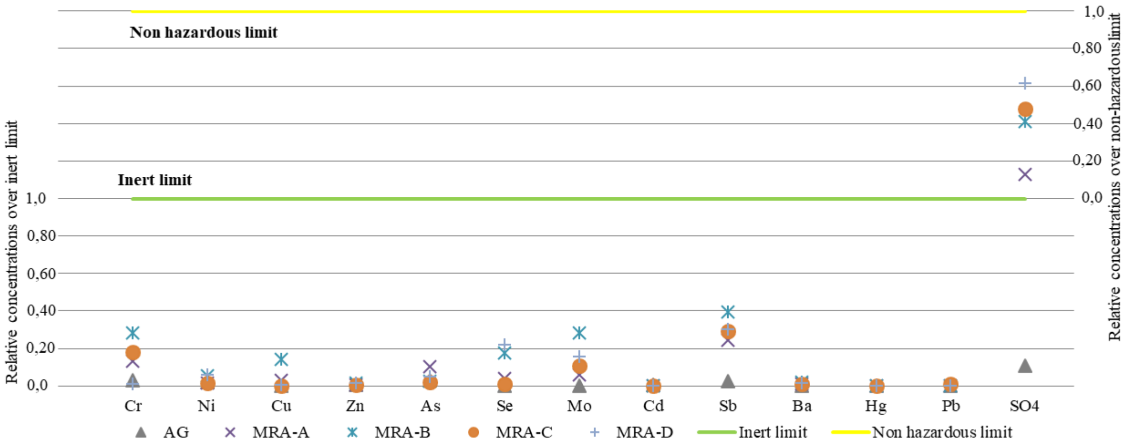


Fig. 4. 12. Relative leached concentration of artificial gravel and mixed recycled aggregates by Compliance test accord inert and non-hazardous limits

In summary, the MRA-A, which comes from a CDW with selective collection at the origin, is suitable for the execution of road layers, and the MRA from CDW without selective collection is classified as non-hazardous and is not suitable, despite its excellent mechanical behavior. However, the acceptance criteria set by the EU Landfill Directive 2003/33 / EC is designed for large accumulations of material but the actual use of MRA is done in layers of 30 cm maximum thickness, so the limit values are considered excessive and not adjusted to reality. A change in the regulations which would include this reality could mean an increase in the use of MRA that come from CDW without selective collection but with adequate in-plant treatment On the contrary, materials such as MRA-D have sulfate levels that are so high that their use is not advised.

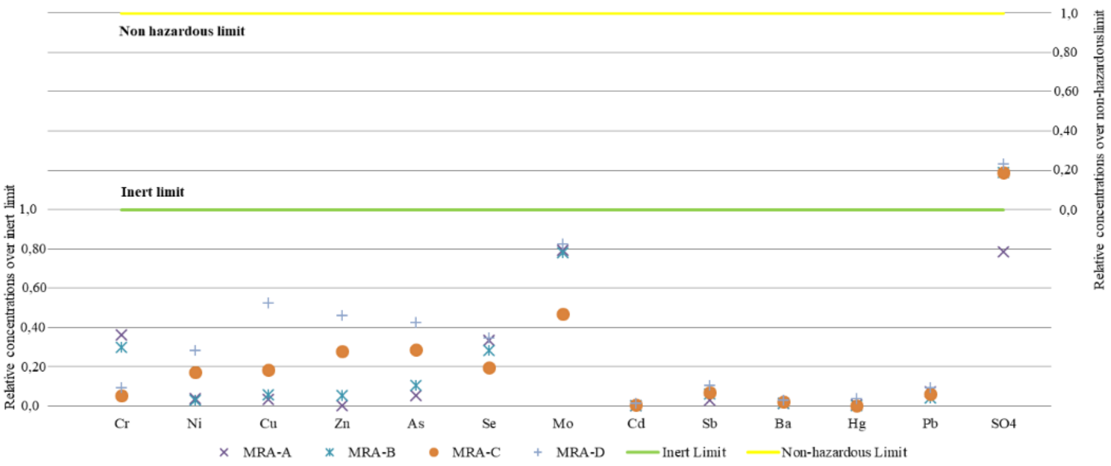


Fig. 4. 13. Relative Leached concentration of mixed recycled aggregates by Percolation test accord inert and non-hazardous limits

Barbudo et al. (2012b) conducted a study in order to analyze the correlation between sulfate content and leached sulfates in MRA. Fig. 4.13 shows the relationships obtained from Barbudo et al. (2012b) study together with the results analyzed in the present study. Fig. 4.14 (up) shows the relationship between water-soluble sulfates (WSS) and the percentage of gypsum in composition, observing a good integration of the data in both studies, presenting the linear regression line for the entire data set with an R2 value similar to the value presented by each study in particular. In contrast, Fig. 4.14 (down) presents a low R2 value, but that still allows us to show the relationship between WSS and leached sulfates, observing that for a value of approximately 0.70% of WSS the material can be classified as non-hazardous, presenting a value of 6000 mg / kg of leached sulfates.

Analyzing Fig. 4.15, made from the data of the present study, a very high value of R2 is observed for both relationships. Fig. 4.15 (up), indicates the correlation between Sulfates leached in a compliance and percolation test, confirming the reaction between 1500 mg / l and 6000 mg / kg in both tests. Fig. 4.15 (down) relates the value of 0.70-0.80% WSS again to the current inert limit.

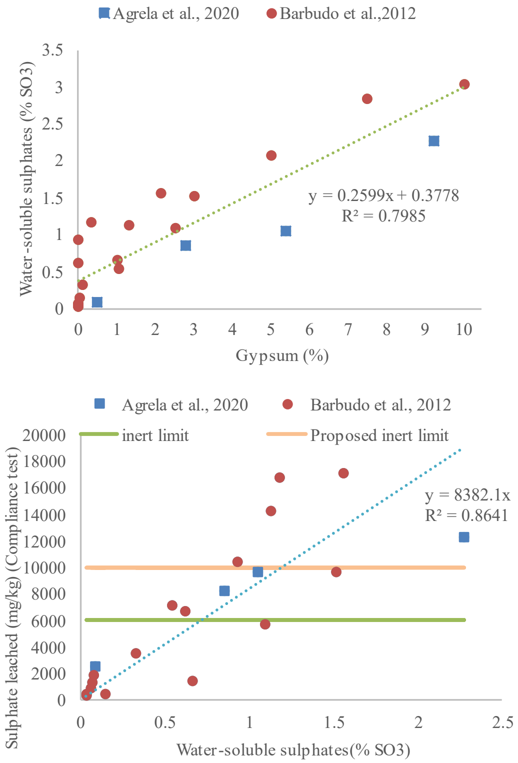


Fig. 4. 14. Correlation between Water-soluble sulfates vs. Gypsum (up) and Sulfates leached in Compliance test (*UNE EN 12457- 4)* vs. Water-soluble sulfate (down)

According to the results, the present study proposes to increase the theoretical limit of leached sulfates in percolation tests from 1500 mg / l to 1800 mg / l. This value, which is very far from the non-hazardous limit (8500 mg / l), would imply an increase in the inert limit to a value of 10000 mg / kg of sulfates leached in compliance test, a value found in the first quartile of the environment inert / not hazardous. This increase in limits would considerably increase the range of use of MRA in the execution of roads layers, without negatively affecting the environment. Theoretically, MRA with an amount of up to 1.5% WSS could be used, but due to the uncertainty of the data, the criteria established by De Brito, Agrela and Silva (2019) in their classification, with a value of 1.2% of WSS, is accepted and maintained.

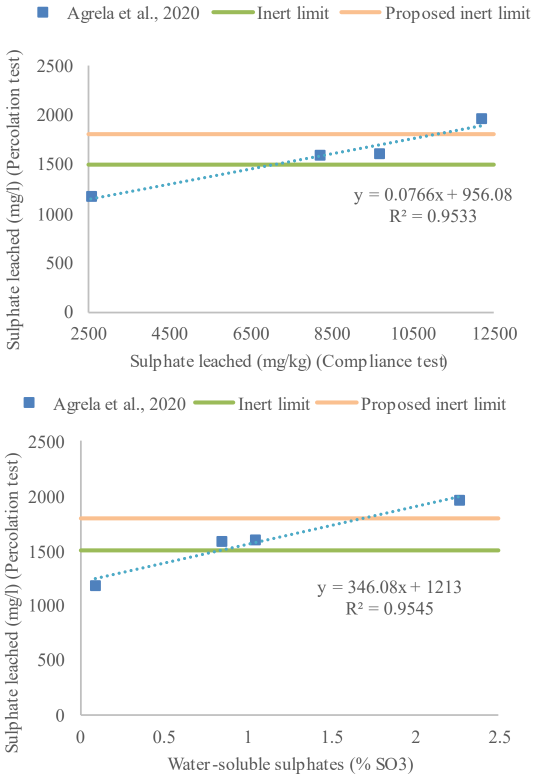


Fig. 4. 15. Correlation between Sulfates leached in Percolation test vs. Sulfate leached in Compliance test (up) and Sulfates leached in Percolation test vs. Water-Soluble Sulfate (down)

The main leaching mechanism is the liquid transport that will determine the intensity and speed of the leaching process. The mobilization of contaminants from the recycled aggregate as granular material preferably takes place by percolation. However, if MRA were treated with cement, the concentration of the leachate was greatly reduced because the leaching potential of recycled aggregates incorporated in a cement matrix, is mainly given by diffusion mechanisms (Sani et al 2005; Barbudo et al 2012b; Galvin et al, 2014; Cabrera et al., 2016), measured by the tank leaching test (EA NEN 7375: 2004) to levels of compliance with the water parameters suitable for human consumption, according to the United States Environmental Protection Agency (EPA)

4.4.3. Discussion of the results of LCA

According to the results obtained, the impact values were lower in the road sections where MRA was used, due to the lower impact generated during its production, as has been determined in the previous section (6.2.1). In addition, the amounts of MRA-A and MRA-B required to build the road sections are lower than that of AG, according to the Proctor density values (Table 4.7). Also, the use of MRA-A in the construction of unbound granular sub-base layer achieved the best environmental result; MRA-A treatment included selection at the site of demolition, crushing and screening, obtaining a high quality MRA. Likewise, the environmental benefit generated by using MRA as a replacement for AG should be considered, thus avoiding its production, and consequently, the maintenance of natural resources.

Fig. 4.16 represents the characterization values for the ADe and GW impact categories. MRA-D presented the lowest values since its treatment was carried out through a single pre-screening process; however, it did not reach the required technical feasibility since no selection was made at source. In contrast, MRA-A treatment included selective collection at the site of demolition, crushing and screening, which resulted in low impact values and a high quality MRA.

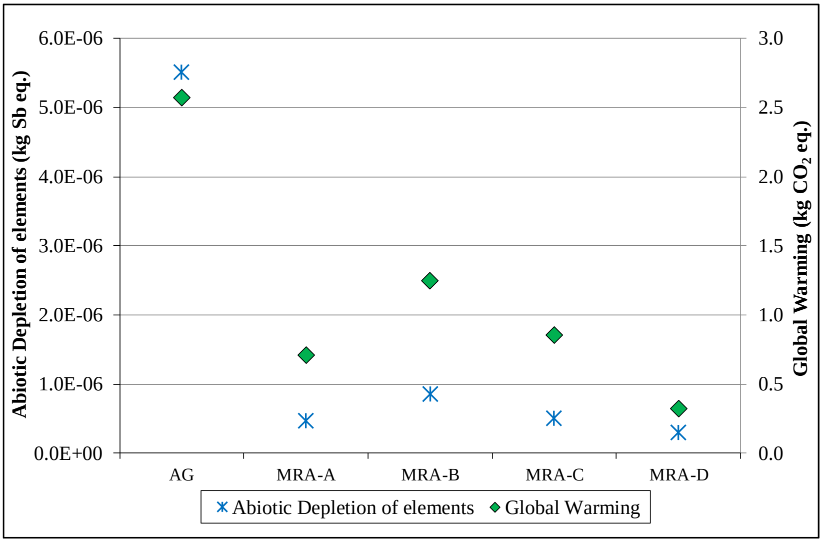


Fig. 4. 16. Values of ADe and GW impact categories for aggregates

Proper management of C&DW can be achieved by separating waste streams at the demolition site. It can bring great benefits regarding quality of RA and their environmental impact. If the selection is not made at the demolition site, the treatment to obtain an MRA with good mechanical behavior will be more complex as it requires more processes, and therefore, the energy consumption and emissions generated during its production will be increased.

In this regard, the European Commission has recently published several documents that can boost trust for RA from C&DW, such as the "EU Construction and Demolition Waste Management Protocol" and the "Guidelines for the waste audits before demolition and renovation works of buildings". (European Commission, 2018).

4.5.- Conclusions

This work shows an exhaustive study of the feasible use of mixed recycled aggregates applied in road layers compared to natural aggregates. The study covers the analysis of physical-chemical properties, mechanical behavior, environmental impact of the leachates and life cycle analysis of the natural and recycled materials analyzed.

The following specific conclusions are obtained:

Selective collection at the origin of construction and demolition waste improves the physicochemical properties of mixed recycled aggregates, obtaining MRA with a lower amount of gypsum and other materials such as wood, glass or paper in composition, and selective collection systems allow mixed recycled aggregates to be obtained with adequate particle size distribution and suitable compositions, reducing processing at treatment plants, which results in less environmental impact and reduction of greenhouse gas emissions.

Mixed recycled aggregate composition influences the maximum dry density and the optimum moisture content, whereby MRA that are composed of high percentages of natural aggregate and concrete have higher densities and lower optimum humidity.

The maximum dry density of recycled aggregates is always lower than the density of natural aggregates, which translates into a lower consumption of resources in the execution of road layers, without compromising the structural behavior of the road, due to the high bearing capacity of recycled aggregates.

Leachates analyzed have shown that the mixed recycled aggregates produced from CDW without selective collection at the origin are classified as non-hazardous waste due to the high concentration of sulfates from the composition plaster. In contrast, MRA manufactured from CDW with selective collection can be classified as inert waste.

Mixed recycled aggregates with a gypsum composition greater than 5% by weight, are not suitable for road layer construction, despite the adequate mechanical behavior that all samples of MRA present, due to the sulfate leaching problems associated with this component.

A lack of specific regulations that contemplate the actual concentrations that MRA layers could generate can be observed, since European directive 2003/33 / EEC of landfill admission present excessive limit values for the application of MRA in roads because thicknesses made in works are lower than those contemplated by the landfill directive.

The impact values caused during the production of AG were the highest, due to the consumption of natural resources (granite), as well as the emissions generated during its blasting extraction process.

In contrast, the production of MRA-D generated the lowest impacts as a result of the lower number of processes required for its treatment, since it was obtained directly in the pre-screening process. However, the selection at source was not carried out, so the high sulfate content in its composition prevented its use as unbound granular sub-base layer.

MRA-A production caused the second least impact in all categories. In addition, its physico-mechanical and chemical characteristics confirmed an adequate behavior in the formation of unbound granular sub-base layer; which resulted in the road section with the least amount of materials (MRA and water), and therefore, generated the least environmental impact.

Likewise, it has been possible to verify the technical and environmental feasibility of the use of MRA-B and MRA-C as a granular unbound sub-base layer in road construction.

As a general conclusion we can say that it is feasible to use mixed recycled aggregates in the execution of road sub-base layers, due to the high bearing capacity they possess, provided that the leaching properties present appropriate results that are classified as inert materials. As a consequence, the use of these materials will reduce environmental impact using these recycled aggregates, compared to the quarry extracted material in civil works and building constructions, thus promoting a model of circular and sustainable economy.

Standards used in the experimental work

EA NEN 7375:2004. Leaching characteristics of moulded or monolithic building and waste materials.

EN 15804:2012+A1:2013.Sustainability of construction works. Environmental product declarations. Core rules for the product category of construction products.

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UNE-EN ISO 14040:2006a. Environmental management - Life cycle assessment - Principles and framework

UNE-EN ISO 14044:2006b. Environmental management - Life cycle assessment - Requirements and guidelines

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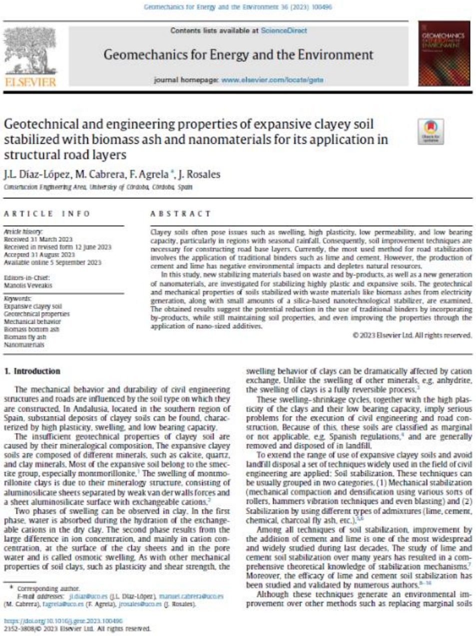
CAPÍTULO V

GEOTECHNICAL AND ENGINEERING PROPERTIES OF EXPANSIVE CLAYEY SOIL STABILIZED WITH BIOMASS ASH AND NANOMATERIALS FOR ITS APPLICATION IN STRUCTURAL ROAD LAYERS

Díaz-López, J.L., Cabrera, M., Agrela, F., Rosales, J.



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Geotechnical and engineering properties of expansive clayey soil stabilized with biomass ash and nanomaterials for its application in structural road layers

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ABSTRACT

Clayey soils often pose issues such as swelling, high plasticity, low permeability, and low bearing capacity, particularly in regions with seasonal rainfall. Consequently, soil improvement techniques are necessary for constructing road base layers. Currently, the most used method for road stabilization involves the application of traditional binders such as lime and cement. However, the production of cement and lime has negative environmental impacts and depletes natural resources.

In this study, new stabilizing materials based on waste and by-products, as well as a new generation of nanomaterials, are investigated for stabilizing highly plastic and expansive soils. The geotechnical and mechanical properties of soils stabilized with waste materials like biomass ashes from electricity generation, along with small amounts of a silica-based nanotechnological stabilizer, are examined. The obtained results suggest the potential reduction in the use of traditional binders by incorporating by-products, while still maintaining soil properties, and even improving the properties through the application of nano-sized additives.

1. Introduction

The mechanical behavior and durability of civil engineering structures and roads are influenced by the soil type on which they are constructed. In Andalusia, located in the southern region of Spain, substantial deposits of clayey soils can be found, characterized by high plasticity, swelling, and low bearing capacity.

The insufficient geotechnical properties of clayey soil are caused by their mineralogical composition. The expansive clayey soils are composed of different minerals, such as calcite, quartz, and clay minerals. Most of the expansive soil belong to the smectite group, especially montmorillonite.[1] The swelling of montmorillonite clays is due to their mineralogy structure, consisting of aluminosilicate sheets separated by weak van der walls forces and a sheet aluminosilicate surface with exchangeable cations.[2]

Two phases of swelling can be observed in clay. In the first phase, water is absorbed during the hydration of the exchangeable cations in the dry clay. The second phase results from the large difference in ion concentration, and mainly in cation concentration, at the surface of the clay sheets and in the pore water and is called osmotic swelling. As with other mechanical properties of soil clays, such as plasticity and shear strength, the swelling behavior of clays can be dramatically affected by cation exchange. Unlike the swelling of other minerals, e.g. anhydrite, the swelling of clays is a fully reversible process.[3]

These swelling–shrinkage cycles, together with the high plasticity of the clays and their low bearing capacity, imply serious problems for the execution of civil engineering and road construction. Because of this, these soils are classified as marginal or not applicable, e.g. Spanish regulations,[4] and are generally removed and disposed of in landfill.

To extend the range of use of expansive clayey soils and avoid landfill disposal a set of techniques widely used in the field of civil engineering are applied: Soil stabilization. These techniques can be usually grouped in two categories. (1) Mechanical stabilization (mechanical compaction and densification using various sorts of rollers, hammers vibration techniques and even blasting) and (2) Stabilization by using different types of admixtures (lime, cement, chemical, charcoal fly ash, etc.).[5,6]

Among all techniques of soil stabilization, improvement by the addition of cement and lime is one of the most widespread and widely studied during last decades. The study of lime and cement soil stabilization over many years has resulted in a comprehensive theoretical knowledge of stabilization mechanisms.[7] Moreover, the efficacy of lime and cement soil stabilization has been studied and validated by numerous authors.[8–16]

Although these techniques generate an environmental improvement over other methods such as replacing marginal soils

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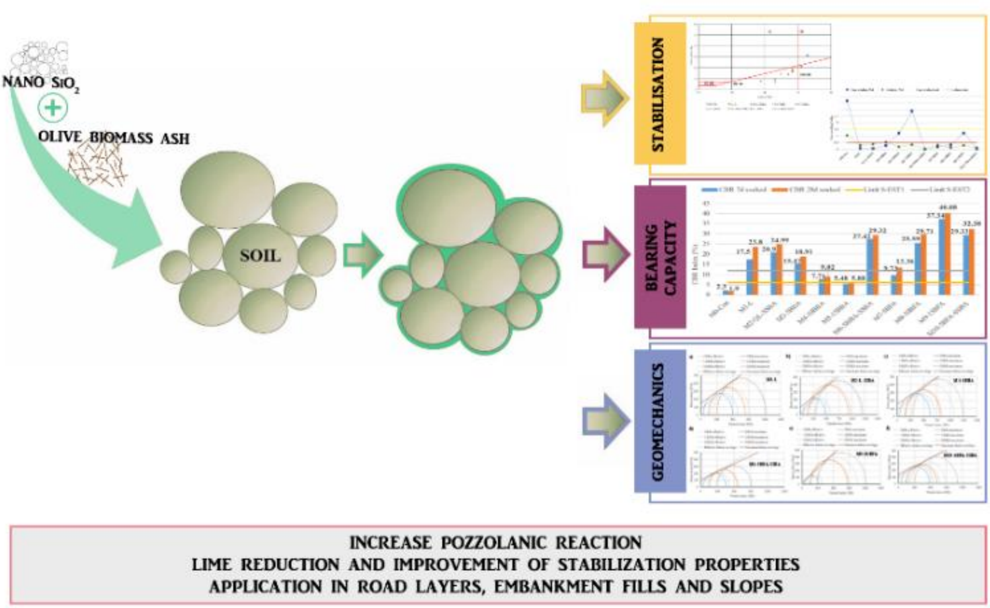
V

GEOTECHNICAL AND ENGINEERING PROPERTIES OF EXPANSIVE CLAYEY SOIL STABILIZED WITH BIOMASS ASH AND NANOMATERIALS FOR ITS APPLICATION IN STRUCTURAL ROAD LAYERS

Díaz-López, J. L., Cabrera, M., Agrela, F., & Julia, R. (2023). Geotechnical and Engineering Properties of Expansive Clayey Soil Stabilised with Biomass Ash and Nanomaterials for its Application in Structural Road Layers. Geomechanics for Energy and the Environment. 36, 100496



Abstract

Clayey soils often pose issues such as swelling, high plasticity, low permeability, and low bearing capacity, particularly in regions with seasonal rainfall. Consequently, soil improvement techniques are necessary for constructing road base layers. Currently, the most used method for road stabilization involves the application of traditional binders such as lime and cement. However, the production of cement and lime has negative environmental impacts and depletes natural resources.

In this study, new stabilizing materials based on waste and by-products, as well as a new generation of nanomaterials, are investigated for stabilizing highly plastic and expansive soils. The geotechnical and mechanical properties of soils stabilized with waste materials like biomass ashes from electricity generation, along with small amounts of a silica-based nanotechnological stabilizer, are examined. The obtained results suggest the potential reduction in the use of traditional binders by incorporating by-products, while still maintaining soil properties, and even improving the properties through the application of nano-sized additives.

Keywords

Expansive clayey soil; geotechnical properties; mechanical behavior; biomass bottom ash; biomass fly ash; nanomaterials.

5.1.- Introduction

The mechanical behavior and durability of civil engineering structures and roads are influenced by the soil type on which they are constructed. In Andalusia, located in the southern region of Spain, substantial deposits of clayey soils can be found, characterized by high plasticity, swelling, and low bearing capacity.

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Two phases of swelling can be observed in clay. In the first phase, water is absorbed during the hydration of the exchangeable cations in the dry clay. The second phase results from the large difference in ion concentration, and mainly in cation concentration, at the surface of the clay sheets and in the pore water and is called osmotic swelling. As with other mechanical properties of soil clays, such as plasticity and shear strength, the swelling behavior of clays can be dramatically affected by cation exchange. Unlike the swelling of other minerals, e.g. anhydrite, the swelling of clays is a fully reversible process [3].

These swelling-shrinkage cycles, together with the high plasticity of the clays and their low bearing capacity, imply serious problems for the execution of civil engineering and road construction. Because of this, these soils are classified as marginal or not applicable, e.g. Spanish regulations [4], and are generally removed and disposed of in landfill.

To extend the range of use of expansive clayey soils and avoid landfill disposal a set of techniques widely used in the field of civil engineering are applied: Soil stabilization. These techniques can be usually grouped in two categories. 1) Mechanical stabilization (mechanical compaction and densification using various sorts of rollers, hammers vibration techniques and even blasting) and 2) Stabilization by using different types of admixtures (lime, cement, chemical, charcoal fly ash, etc.) [5, 6].

Among all techniques of soil stabilization, improvement by the addition of cement and lime is one of the most widespread and widely studied during last decades. The study of lime and cement soil stabilization over many years has resulted in a comprehensive theoretical knowledge of stabilization mechanisms [7]. Moreover, the efficacy of lime and cement soil stabilization has been studied and validated by numerous authors [8-16]

Although these techniques generate an environmental improvement over other methods such as replacing marginal soils with granular soils, the production of quicklime and cement through lime calcination generates a large environmental impact both in terms of CO_2 emissions and depletion of natural resources [17].

As a result, in recent years several authors have explored the possibility of applying waste and by-products from different industrial sources as soil stabilizing agent. For example, phosphogypsum has been investigated for its use in the formation of embankments and slopes [18], steel mill slag has been used to reduce expansivity and improve bearing capacity [19], magnesium oxide has been proposed as an alternative material to cement [20], etc.

Among the different types of residues and by-products, biomass bottom ash and biomass fly ash have been extensively studied in last years for their application in road sub-base execution [21, 22] or soil stabilization [23-25].

In addition to the study of new stabilizing materials from waste and by-products, a new line of stabilizing materials has emerged in recent years: nanomaterials.

Nanomaterials have a particle size between 1 and 100 nm, which results a very high specific surface area. This implies a high reactivity with soil and other added stabilizing materials [26].

Nanomaterials can have different compositions, but the most common ones are composed of simple oxides, such as SiO_2 and TiO_2 [27], being the most widespread for soil stabilization tasks the silica-based nanoparticles, due to their high reactivity with lime and cement and their pozzolanic capacity that allows the development of hardening by forming calcium silicate gels [28].

Currently, there a few experiences regarding soil stabilization by adding of nanomaterials in combination with lime. However, the effects of cement/nanomaterials stabilization have been widely studied. [29] investigated the stabilization of a soft clay by adding of a nano organosilane and cement. A dosage of 0.03% nanomaterial and 1.5% cement increased the bearing capacity of the soil threefold compared to untreated soil, as well as produced a reduction in plasticity index and an improvement in unconfined compressive strength.

Bahmani et al. [30, 31] analyzed the improvement of mechanical behavior of residual soil by addition of nanosilica together with cement. The addition of nanosilica promoted the pozzolanic reaction, transforming portlandite into calcium silicate hydrated gel, which implies an improvement the properties of soil.

Despite the current research on new additives for clay soil stabilization, there is a lack of research on the combined effect of applying wastes and by-products with nanomaterials. In the present study, the effects of adding biomass bottom ash and biomass fly ash together nanosilica in the stabilization process of an expansive clay soil were analyzed. Physicochemical and mineralogical properties of the materials have been analyzed. As well as the geotechnical and engineering properties of the developed mixtures of soil and stabilizing materials, showing the stabilizing potential and the high reactivity between waste and by-product materials and state-of-the-art nanomaterials.

5.2.- Materials

In this section, physicochemical properties of material involved in the study are presented. The materials included an expansive clayey soil, quicklime, biomass bottom ash, biomass fly ash, and nanomaterials admixture.

5.2.1.- Clayey soil

Clayey soil (CS) analyzed in the present work was called marly soil. Marly soil is characteristic of the south-eastern Iberian Peninsula. The sample was collected in Jaen, Andalusia, Spain. This soil was mainly composed of calcite, quartz as non-clayey mineral and montmorillonite as clayey mineral. It exhibited high plastic, expansive and a very low bearing capacity. Due to these properties, this soil cannot be used in the execution of subgrade according to Spanish regulations, PG-3, and previous studies carried out [32-34].

Physical and chemical properties are summarized in Table 5.1 and Figure 5.1, as well as XRD patterns are shown in Figure 5.2.

5.2.2.- Lime

An air lime (L) is obtained from the calcination and crushing of limestone was used as a control stabilizing product. The lime used is commercial type

CL-90-Q in accordance with Spanish standard UNE-EN 459-1 (2002b). The composition of the lime obtained by XRF is shown in Table 5.1.

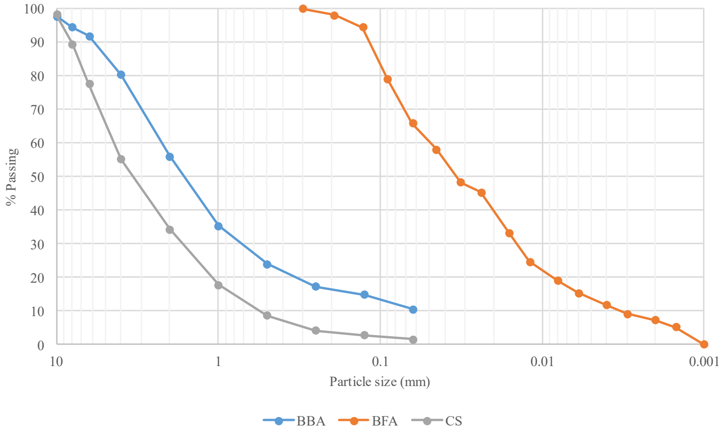

Fig. 5. 1. Particle size distribution of CS, BBA and BFA

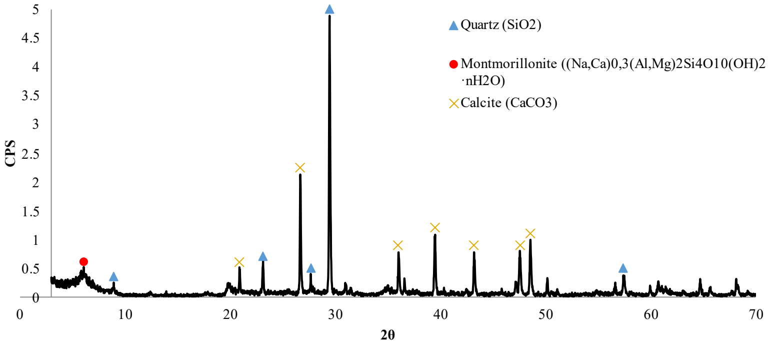

Fig. 5. 2. XRD of CS

5.2.3.- Biomass bottom ash and biomass fly ash

Two types of biomass ashes have been studied: biomass bottom ash (BBA) and biomass fly ash (BFA), both obtained from the biomass combustion power plant Sacyr Valoriza SA, located in the town of Linares, province of Jaén.

Biomass bottom ash is a residue generated during the combustion of biomass, collected at the bottom of the combustion boilers.

Biomass fly ash consists of the fine particles that are carried away by the flue gas during combustion and collected in the chimney filters.

The physicochemical properties of both biomass bottom ash and biomass fly ash are presented in Table 5.1. The mineralogical analysis of both ashes using X-ray diffraction is depicted in Figures 5.3 and 5.4.

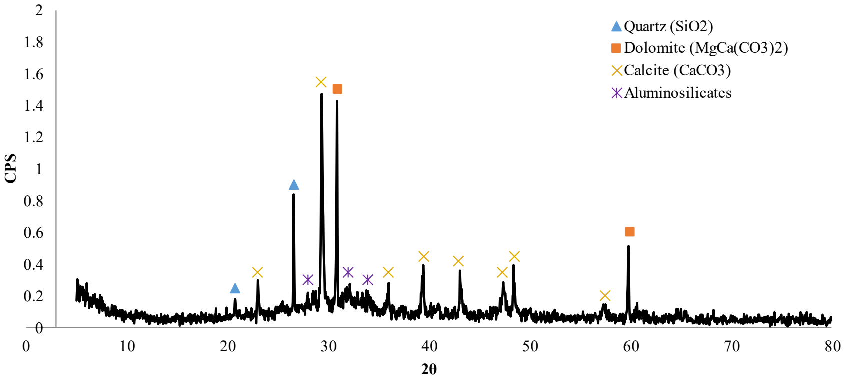

Fig. 5. 3. DRX of BBA

Table 5. 1. Physicochemical properties of CS, L, BBA and BFA

Properties	CS	L	BBA	BFA	Standard Test
Atterberg limits					UNE-EN ISO 17892-12:2019
Liquid Limit	53	-	Non plastic	Non plastic	
Plastic limit	21.6	-	Non plastic	Non plastic	
Index plasticity	31.4	-	Non plastic	Non plastic	
Grain size distribution as received					UNE-EN ISO 17892-4:2019
> 4 mm (Gravel size) (%)	44.8	-	19.7	0	
0.063-4mm (Sand size) (%)	53.7	-	69.8	34.13	
<0.063 mm (Silt and clay size) (%)	1.5	-	10.5	65.87	
Organic matter content (%)	0.27	0	1.51	0.14	UNE 103204:2019
Water-soluble sulphate (% SO3)	0	0	0	2.55	UNE-EN 1744-1:2010+A1:2013
Main components XRF (%)					
P	0.08	0.02	0.1	0.12	
Si	25.57	1.11	15.75	3.74	
Ca	14.16	40.9	15.75	8.18	
Al	2.69	0.78	1.6	0.69	
S	0.03	0.24	0.11	2.19	
K	1.66	0.06	11.67	45.1	
Mg	1.37	8.3	3.59	1.97	
Fe	2.42	0.15	1.76	0.51	

As shown in Table 5.1 and Figure 5.1, the BBA had a coarse grain size, equivalent to that of a gravel, with maximum soil sizes up to 80 mm. The BBA had a high percentage of organic matter, due to the unburnt residues produced during combustion in the boilers of the power plant. The mineralogy of BBA, as observed in Figure 5.3, is rich in quartz, dolomite, calcite, and calcium aluminate compounds, which are typical minerals found in BBA resulting from the combustion of olive waste [22, 23, 35].

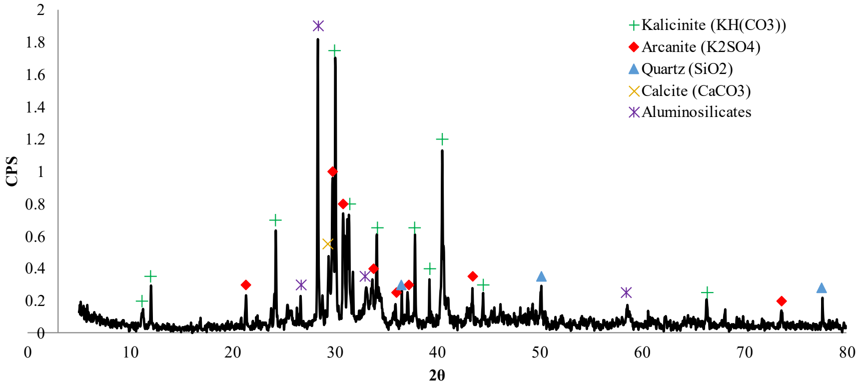

Fig. 5. 4. XRD of BFA

BFA had a fine particle size distribution with more than 65% of its mass consisting of particles smaller than 63 microns. It had a low amount of organic matter, as well as a high percentage of water-soluble sulphates and a high potassium composition.

The X-ray diffraction analysis of BFA, as shown in Figure 5.4, indicates a mineralogical composition predominantly composed of phases with high potassium content, carbonates, potassium sulphates, and minor phases such as quartz and silicates, which is consistent with previous studies [35, 36].

5.2.4.- Nanosilane: Sodium silicate-based admixture

The nanomaterial utilized in this study is a silica-based liquid solution known as SSBA, which is used for soil stabilization in combination with lime or cement.

As presented in Table 5.2, it is mainly composed of silicon and sodium. This high silica content together with the high level of fineness of SSBA gives it a high reaction potential with the calcium compounds in lime and the aluminosilicates in clay, promoting the formation of calcium silicate (CS) and calcium silicate hydrate (CSH) gels which leads to an increase in the mechanical properties of the stabilized soils [37].

Table 5. 2. Main composition of SSBA

Density (kg/dm^3)	1.4
Main components XRF (%)	
P	-
Si	24.2
Ca	0.279
Al	0.18
S	0.02
K	0.026
Mg	0.15
Na	12.1
Fe	0.057

Upon analyzing the diffractogram depicted in Figure 5.5 it can be observed that the nanomaterial was mainly composed by amorphous phase corresponding to the nanosilica, as indicated by the diffraction halo. Additionally, mineral phases such as dolomite, quartz and sodium silicate were found. There are previous experiences that have demonstrated the advantages of sodium silicate as a soil stabilizer. Stabilization of montmorillonite-rich clay soils with sodium silicate powder and lime achieved high mechanical properties. [16, 37-40]

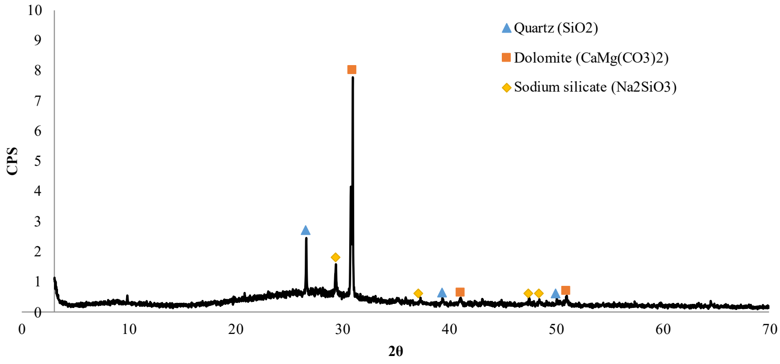

Fig. 5. 5. XRD of SSBA

5.3.- Experimental methods

5.3.1.- Mix design and preparation of samples for the test

In the laboratory, the mixture was prepared following an initial drying of the materials at 60ºC for 24 hours in an air oven to control the moisture content during compaction or wetting in the testing process.

The properties of the clayey soil and ten laboratory-prepared mixtures were analyzed.

Lime, biomass bottom ash, biomass fly ash, and nanomaterial SSBA were added to the clayey soil in the required amount for each test as a percentage of the total dry mass to be stabilized, according to the proportions specified in Table 5.3.

As depicted in Figure 5.1, the particle size of BBA was up to 10 mm, and therefore, it undergoes a dry screening process to remove particles larger than 2 mm.

Table 5. 3. Mixtures proportions and designation

Designation	Mixture	Mix proportions (%)				
		CS	L	BBA	BFA	SSBA
M0-Con	CS	100	-	-	-	-
M1-L	CS+ 1.5% L	100	1.5	-	-	-
M2-L-SSBA	CS + 1.5%L + SSBA	100	1.5	-	-	0.056
M3-5BBA	CS + 5%BBA + 1%L	100	1	5	-	-
M4-10BBA	CS + 10%BBA + 0.5%L	100	0.5	10	-	-
M5-15BBA	CS + 15%BBA	100	0	15	-	-
M6-5BBA-SSBA	CS + 5%BBA+0.5%L + SSBA	100	0.5	5	-	0.056
M7-5BFA	CS + 5%BFA + 1%L	100	1	-	5	-
M8-10BFA	CS + 10%BFA + 0.5%L	100	0.5	-	10	-
M9-15BFA	CS + 15%BFA	100	0	-	15	-
M10-5BFA-SSBA	CS + 5%BFA+0.5%L + SSBA	100	0.5	-	5	0.056

The nanomaterial was mixed with the water for each mixture, with a dosage of 0.65 liters of SSBA per cubic meter of soil to be stabilized. Considering the soil density, this results in a dosage of 0.56 g of SSBA per 1 kg of clayey soil (0.056%).

5.3.2.- Compaction test

The Modified Proctor test determines the maximum dry density of a soil and the optimum moisture content required to achieve this density. For this purpose, a cylindrical mold with a capacity of 2,320 cm3 is used and a 4,535 kg sledgehammer is dropped from a height of 457 mm. Five layers of material are compacted, giving 25 blows per layer, in accordance with the UNE 103501:1994 standard.

5.3.3.- Plasticity index

Determination of the liquid limit, plastic limit and plasticity index of the mixtures according to UNE-EN ISO 17892-12:2019. The measurement of the plasticity indexes allows a first approximation of the degree of stabilization achieved with the addition of the stabilizing materials used.

5.3.4.- Odometer tests: free swelling and collapse test

The determination of free swelling is regulated by the UNE 103.601-96 standard. It is determined as the increase in height, expressed as a percentage of the initial value, experienced by a soil specimen when it is laterally confined, subjected to a vertical pressure of 10 kPa and flooded with water.

The percentage collapse potential according to the UNE 103406:2006 standard, is the collapse value determined, for 200kPa vertical pressure, as the percentage decrease in height experienced by the specimen when flooded, once equilibrium is reached under the action of the selected vertical pressure, with respect to the initial height of the specimen.

The samples are compacted according to the optimum moisture content to achieve de maximum dry density obtained in the Modified Proctor test and storage at 20ºC for 7 days.

5.3.5.- Bearing capacity test: CBR Index

The CBR test is used to evaluate the bearing capacity of compacted soils. The CBR soil test consists of compacting a soil in standardized molds, immersing them in water and applying a punch to the surface of the soil using a standardized piston, in accordance with the UNE 103502:1995 standard. The test is carried out by applying a load with a 19.35 cm2 circular piston to introduce it into a soil sample at a speed of 1.27 mm/min and until a penetration of 2.54 mm is obtained. The samples are compacted to the optimum moisture content obtained in the Modified Proctor test and soaked for 7 and 28 days.

5.3.6.- Shear strength test

Determination of the maximum and effective shear parameters of soil mixtures in triaxial cell according to UNE-EN ISO 17892-9:2019, consolidated undrained test (CU). In the triaxial CU test, the cylindrical specimen (76 mm length and 38 mm diameter) is saturated and consolidated under isotropic conditions, allowing drainage. The deviatoric stress is applied, on the other hand, without allowing drainage but at the appropriate rate such that the interstitial pressure is uniform throughout the specimen, and its evolution is measured throughout the process.

The CU type triaxial test process is divided into:

- Saturation process: The objective of this process is to ensure that all the existing pores in the specimen are occupied by water, allowing the correct measurement of the interstitial pressures. The back pressure is the pressure we need to apply inside the partially saturated soil sample to help reach 100% saturation.

- Immediately after the sample is saturated, the consolidation process must be started: It consists of applying an isotropic pressure until complete consolidation of the soil sample. The objective is to bring the specimen to the desired state of effective stresses before starting the cracking process. The test is repeated at three conditions of effective pressure (50KPa, 150KPa and 300KPa), thus obtaining the angle of internal friction and the effective cohesion of the mixtures.

- Shearing process: During the deviatoric stress (σd) application phase of the specimen, the external pressure in the cell is kept constant ($\sigma 3$) and no drainage is allowed, thus, the moisture content of the specimen remains constant. The method used for the execution of this test is that of controlled deformation, applying a rupture velocity of 0.0125 mm/min. Once failure has been reached, the test shall be continued in such a way as to ensure that failure has occurred. If the process is plastic deformation, the test is stopped when 12% deformation is reached.

Finally, the deviatoric stress - axial deformation behavior (σd - εa), the interstitial pressure variation with respect to axial deformation (Δu- εa) and the Morh-Coulomb failure envelope were evaluated.

5.3.7.- Hydraulic conductivity test

The hydraulic conductivity coefficient is determined by means of a constant load permeameter with a flexible wall in a triaxial cell. The test consists of measuring the volume of water (q) which, under the effect of a constant pressure drop (Δh), passes through the soil sample 76 mm long (L) and 38 mm in diameter (d) in each time (t). Previously, the soil sample has been subjected to a saturation process identical to the one applied for the triaxial CU test.

The volume of water per unit of time recorded under the established pressure conditions, the coefficient k is determined by applying the following expression, obtained from Darcy law:

$$k\ (cm\ /\ s) = (Q * L)/(A *\ \Delta h)$$

Where k, is the permeability coefficient (cm/s), Q is the flow rate (cm3/s), L is the length of the specimen (cm), A is the cross section of the specimen (cm2) and Δh is the gradient of pressures applying, with value 1020 centimeters of water column.

5.4.- Results and discussion

This section shows the results obtained in the different tests carried out on the stabilized soil mixtures and analyses the effects produced by the additions of BBA, BFA and SSBA on the mechanical and geotechnical properties of the mixtures.

5.4.1.- The effect of BA and SSBA on compaction parameters

The compaction curves for the untreated clayey soil and for the ten stabilized soil mixtures tested have been determined, as it is depicted in Figure 5.5. In addition, the maximum dry density (MDD) and optimum moisture content (OMC) for each mixture are presented in Table 5.4.

As it is shown in Table 5.4 and Figure 5.6.a, the untreated clayey soil had an MDD of 1.63g/cm3, for an OMC of 17.10%. Mixtures M1-L and M2-L-SSBA, apply the highest percentage of lime of all mixtures, 1.50%, which implied a shift to the right and down the compaction curve, presenting MDD of 1.58 g/cm3 and an OMC of approximately 19% in both cases. This behavior in the compaction properties of quicklime stabilized clayey soils is well known and has been described extensively in the literature. [41-45]

Table 5. 4. Maximum dry density and optimum moisture content of mixtures

Mixture	Maximum dry density (MDD) (g/cm^3)	Optimum moisture content (OMC) (%)
M0-Con	1.63	17.10
M1-L	1.58	19.30
M2-L-SSBA	1.58	19.00
M3-5BBA	1.59	19.90
M4-10BBA	1.56	16.80
M5-15BBA	1.54	20.90
M6-5BBA-SSBA	1.48	26.90
M7-5BFA	1.61	19.00
M8-10BFA	1.60	21.20
M9-15BFA	1.59	19.80
M10-5BFA-SSBA	1.50	23.40

However, the addition of lime together with the nanomaterials had not implied a significant change in soil compaction properties in the proportions applied in this study, as it has been observed in other studies on the application of nanosilica for stabilization of expansive soils [46], nevertheless, significant increases in OMC and decreases in MDD have been reported by applying higher percentages of nanosilica, above 1%, in combination with cement. [47]

The results in Table 5.4 and Figure 5.6.b show the effect of the addition of BBA as stabilizing material in different proportions. Analyzing the compaction curves and the MDD and OMC values for the mixtures M3-5BBA, M4-10BBA and M5-15BBA, a progressive decrease in MDD and a decrease in OMC could be observed, a similar behavior observed by different authors. [21-23]. Furthermore, analyzing at Figure 5.6.c, a similar behavior was observed with the addition of BFA with a decrease in MDD associated with an increase in the amount of ash. [24, 48].

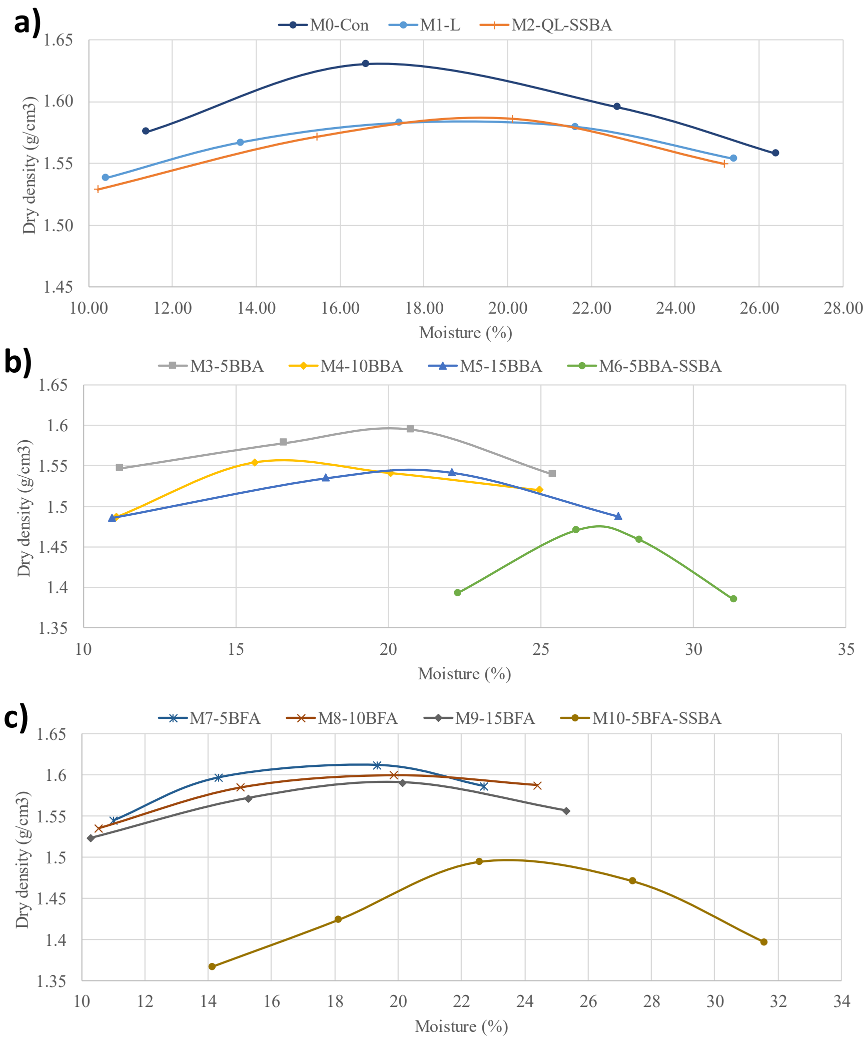

Fig. 5. 6. Moisture-dry density curves

The decrease in MDD of mixtures made with BBA and BFA was associated with the lower specific density of these materials compared to lime and soil, as well as the large additions of these materials. [48, 49].

Finally, analyzing the behavior of the M6-5BBA-SSBA and M10-5BFA-SSBA mixtures, a drastic decrease of the MDD and a significant increase of the OMC were noticed. This behavior may be due to pozzolanic reactions between the biomass ashes, high in Si and Ca, and the nanomaterials which increased the water demand and the OMC. The decrease in density was directly attributed to flocculation/aggregation and the formation of cementitious products. [30, 50]

5.4.2.- The effect of BA and SSBA on plasticity, swelling and collapse

The level of stabilization of a clayey soil may be assessed at an early stage by the reduction of plasticity, free swelling, and collapse potential. Table 5.5 presents the results obtained for all the stabilized soil mixtures for the three tests mentioned above.

Figure 5.7 depicts the plasticity chart with the classification proposed by the Unified Soil Classification System (USCS) and the results obtained for the ten mixtures analyzed. Additionally, Figure 5.8 shows the results obtained for free swelling and percentage collapse potential together with the limits established by Spanish regulations for their application in road layers and embankment fills. [32]

As it can be observed in Figure 5.7 and Figure 5.8, the untreated clayey soil was classified as a high plasticity clayey soil (CH), showing a high expansion and collapse potential, exceeding by double the limits allowed by the Spanish regulations for its application in road layers and embankment fillings.

However, for the ten stabilized mixtures, a reduction in plasticity was observed, all of them being classified as low plasticity silts or organic matter, in addition to a very marked reduction in expansion and collapse to within the limits for their application according to Spanish regulations, apart from mixture M5-15BBA, which was classified as high plasticity silt in addition to exceeding the expansion limit.

Table 5. 5. Plasticity, swelling and collapse results

Designation	Liquid Limit	Plastic limit	Index plasticity	Free swelling (%)	Collapse potential (%)
M0-Con	53	21.6	31.4	3.645	1.06
M1-L	48.5	30.9	17.6	0.08	0.31
M2-L-SSBA	43.1	33.9	9.6	0.06	0.39
M3-5BBA	45	30.39	14.61	0.29	0.54
M4-10BBA	48.3	31.9	16.4	1.22	0.19
M5-15BBA	51.1	29.5	21.6	2.88	0.355
M6-5BBA-SSBA	47.1	32.9	14.2	0.01	0.05
M7-5BFA	38.9	31.1	7.8	0.20	0.320
M8-10BFA	NP	NP	NP	0.34	0.130
M9-15BFA	NP	NP	NP	1.23	0.300
M10-5BFA-SSBA	43.1	35.9	7.2	0.04	0.135

As it can be seen in Table 5.5 and Figure 5.8, all ten stabilized mixtures allowed a reduction of the collapse potential to acceptable limits for application. All mixtures improved the soil macrotexture and workability through a flocculation process which allowed control over the collapse potential.

The addition of lime for soil stabilization typically led to a decrease in the plasticity index, primarily attributed to an increase in the plastic limit. This observation was well-documented in the literature. [12, 41]. When lime was combined with nano silane, it further reduced the plastic limit of the soil, thereby reducing its plasticity. However, this combined effect did not result in a significant change when compared to the addition of lime alone.

In the same way, the addition of 1.50% lime allowed control of the expansivity and collapse, reducing them from 3.65% to 0.08% and from 1.06% to 0.31%, respectively. Similarly, no relevant effect was observed on the expansion and collapse potential measurements after the addition of lime and nano silane, indicating the limited interaction between lime and the nanomaterials.

The addition of BBA had a moderate effect on the plasticity index, as it increased the plastic limit and reduced the liquid limit when combined with lime. However, the addition of 15% BBA without lime did not result in a substantial modification of the untreated soil.

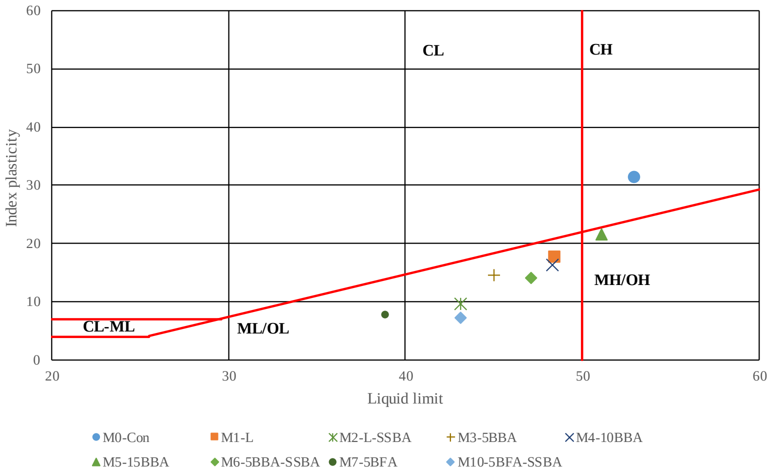

Fig. 5. 7. USCS classification chart and values of mixtures

Similar behavior was observed in the swelling evolution with the addition of BBA. The M3-5BBA mixture showed an expansion with a value of 0.29%, which increased to 2.88% for the M5-15BBA mixture. This fact could be attributed to the low reactive potential of BBA with the soil, leading to stabilization through a physical alteration of the soil properties due to the high percentages of BBA added. Similar behavior had been observed in previous studies where BBA from olive pruning burning and residues from the olive oil production industry were applied. [21, 23].

However, both the M6-5BBA-SSBA mixture and the M10-5BFA-SSBA mixture exhibited a similar reduction in plasticity and expansivity as the M2-L-SSBA mixture. This reduction was attributed to the interaction between the highly reactive silica from the nanomaterial and both the calcium and silicate components from the biomass ash. As a result, chemical stabilization occurred through the formation of CS and CSH gels, as well as flocculation processes. The literature describes these pozzolanic reactions involving the addition of silica-based nanomaterials interacting with cement components [28], as well as secondary reactions with Ca and aluminosilicate compounds found in clay minerals [30, 31]. These reactions were enhanced in the presence of residues containing high calcium and aluminosilicate content, such as fly ash and biomass bottom ash, improving the properties of the stabilized soil.

On the other hand, the effect of adding BFA on the plasticity index was analyzed, and a drastic reduction in plasticity was observed with an addition of 5%. With percentages of 10% and 15%, the plasticity was completely eliminated, even in the absence of lime. This reduction was attributed to the increased flocculation of the soil caused by the addition of BFA, resulting in a decrease in both plasticity and permeability [51]. The M10-5BFA-SSBA mixture exhibited a behavior similar to that of the M7-5BFA mixture but with slight modifications in the liquid limit and plastic limit due to the addition of SSBA, as observed in the M2-L-SSBA mixture.

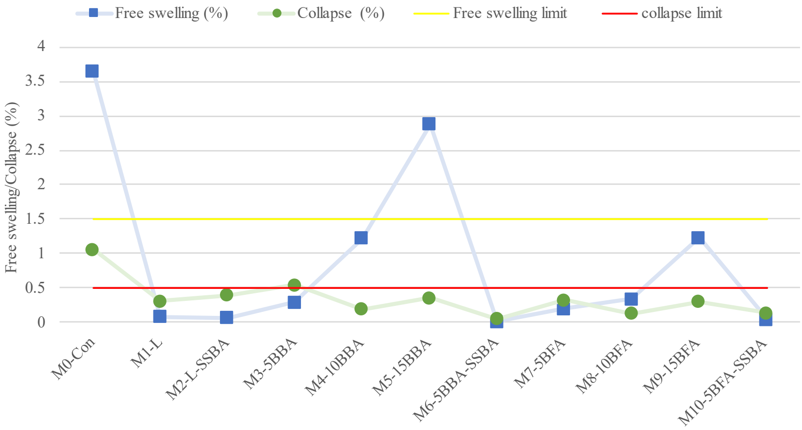

Fig. 5. 8. Free swelling and potential collapse of mixtures and limit of Spanish regulations

Analyzing the expansion of the mixtures, as shown in Figure 5.8, from M7-5BFA to M10-5BFA-SSBA, a similar behavior to that exhibited by BBA is observed. However, a reduction in expansion is noted, even in the sample M9-15BFA without the addition of lime, with a value of 1.23% which falls within the limits for application according to Spanish regulations.

This discrepancy in the trend between plasticity and expansion with an increasing amount of added BFA can be attributed to the different stabilization processes governing the development of each property. The reduction in plasticity and collapse is influenced by the modification of physical properties, specifically the flocculation of clay soil particles, which is promoted by BFA. Additionally, the modification of chemical properties occurs through an ion exchange process facilitated by the addition.

5.4.3.- The effect of BA and SSBA on bearing capacity

The CBR index is an indicator of the bearing capacity of soils for application in road layers, civil engineering and building works. Spanish regulations classify three types of stabilized soils according to their bearing capacity, type of binder and amount of binder (among other properties) [32]. For lime-stabilized soils, two types are distinguished: stabilized soil type 1 (S-EST1) with a minimum CBR of 6, and stabilized soil type 2 (S-EST2) with a minimum CBR of 12 to 7 days soaked.

Figure 5.9 depicts the CBR index results for the untreated soil and the stabilized soil samples tested at 7 and 28 days submerged, as well as the application limits according to Spanish regulations.

As seen in Figure 5.9, all ten stabilized samples demonstrated an increase in the bearing capacity compared to the untreated soil. However, mix M5-15BBA did not exhibit a sufficient increase in bearing capacity to meet the requirements for its application in road layers. Out of the ten mixes, nine surpassed the limit set by Spanish regulations. The M4-10BBA mix is classified as S-EST1, while the remaining mixes are categorized as S-EST2. These classifications indicate that the stabilized soil possesses a medium-high quality and is suitable for use as an esplanade in high-intensity road applications.

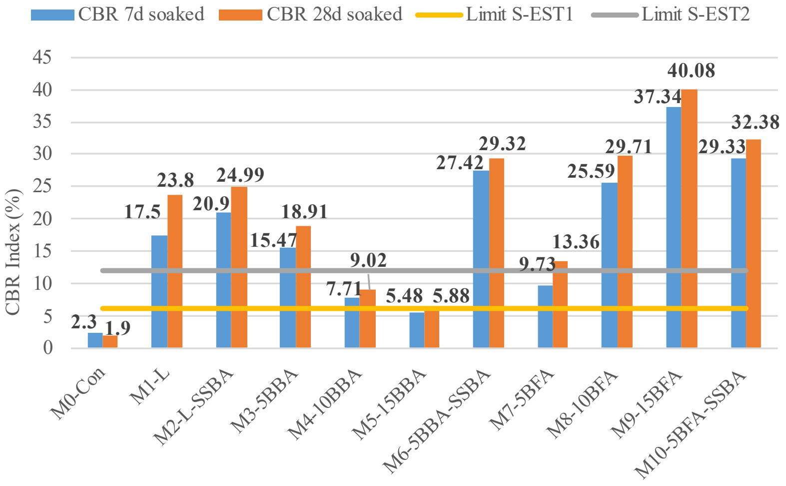

Fig. 5. 9. CBR index and Spanish regulation limit to stabilized soils

The M1-L mix exhibited an increase in the CBR index from 2.3% in the untreated soil to 17.5% after 7 days of curing with an addition of 1.50% lime. When SSBA was added without altering the lime content, a further increase in bearing capacity was observed. The curing period of 7 days resulted in an increase of up to 20.9%, while the 28-day curing period showed a higher improvement of 24.99% compared to the 23.8% value obtained for the M1-L mix. This behavior aligns with the stabilization results mentioned earlier, which can be attributed to the limited interaction between lime and the nanomaterials.

Similarly, significant increases were observed in the M6-5BBA-SSBA and M10-5BFA-SSBA mixtures compared to the M2-L-SSBA mixture. This can be attributed to the interaction between the amorphous nanosilica, and the calcium and silicate compounds present in both BA. The stabilization response of these mixtures aligns with the process described earlier.

However, if the behavior of adding BBA is analyzed, it was observed that the higher the percentage added and the lower the amount of lime, the bearing capacity decreases from 15.47% for the M3-5BBA mix to 5.48% for the M5-15BBA mix.

The opposite behavior was observed with the addition of BFA, showing an increase in bearing capacity from 9.73% for the M7-5BFA mix to 37.34% for the M9-15BFA mix. This increase in bearing capacity by addition of BFA has been described in the literature [35, 52]. It was attributed to both the stabilization of soils through the flocculation mechanics mentioned earlier and a self-hardening process typically exhibited by fly ash with high CaO content, which increased the strength of the treated soils [53].

5.4.4.- The effect of BA and SSBA on consolidated unconfined shear strength

After studying the compactness, stabilization and bearing capacity properties of the ten mixtures described in the previous sections, the triaxial cell test specimen rupture test was performed in consolidated undrained conditions (CU) with interstitial pressure measurement for the untreated soil and the 6 most representative samples, shown in Table 5.6.

Following the triaxial CU test, the stress-strain behavior of the stabilized soils was analyzed in greater detail compared to the unconfined uniaxial compression tests. This allowed for the determination of the effective and maximum shear strength parameters.

Figure 5.10 illustrates the triaxial CU results obtained for the untreated soil. Figure 5.10 a) shows the behavior of deviatoric stress versus deformation, Figure 5.10 b) the interstitial pressure development versus deformation, and Figure 5.10 c) the stress state and failure envelopes at both effective and maximum pressures.

Table 5. 6. Maximum and effective shear strength parameters of mixtures

Mixture	**Maximum parameters**		**Effective parameters**	
	φ (˚)	**c (KPa)**	**φ´ (˚)**	**c´ (KPa)**
M0-Con	21	55	28	35
M1-L	27	140	31	80
M2-L-SSBA	29	100	37	15
M3-5BBA	24	150	29	80
M6-5BBA-SSBA	25	105	30	45
M8-10BFA	33	55	36	0
M10-5BFA-SSBA	25	130	28	75

Figures 5.11, 5.12 and 5.13 show the plots of the deviatoric stress versus strain, the interstitial pressure development versus strain and the stress state and failure envelopes at the effective and maximum pressures, respectively, for the 6 stabilized specimens analyzed. In addition, Table 5.6 provides the shear strength parameters obtained from Figure 5.10.c) and 5.13.

The rupture curves in Figure 5.10a depict the behavior of the three untreated clayey soil specimens, consolidated under varying confining pressures. These curves exhibit plastic rupture characteristics. It was observed that as the confining pressure increased, a higher deviatoric stress was required to cause specimen rupture. The maximum deviatoric stress value of approximately 550 kPa was observed for the highest confining pressure of 300 kPa. No brittle failure was observed in any of the three cases; instead, the curves gradually approached a nearly horizontal slope with a slight tendency towards hardening. As a result, the rupture deformation was determined to be 12%

Regarding the interstitial pressure development illustrated in Figure 5.10.b for the three confining pressures applied, it was observed that the soil exhibited an increase in interstitial pressure throughout the test, in the samples subjected to 50 and 300 kPa, and during 70% of the test for the 150 kPa pressure. This increase in interstitial pressure indicated a tendency towards shrinkage in the soil mass subjected to rupture under different confining pressures, a behavior which is typically observed normally consolidated clays [54].

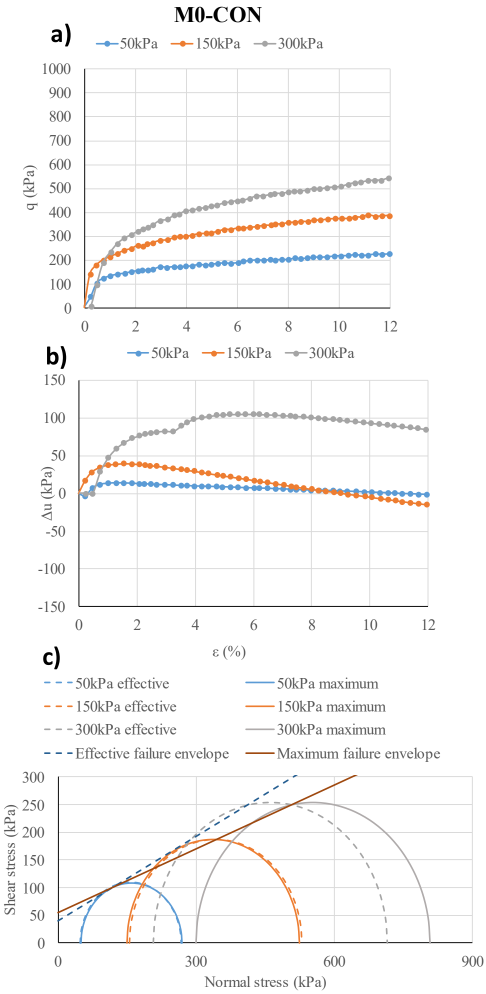

Fig. 5. 10. M0-Con triaxial test CU results; a) deviatoric stress – deformation relationship; b) Pore pressure variation – deformation relationship; c) shear stress – normal stress relationship and failure envelopes

Finally, Figure 5.10.c displays the stress state for the soil mass in the different consolidation states. Based on this figure, the maximum and effective shear strength parameters were extracted and presented in table. The sample had an internal friction angle value of 21 and a cohesion value of 55kPa in total stresses. These parameters showed a different behavior in effective stresses, with an increase of the internal friction angle up to a value of 28 and a reduction of the cohesion up to 35kPa.

Figure 5.11 illustrates the relationship between deviatoric stress and deformation for the six selected samples studied in the triaxial CU test. As it can be seen, the stabilization of the soil through the 6 selected samples implied a substantial increase in the strength of the soils under the three confining pressures.

Under the lowest confining pressure, 50kPa, an increase was observed from 200kPa, up to values between 300kPa (sample M8-10BFA), up to 600kPa (M3-5BBA) with intermediate values for the rest of the samples. For the confining pressures of 150 and 300 kPa, a similar behavior was observed with increases in strength of up to double the values presented by the untreated soil.

In addition to the increase in deviatoric strength of the stabilized samples, a change in their behavior was observed, indicating a tendency towards stiffening. The greatest increase in stress was observed at around 2% deformation for all cases. Some samples, such as M1-L under a confining pressure of 150 kPa, and M3-5BBA and M8-10BFA under a confining pressure of 50 kPa, exhibited eventual stress peaks, suggesting a transition towards brittle behavior. However, despite this trend, the mixtures did not display a clearly brittle behavior and instead showed a tendency to harden with increasing strain and the application of deviatoric stress. Therefore, the criterion of establishing failure at 12% strain was maintained

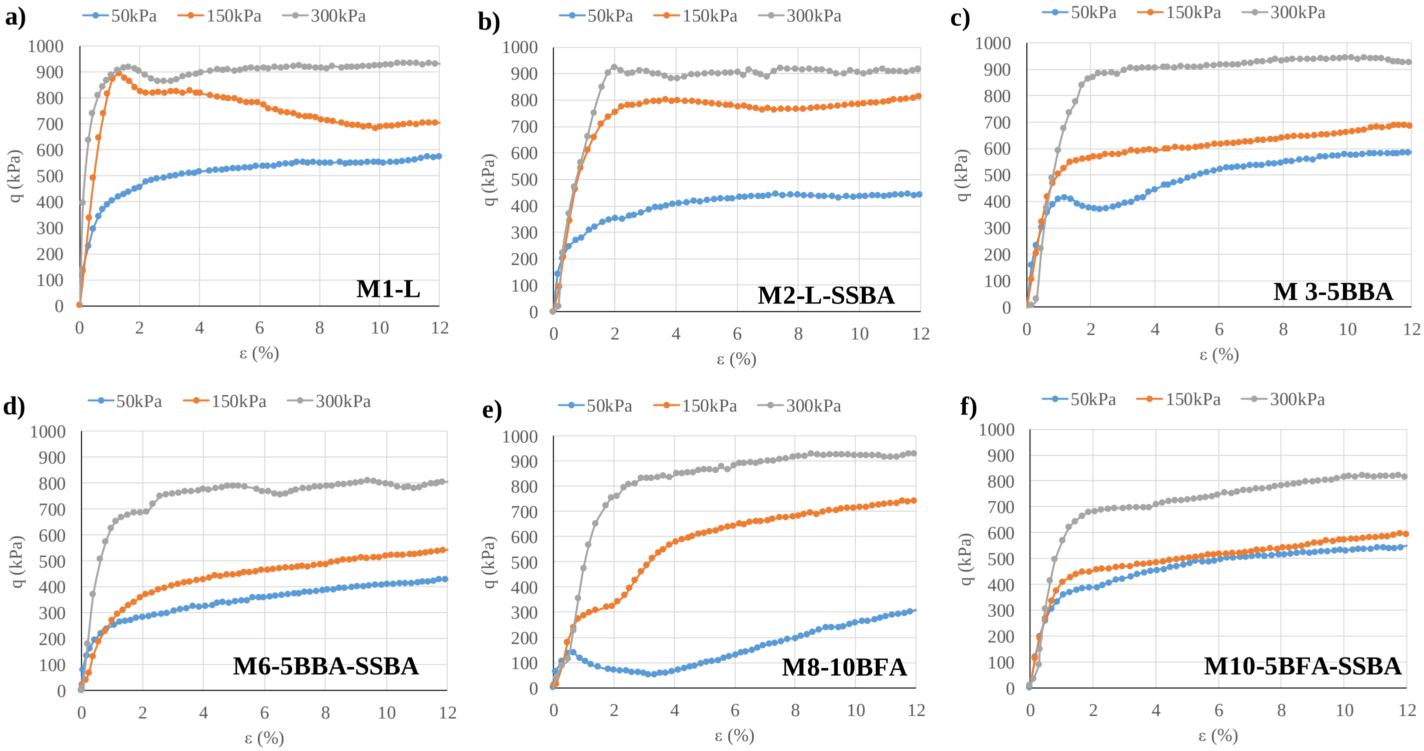

Fig. 5. 11. Triaxial test CU results: deviatoric stress – deformation relationship; a) M1-L mixture; b) M2-L-SSBA mixture; c) M3-5BBA mixture; d) M6-5BBA-SSBA mixture; e) M8-10BFA; f) M10-5BFA-SSBA

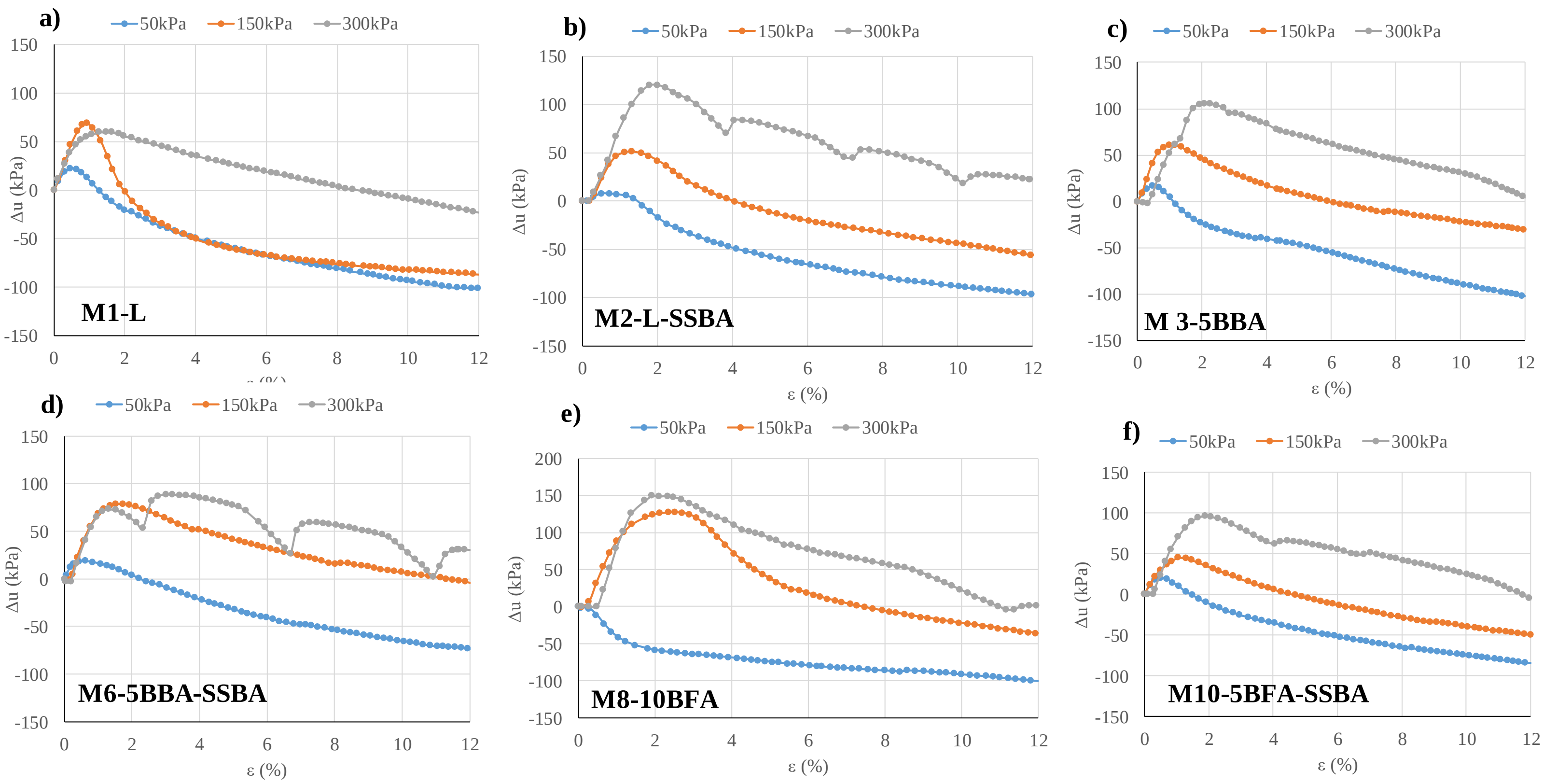

Fig. 5. 12. Triaxial test CU results: pore pressure variation – deformation relationship; a) M1-L mixture; b) M2-L-SSBA mixture; c) M3-5BBA mixture; d) M6-5BBA-SSBA mixture; e) M8-10BFA; f) M10-5BFA-SSBA

Regarding the development of the interstitial pressures, as shown in Figure 5.12, all the mixtures had modified their behavior with respect to the untreated soil. For the six mixtures analyzed, a shrinkage trend was shown from the beginning of the test up to approximately 2% deformation of greater magnitude for higher confining pressures. After the initial shrinkage, all mixtures exhibited a decrease of the interstitial pressure to negative values, thus showing an expansion behavior. Therefore, the deviatoric stresses of all three specimens continued to increase up to high deformations, as shown in Figure 5.11. This increase in deviatoric stresses was attributed to the decrease in interstitial pressures.

This behavior is reported in the literature typical of sandy soils [54] indicating that the clayey soil mass has undergone a change in its properties due to the processes of flocculation, formation of carbonates and CS and CSH gels, consistent with the results shown in the previous tests.

From the stress state and failure envelope plots shown in Figure 5.13, the shear strength parameters for the six stabilized samples were determined, considering both total and effective stresses. These parameters are presented in Table 5.6.

Similar to the untreated soil, the effective shear strength parameters showed a decrease in cohesion and an increase in the angle of internal friction, due to not considering the effect of interstitial pressures in the soil mass.

Analyzing the effective parameters, an increase in shear strength was observed for all the samples analyzed. Mixtures M1-L, M3-5BBA, M6-5BBA-SSBA and M10-5BFA-SSBA, slightly increased their internal friction angle but showed a sharp increase in the increase of cohesion, even in effective stresses. The M2-L-SSBA and M8-10BFA samples, however, showed a reduction in cohesion to show a very significant increase in the angle of internal friction.

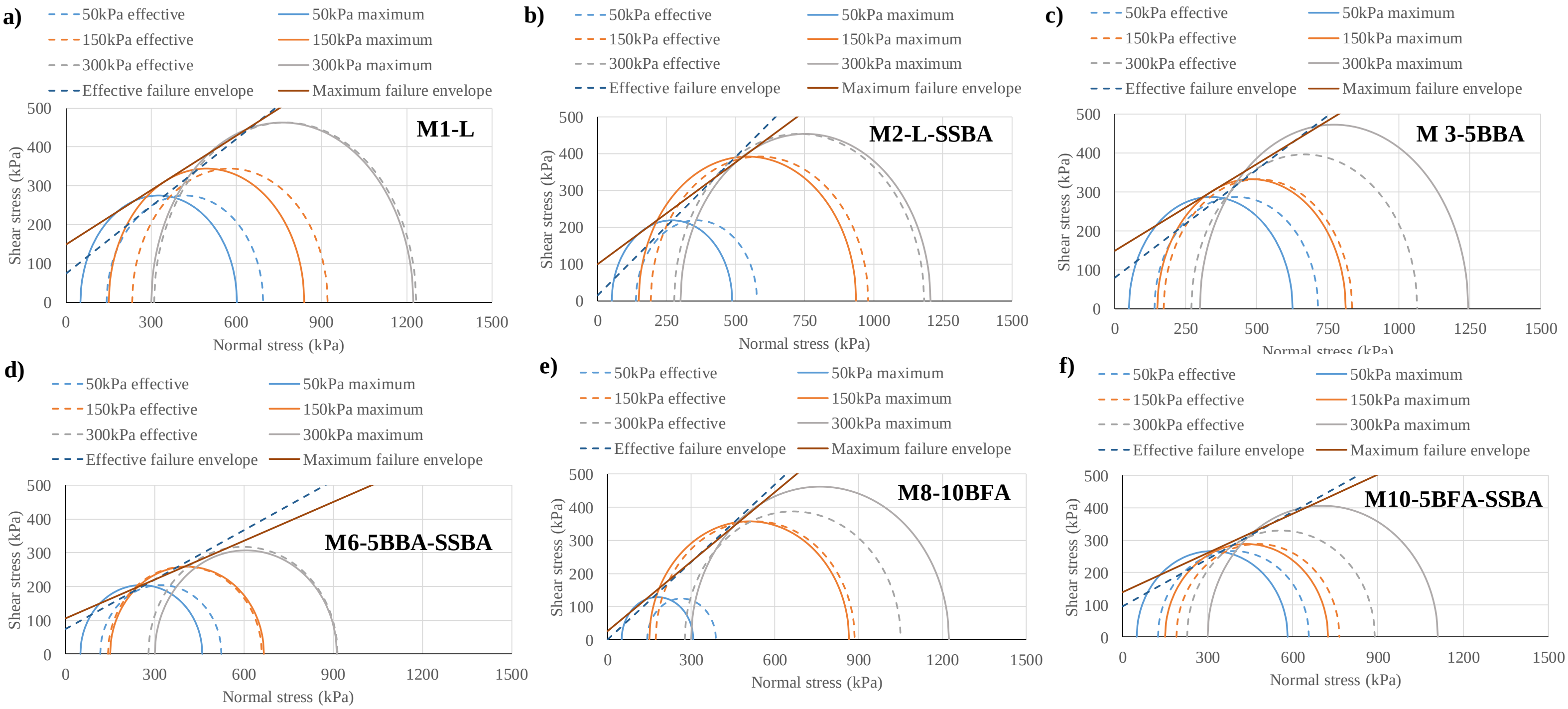

Fig. 5. 13. Triaxial test CU results: shear stress – normal stress relationship and failure envelopes; a) M1-L mixture; b) M2-L-SSBA mixture; c) M3-5BBA mixture; d) M6-5BBA-SSBA mixture; e) M8-10BFA; f) M10-5BFA-SSBA

5.4.5.- The effect of BA and SSBA on hydraulic conductivity

The hydraulic conductivity coefficient, k, was determined for the untreated soil sample and for the six most representative samples of the study, as performed in the triaxial CU test. The samples were compacted and stored for 7 days prior to testing with the flexible wall permeameter in a triaxial cell. The effective confining pressure, σ`c, was 100 kPa. The results of the flow rate curves for each sample are shown in Figure 5.14, which are related by the equation derived from Darcy's law to obtain the coefficient k shown in Table 5.7.

As it can be seen in Table 5.7, the permeability coefficient of the untreated soil was of the order of magnitude of 10-8, a typical value observed in clayey soils of the montmorillonite type, belonging to the smectite group [55, 56].

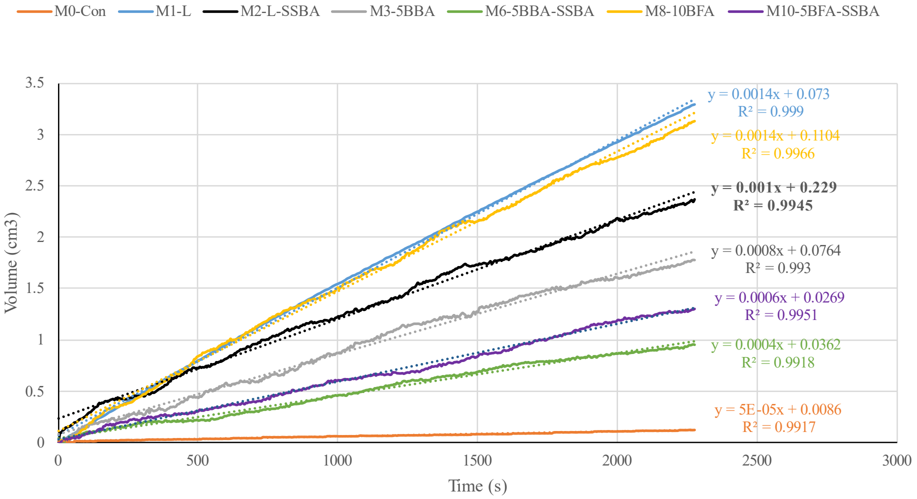

Fig. 5. 14. Volume – time relationship

A general increase in permeability coefficient was observed after seven days in the six samples analyzed. There was an increase in permeability of up to two orders of magnitude in the case of samples M1-L, M2-L-SSBA and M8-10BFA, with values between 6 and 9E-7 for both cases. The permeability coefficients for the remaining samples ranged between 2E-7 and 5E-7.

Table 5. 7. Permeability coefficient of mixtures

Mixture	k (cm/s)
M0-Con	3.28061E-08
M1-L	9.18571E-07
M2-L-SSBA	6.56122E-07
M3-5BBA	5.24898E-07
M6-5BBA-SSBA	2.62449E-07
M8-10BFA	9.18571E-07
M10-5BFA-SSBA	3.93673E-07

The large increase in permeability for samples M1-L and M2-L-SSBA was caused by the addition of lime, which caused a short-term increase in permeability [44]. In the case of sample M8-10BFA, the increase in permeability, related to the drop in plasticity due to the addition of 10% BFA, may be due to the hardening process of the fly ash. [53] .

The effect of the addition of SSBA on permeability at 7 days was insignificant, it could be associated with the hardening and stabilization process through pozzolanic reactions, which have a greater effect over time. [45].

5.5.- Conclusions

This study analyzed the effect of stabilizing an expansive clayey soil by adding lime, biomass bottom ash, biomass fly ash (derived from olive biomass) and silica-based nanomaterials. For this purpose, the geotechnical and engineering properties of different stabilized soil mixtures were studied and the following conclusions were drawn from the study:

1. The combined addition of nanosilica with lime for an expansive clayey soil stabilization resulted in a slight improvement in stabilization parameters and bearing capacity of the stabilized soils. However, there was no significant change observed in the compaction parameters.

2. The addition of biomass bottom ash did not exert a substantial stabilizing effect on compaction, stabilization or bearing capacity properties of the soils. However, it did lead to an improvement of the properties due to

the physical modification of the soil by the high replacement rates.

3. The addition of biomass fly ash resulted in improved the compaction, stabilization and bearing capacity properties of soils. This improvement can be attributed to flocculation reactions of the clayey soil and self-hardening processes associated with fly ash.

4. The interaction between nanosilica and biomass bottom and fly ash implied a very significant improvement of all analyzed parameters. The combination of the highly reactive silica from the nanomaterials in interaction with the calcium and silicates present in the biomass ashes promoted pozzolanic reactions and the formation of CS and CSH gels. This, in turn, reduced plasticity, expansion, and collapse of the soils while enhancing their bearing capacity and shear strength parameters.

5. According to the results obtained in the development of the triaxial CU test, an increase in the resistance to deflection stress of the mixtures stabilized with lime, biomass ash and nanomaterials was observed. This increase in strength implied an improvement in the shear strength parameters of the stabilized soil mass, widening its range of use for the construction of slopes and embankments.

6. All mixtures analyzed increased the hydraulic conductivity of soils, increasing their range of use and avoiding problems associated with water retention in civil engineering and road works. However, the improvement observed was similar in all the mixtures, except for the samples with the highest percentage of lime and biomass fly ash, being the mixtures that most modify the texture and workability of the soil. The samples additivities with nanosilica did not imply a significant increase in hydraulic conductivity at 7 days, although it was necessary to analyze in the long term to evaluate the effects of the full development of pozzolanic activity.

Based on this research, it can be concluded that percentages of 5% biomass ash, 0.50% lime and 0.056% nanosilica allow to reduce the amount of lime by up to 66%, to apply waste and by-products to improve the geotechnical and mechanical properties of the soil with respect to stabilization with lime alone. It represents a technical, economic, and ecological alternative to conventional materials, reducing the consumption of natural resources by utilizing recycled materials and promoting a sustainable circular economy model.

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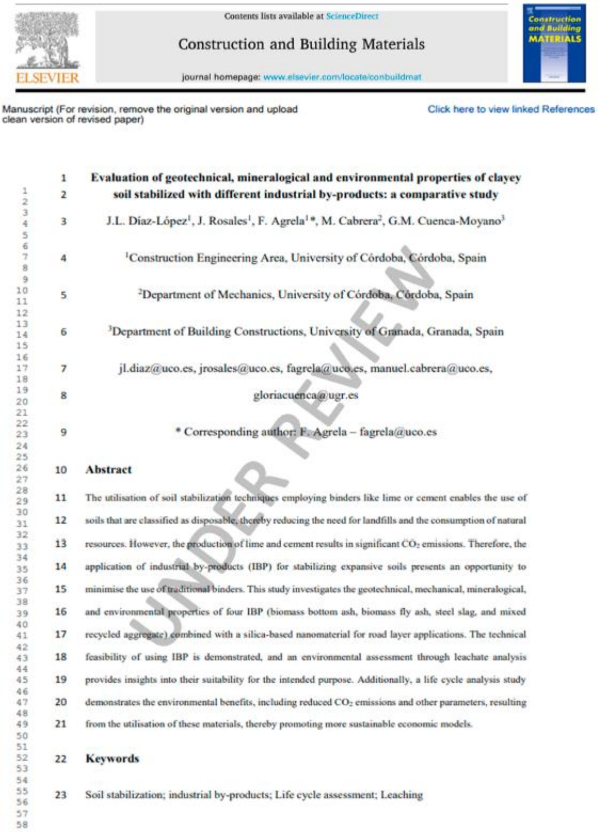
CAPÍTULO VI

EVALUATION OF GEOTECHNICAL, MINERALOGICAL AND ENVIRONMENTAL PROPERTIES OF CLAYEY SOIL STABILIZED WITH DIFFERENT INDUSTRIAL BY-PRODUCTS: A COMPARATIVE STUDY

Díaz-López, J.L., Rosales, J., Agrela, F., Cabrera, M., Cuenca-Moyano, G.M.



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Evaluation of geotechnical, mineralogical and environmental properties of clayey soil stabilized with different industrial by-products: a comparative study

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Abstract

The utilisation of soil stabilization techniques employing binders like lime or cement enables the use of soils that are classified as disposable, thereby reducing the need for landfills and the consumption of natural resources. However, the production of lime and cement results in significant CO_2 emissions. Therefore, the application of industrial by-products (IBP) for stabilizing expansive soils presents an opportunity to minimise the use of traditional binders. This study investigates the geotechnical, mechanical, mineralogical, and environmental properties of four IBP (biomass bottom ash, biomass fly ash, steel slag, and mixed recycled aggregate) combined with a silica-based nanomaterial for road layer applications. The technical feasibility of using IBP is demonstrated, and an environmental assessment through leachate analysis provides insights into their suitability for the intended purpose. Additionally, a life cycle analysis study demonstrates the environmental benefits, including reduced CO_2 emissions and other parameters, resulting from the utilisation of these materials, thereby promoting more sustainable economic models.

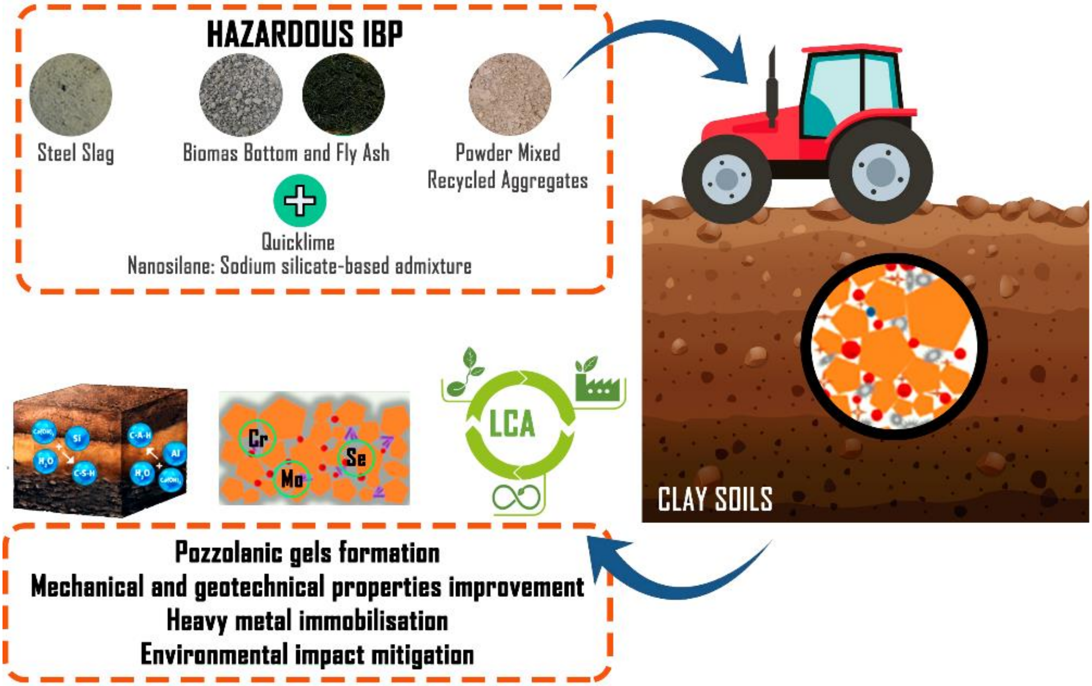
Keywords

Soil stabilization; industrial by-products; Life cycle assessment; Leaching

VI

EVALUATION OF GEOTECHNICAL, MINERALOGICAL AND ENVIRONMENTAL PROPERTIES OF CLAYEY SOIL STABILIZED WITH DIFFERENT INDUSTRIAL BY-PRODUCTS: A COMPARATIVE STUDY

Díaz-López, J.L., Rosales, J., Agrela, F., Cabrera, M Cuenca-Moyano, G.M. (2023). Evaluation of geotechnical, mineralogical and environmental properties of clayey soil stabilized with different industrial by-products: a comparative study. Construction and buildings materials. ***Under review***



Abstract

The utilisation of soil stabilization techniques employing binders like lime or cement enables the use of soils that are classified as disposable, thereby reducing the need for landfills and the consumption of natural resources. However, the production of lime and cement results in significant CO_2 emissions. Therefore, the application of industrial by-products (IBP) for stabilizing expansive soils presents an opportunity to minimise the use of traditional binders. This study investigates the geotechnical, mechanical, mineralogical, and environmental properties of four IBP (biomass bottom ash, biomass fly ash, steel slag, and mixed recycled aggregate) combined with a silica-based nanomaterial for road layer applications. The technical feasibility of using IBP is demonstrated, and an environmental assessment through leachate analysis provides insights into their suitability for the intended purpose. Additionally, a life cycle analysis study demonstrates the environmental benefits, including reduced CO_2 emissions and other parameters, resulting from the utilisation of these materials, thereby promoting more sustainable economic models.

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Soil stabilization; industrial by-products; Life cycle assessment; Leaching

6.1.- Introduction

Connectivity in rural areas is vital for improving quality of life and socioeconomic development. Road construction in rural areas is always hampered by geographical limitation and can often be costly and energy inefficient. One of the main objectives of soil stabilization is to improve site materials and create a solid and strong base and sub-base. Subgrade soils require demands due to the traffic loads to which they are subjected, solved by improving the soils with different forms of stabilization. The support must guarantee a good performance through a high bearing capacity; it must be insensitive to atmospheric agents and guarantee the stability of the foundation in the long term. To carry out soil stabilization, the most important aspect to take into account is the composition of the soil under study [1]. A soil stabilization process is mainly based on a preconsolidation, drainage or by the addition of products such as cement or lime.

The use of soil stabilizing materials leads to improved soil properties such as increased strength, reduced plasticity and swelling, increased stiffness and increased toughness [2-6]. Although the use of this type of materials in soil stabilization has historically resulted in adequate solutions, it must be considered that the production of cement and lime generates high environmental impacts, mainly due to the consumption of natural resources, the high cost of production and the high rates of CO_2 emissions [7-9]. Due to the negative environmental impact caused by the use of cement and lime in soil stabilization, studies have been underway for years on the application of new binding materials from waste or by-products as an alternative. An essential characteristic of these types of materials is that they present pozzolanic reactions, that is, they self-harden in the presence of water generating cementitious gels [9] or they have a high content of free lime (CaO) that reacts with alumina and silica oxide upon hydration and generates cement-like products [10].

Chemical stabilization of soils has led to research in recent years into the possibility of applying nanomaterials as alternative materials to lime and cement for the improvement of soil properties. Nanomaterials, due to their small particle size, have a higher percentage of surface atoms and molecules, which in combination with the soil, provide changes in physical properties mainly due to the increase in the surface/volume ratio and the quantum effects that occur in combination with the soil [11]. One of the main advantages of using nanomaterials such as nano-silica, nano-carbon, nano-lime, nano-alumina or nano-clay is that with a small amount high interactions are achieved and lead to specific improvements in soil properties. These include improving unconfined compressive strength [12-16], variation of soil physical properties such as plasticity, pH, density or conductivity [17, 18], reducing layer thickness and soil expansiveness [19] and improving soil bearing capacity due to improved CBR index, modulus of elasticity, shear strength and angle of cohesion and internal friction [20-22].

These improvements in soil bearing capacity through the use of nanomaterials have been corroborated in the actual execution of experimental sections [23, 24]. The use of nanomaterials improves the mechanical behaviour of stabilized soils and allows a reduction in the thickness of the pavement layer, obtaining adequate soil bearing capacity results.

The combined use of waste and nanomaterials in soil stabilization is an alternative to the common stabilizers used, such as lime and cement. This combination leads to the use of materials with a low carbon footprint and improves the bearing capacity of the soil. Studies have shown that the combination of both leads to an improvement in soil properties.

There are few studies on the combined application of waste and nanomaterials in soil stabilization. Biswas and Sarkar [25] showed that the combined use of fly ash and a nanochemical solution led to an increase in the bearing capacity of the soil, a decrease in the plasticity index and an increase in the CBR index. This improvement in properties was mainly due to the cementitious properties of the ash and the formation of Si-O-Si due to the use of nanomaterials that created an impermeable surface in the soil. Wang et al. [26] studied a stabilizer composed of slag, nanosilica and quicklime that showed gelling properties improving soil properties compared to a traditional stabilizer such as cement.

Therefore, it is important to evaluate that the residues used have a high content of oxygen (O) and carbon (C), with high peaks of silicon (Si), aluminum (Al), magnesium (Mg), calcium (Ca) and iron (Fe) generating oxides (SiO_2, Al_2O_3, MgO, CaO, and Fe_2O_3) that cause pozzolanic reactions [27] and lead to the formation of cementitious composites such as calcium silicate hydrate (CaO) and Fe_2O_3, Al_2O_3, MgO, CaO, and Fe_2O_3) that cause pozzolanic reactions and lead to the formation of cementitious compounds such as calcium silicate hydrate (CSH) and calcium aluminate hydrate (CAH) [28-30] and are useful for use in soil stabilization [31].

For this reason, this study proposes the combination of different by-products with a composition that shows their high pozzolanic capacity, such as mixed recycled aggregates (MRA) and biomass ashes (BA) with nanomaterials. MRA have been previously studied and their potential for use in soil stabilization has been proven. Cabalar et al. [32] found that MRA with a suitable spray treatment in combination with expansive clay soils contributed to a reduction in the plasticity and swelling index and an increase in the resilient modulus. Sabat [33] found that powdered MRA improved the shear strength, increased the CBR index values and the unconfined compressive strength of the soil. These improvements in soil bearing capacity are mainly due to the pozzolanic capacity of the waste, as is the case with the application of BA. Cabrera et al. [34] and P. Galvín et al. [35] reported that the addition of biomass bottom ash in all mixtures improved the bearing capacity, mainly due to the high calcium content which increased the pozzolanic activity.

Having proven the pozzolanic capacity of the waste and the improvement produced by the introduction of nanomaterials in expansive soils, it is necessary to study the combination of both. For this reason, this study proposes the combination of waste with a composition that shows its high pozzolanic capacity, such as MRA and BBA, with nanomaterials to improve the properties of expansive soils.

The disadvantage of the use of waste as a chemical soil stabilizer is the toxicity in the soil because it can change the pH of the soil and contaminate groundwater and soils. For this reason, it is necessary to evaluate the leaching of heavy metals that may result from the use of a waste in combination with soil. The residues themselves can have high heavy metal values [36, 37]. However, in combination with soil and nanomaterials, C-S-H and C-A-H gels are created that lead to an immobilization of heavy metals within a cementitious matrix [38, 39].

Therefore, the benefit of using waste and nanomaterials as a substitute for lime and cement in soil stabilization is clear, as it provides an environmental benefit, although it is necessary to quantify this benefit using appropriate tools. Life Cycle Assessment (LCA) quantitatively provides the environmental benefit caused by the application of wastes in soil stabilization and how CO2 emissions are reduced compared to the use of traditional stabilizers. Lime production accounts for more than 75% of energy consumption and CO2 gas emissions in soil stabilization techniques [40]. The use of waste and nanomaterials drastically reduces these adverse environmental effects. Rosales et al. [24] found that the use of nanomaterials in soil stabilization reduced CO2 emissions by up to 20% compared to soil stabilized with lime. In relation to the use of waste as an alternative to lime in soil stabilization, Perri et al. [41] found through a LCA analysis that the use of lignin reduces up to four times the impacts related to the use of lime as a stabilizer. Quintero et al. (2017) [42] tested the environmental impact caused by the application of 2 residues (fly ash and brick dust) as a substitute for lime to stabilize soils, showing that brick dust reduced the Global-warming potential (GWP) index by 25% and fly ash by 18% compared to lime stabilization.

Considering the background, the general objective of this study is to determine the improvement of mechanical properties in stabilized soils by combining by-products such as biomass ash and mixed recycled aggregate with a highly reactive nanomaterial from the morphological and mineralogical point of view. The influence of these new stabilizers on the medium-term behaviour of the soil is evaluated, checking the engineering properties through different geotechnical tests, the chemical composition and internal microstructure of the manufactured mixtures, the influence of the application of waste and nanomaterials as soil stabilization on the emission of heavy metals through leaching studies and the evaluation of the environmental impacts caused through a LCA study.

6.2.- Materials and experimental program

6.2.1.- Raw materials and Industrial by-products

The raw materials used in the study were clay (CS), quicklime (QL), bottom ash and fly ash from biomass combustion (BBA and BFA respectively), electric arc furnance steel slag (SS), a silica-based nanomaterial (SSBA) and pulverised mixed recycled aggregate (pMRA). The main physico-chemical properties of raw materials and IBP are shown in Table 6.1.

6.2.1.1.- Clay soil (CS)

Clayey soil (CS) analysed in the present work is called marly soil, a type of soil that is typical in the south-eastern Iberian Peninsula. The sample was collected in Jaen, Andalusia, Spain. This soil has been studied in previous studies [23, 24] and it was mainly composed of calcite, quartz as non-clayey mineral and montmorillonite as clayey mineral. It is highly plastic, expansive and has a very low bearing capacity. Due to its properties, it is a soil that cannot be used in the execution of subgrade according to Spanish regulations, PG-3, and previous studies carried out

6.2.1.2.- Quicklime (QL)

Quicklime is an oxide of lime, obtained from the firing of a high purity limestone rock. It has a high reactivity, so for some uses in construction it is recommended to slake it beforehand with water. The lime is commercial type CL-90-Q according to the Spanish standard UNE-EN 459-1 (2002b).

6.2.1.3.- Biomass bottom ash and biomass fly ash (BBA and BFA)

Two types of biomass ash have been applied in this study, biomass bottom ash (BBA) and biomass fly ash (BFA), both obtained from the biomass combustion thermal power plant Sacyr Valoriza SA, located in the town of Linares, province of Jaén.

Biomass bottom ash is a residue generated during biomass combustion, collected at the bottom of combustion boilers. It is generated in the highest proportion. Biomass fly ash is the fine particles produced during combustion, which are carried away by the plant's flue gases and collected in the stack filters.

Table 6. 1. Physical and chemical porperties of materials

Properties	**CS**	**QL**	**SSBA**	**BBA**	**BFA**	**SS**	**pMRA**
Grain saiz distribution							
> 4 mm (Gravel size) (%)	44.8	-	-	19.7	0	0	0
0.063-4mm (Sand size) (%)	53.7	-	-	69.8	34.13	32.71	15.7
<0.063 mm (Silt and clay size) (%)	1.5	-	-	10.5	65.87	67.29	84.3
Organic matter content (%)	0.27	0.00	0.00	1.51	0.14	0	0.04
Water-soluble sulphate (% SO_3)	0	0.00	0.00	0	2.55	0	1.3
Main components XRF (%)							
P	0.08	0.02	-	0.1	0.12	0.10	0.2
Si	25.57	1.11	24.20	15.75	3.74	30.60	27.32
Ca	14.16	40.9	0.28	15.75	8.18	5.47	15.8
Al	2.69	0.78	0.18	1.6	0.69	6.02	2.08
S	0.03	0.24	0.02	0.11	2.19	0.01	1.3
K	1.66	0.06	0.03	11.67	45.1	2.10	7.65
Mg	1.37	8.3	0.15	3.59	1.97	1.00	1.02
Na	-	-	12.10	-	0	-	-
Fe	2.42	0.15	0.06	1.76	0.51	4.56	3.06

As shown in Table 6.1, BBA had a coarse grain size, equivalent to that of gravel, with maximum sizes up to 80 mm. The BBA had a high percentage of organic matter, due to unburned residues produced during combustion in power plant boilers.

BFA had a fine particle size distribution, with more than 65% of its mass having a size of less than 63 microns. They had a low amount of organic matter, as well as a high percentage of water-soluble sulphates and a high potassium composition.

6.2.1.4.- steel slag (SS)

The steel slag (SS) used in this study was waste generated in the electric arc furnace steelworks, for the manufacture of common steel, and was collected from the Siderúrgica Sevillana SA plant, located in Alcalá de Guadaíra, Andalusia, Spain.

The steelmaking process in the electric arc furnace steelworks consists of two stages. The first stage corresponds to the melting of the raw materials and the second stage to the refining of the materials produced, including processes such as deoxidation. The slag analysed in this study is a residue resulting from the deoxidation stage.

As shown in Table 6.1, the unprocessed SS had a small particle size, with 67% of the particles smaller than 63 microns. At the same time, SS presented a composition with high percentages of Si, followed by other elements such as Ca, Al or Fe. The composition of the SS analysed showed higher Si values than those typically analysed by Spanish organisations [43].

6.2.1.5.- Nanosilane: Sodium silicate-based admixture (SSBA)

The nanomaterial used in this study is a silica-based liquid solution (called SSBA) for soil stabilization, in conjunction with lime or cement.

As shown in Table 6.1, it is mainly composed of silicon and sodium and as shown in previous studies [23, 24] had a mainly amorphous mineralogy, with light sodium silicate compounds found. This amorphous mineralogy, together with its high fineness, makes SSBA a very reactive material, which potentiated pozzolanic reactions with clays.

6.2.1.6.- Powdered mixed recycled aggregates (pMRA)

The aggregate used came from the company GECORSA, located in Cordoba, Andalusia (Spain). Mixed recycled aggregate has been classified according EN 933-11 normative. The concrete values (Rc) are 43.7 %, aggregates values (Ru) are 33.8 % and ceramic values (Rb) are 20.9 %.

The mixed recycled aggregate was crushed and screened, obtaining approximately 85% of particles smaller than 0.063 mm. This material was called powdered mixed recycled aggregate (pMRA)

A study of the physical and chemical properties of the waste after crushing and screening and processes was carried out and the results are shown in Table 6.1, indicating a composition rich in silica and calcium, consistent with previous studies [44].

6.2.2.- Stabilized soil mixtures

This section shows the proportions of each material to be used for the different stabilizing mixtures. Table 6.2 shows the dosages of each material, and the designated nomenclature.

Table 6. 2. Mix proportion of stabilized soil

Designation	Mixture	Mix proportions (%)						
		CS	QL	BBA	BFA	SS	pMRA	SSBA
M0-Con	CS	100	-	-	-	-	-	-
M1-1.5QL	CS+ 1.5% QL	100	1.5	-	-	-	-	-
M2-1.5QL-SSBA	CS + 1.5%QL + SSBA	100	1.5	-	-	-	-	0.056
M3-5BBA-1QL	CS + 5%BBA + 1%QL	100	1	5	-	-	-	-
M4-5BBA-0.5QL-SSBA	CS + 5%BBA+0.5%QL + SSBA	100	0.5	5	-	-	-	0.056
M5-5BFA-1QL	CS + 5%BFA + 1%QL	100	1	-	5	-	-	-
M6-5BFA-0.5QL-SSBA	CS + 5%BFA+0.5%QL + SSBA	100	0.5	-	5	-	-	0.056
M7-5SS-1QL	CS + 5%SS + 1%QL	100	1	-	-	5	-	-
M8-5SS-0.5QL-SSBA	CS + 5%SS + 0.5%QL + SSBA	100	0.5	-	-	5	-	0.056
M9-5pMRA-1QL	CS + 5%pMRA + 1%QL	100	1	-	-	-	5	-
M10-5pMRA-0.5QL-SSBA	CS + 5%pMRA + 0.5%QL + SSBA	100	0.5	-	-	-	5	0.056

The mixtures were made by sieving the BBA and SS below 1 mm and the pMRA was crushed and sieved to achieve a particle size with a high percentage below 0.063 mm. All samples were oven-dried at a temperature of 60°C for 24 hours.

6.2.3.- Experimental program

This section describes the experimental methods applied in this work. The experimental methods applied are divided into four phases:

Phase 1: Study of the mechanical and geotechnical properties of stabilized mixtures

Modified Proctor UNE 103-501-94

The Modified Proctor test is a test to measure the degree of compaction of a soil. The more compacted it is, the lower the number of pores will be, so the smaller the changes will depend on the humidity and moisture of the soil, and therefore will be able to support greater loads. The Proctor test gives us the maximum density, in gr/cm3, corresponding to an optimum moisture percentage.

California Bearing capacity (CBR) UNE-EN 103-502

The CBR test measures the bearing capacity of a soil and is used to evaluate the quality of the soil for subgrade, subbase and pavement base. It is carried out under controlled Modified Proctor conditions of moisture and density and the value obtained indicates the ratio between the pressure required for the piston to penetrate the soil to a certain depth, and the pressure corresponding to that same penetration in a standard sample of crushed gravel.

Plasticity limits

The liquid limit (water content at which the behaviour of a clayey soil changes from a plastic to a liquid state), the plastic limit (moisture content at which a soil changes from a plastic to a semi-solid consistency as its moisture content decreases) and the plasticity index of the mixtures were determined according to the UNE-EN ISO 17892-12:2019 standard.

Free swelling and percentage potential for collapse in an oedometric cell

The determination of free swelling is regulated by the UNE 103 601-96 standard. It consists of the wetting of the soil allowing the vertical expansion of the specimen in the laterally confined soil cell to avoid horizontal deformations.

The percentage collapse potential according to UNE 103406:2006, is the collapse value determined, for a vertical pressure of 200 kPa, as the percentage decrease in height experienced by the specimen when flooded, once equilibrium is reached under the action of the selected vertical pressure, with respect to the initial height of the specimen.

Phase 2: Morphological and mineralogical study of stabilized blend

X-ray diffraction

It is one of the non-destructive techniques that allows the identification of the different phases in the sample, as well as the structural and microstructural characterisation of solids. The X-ray diffraction study was carried out on a BRUKER Theta-Theta model D8 Advance X-ray diffractometer without monochromator and 2.2 KW Cu anode. The diffractogram of crystalline powder was recorded from 5º to 70º. The current and voltage applied to the X-ray generator tube was 30 mA and 40 Kv and a variable divergence slit of 6 mm.

Scanning Electron Microscope (SEM)

The scanning electron microscope is a very versatile instrument, allowing the observation and surface characterisation of materials, providing morphological and chemical composition information quickly, efficiently and simultaneously of the analysed material. Its versatility is given by its high resolution (from 20 to 50 Å) and three-dimensional appearance of the images. The microscope used was a GEMINI High Resolution Field Emission Scanning Electron Microscope (FESEM) CARL ZEISS

Phase 3: Environmental assessment study of raw materials and stabilized blends

The industrial by-products used in the study may contain heavy metals that are toxic to the environment. The process of dissolution and transport of these components is called leaching. When the materials under study are applied outdoors, rainwater, surface water or groundwater can be responsible for leaching processes to occur. If the concentration of a leached toxic component is very high, a potential threat to the environment occurs. Leaching tests are indispensable for the feasibility of using any by-product as a building material. In this phase, the EN 12457- 4: 2003 compliance test for raw materials and the EN 14405:2017 percolation test for stabilized mixtures and raw materials were carried out.

Compliance test EN 12457- 4: 2003

The conformance batch test (UNE-EN 12457-4:2003) for the basic characterisation of the leaching levels of materials with a solid liquid ratio of 10 L/kg. This two-step method uses a suspension of 90 g dry mass of material (particle size < 4 mm) with 900 g deionised water and the suspension is then stirred for 18 ± 0.5 h. The solution is then extracted from the solution and mixed with water. Finally, the solution is extracted, filtered and analysed.

The leachate concentrations obtained are compared with the legal limit of the Landfill Directive to be classified according to their polluting potential.

Table 6. 3. Limit values according to Landfill Directive 2003/33/EC

	Inert Limit		***Non-hazardous limit***		***Hazardous limit***	
	Compliance test (mg/kg)	*Percolation test (mg/l)*	*Compliance test (mg/kg)*	*Percolation test (mg/l)*	*Compliance test (mg/kg)*	*Percolation test (mg/l)*
Cr	0.5	0.1	10	2.5	70	15
Ni	0.4	0.12	10	3	40	12
Cu	2º	0.6	50	30	100	60
Zn	4	1.2	50	15	200	60
As	0.5	0.06	2	0.3	25	3
Se	0.1	0.04	0.5	0.2	7	3
Mo	0.5	0.2	10	3.5	30	10
Cd	0.04	0.02	1	0.3	5	1.7
Sb	0.06	0.1	0.7	0.15	5	1
Ba	20	4	100	20	300	60
Hg	0.01	0.002	0.2	0.03	2	0.3
Pb	0.5	0.15	10	3	50	15

Percolation test EN 14405:2017

The test is performed using a dynamic seepage column to determine the leaching (release) for a liquid/solid (L/S) ratio of 0.1.

The column determines the mobility and availability of the contained elements that may be released into the environment and have an adverse environmental impact. The column is filled with the material under study in dynamically compacted layers. Once filled, the fluid (eluent) is circulated until it is saturated. Once equilibrium is reached, the eluent is pumped countercurrent at a given flow rate and leachate fractions are removed as successive liquid/solid (L/S) ratios are reached.

The directive includes limit values for an L/S ratio: 0.1, therefore only the values for this ratio will be given in this report. Table 6.3 shows the limit values according to Landfill Directive 2003/33/EC.

Phase 4: Life cycle assessment of in-situ stabilized soil materials and mixes

LCA is defined in the standards ISO 14040 [45] and ISO 14044 [46] as the collection and evaluation of the inputs, outputs and potential environmental impacts of a product system throughout its life cycle. The object of this LCA study was to determine and compare the environmental impacts associated with the execution of eleven experimental mixtures according to the dosages listed in Table 6.2. For this, and according to the methodology established in ISO 14040 [45] in this section phases 1 and 2 of the LCA are described, that is, the objective and scope are established (phase 1) and the life cycle inventory is developed (phase 2). The life cycle impact assessment (phase 3) and interpretation (phase 4) are developed in the Results and Discussion Section.

The selected functional unit corresponded to an experimental section of 100 m long and 8 m wide with a thickness of 30 cm.

System boundaries included materials production, (CS, QL, SSBA, BBA, BFA, SS, pMRA and water), the transport and execution of the experimental road section, that is, the LCA was limited from cradle to gate. The following stages were included in the system boundaries:

- Materials stage: The production of the component materials of the mixtures: CS, QL, SSBA, BBA, BFA, SS, pMRA and water.
- Transport stage: The transport of materials from the manufacturing site to the mixing area. The transportation distances considered were: (i) 0.1 km for water; and (ii) 100 km for the rest of the materials, except for CS that was obtained on site.
- Execution stage: The construction of the experimental mixtures of stabilized clayey soil through the following activities: preparation of the land, spreading and mixing of the materials, irrigation, leveling, and finally, compaction.

The life cycle inventory was developed by collecting the most important and influential inputs and outputs of the evaluated processes in the system. In this way, the primary data provided by the producers of the materials and the machinery required for the in-situ execution of the stabilized soil mixtures were collected. As secondary data, and to establish the inventory of generic materials, energy and transport processes, the processes of the Ecoinvent V3.8 (cut-off) database were used [47]. The characteristics of the equipment and processes included within the system boundaries are listed in Table 6.4.

Table 6. 4. Characteristics of the equipment and processes used in experimental mixtures of stabilized soils

LCA stage	*Process*	*Equipment*	*Amount*	*Power (kW)*	*Production (t/h)*	*Operation time (h)*	*Electrical consumption (kWh/t)*	*Distance (km)*
Materials								
CS	Clay, unspecified, in ground							
	Extraction	Rotavator	1	-	-	0.00245	-	-
QL	Lime, hydrated, loose weight (CH/ES) production, Cut off, U, (Ecoinvent v3.8).							-
SSBA	Sodium silicate, solid (RoW) Cut-off, U (Ecoinvent v3.8, Rosales et al., 2020).							-
	Sulfuric acid (RoW) Cut-off, U (Ecoinvent v3.8, Rosales et al., 2020).							-
	Tap water (RoW) Cut-off, U (Ecoinvent v3.8).							-
BBA	Handling/Transport	Shovel loader	2	-	-	0.006	-	0.03
BFA	Transport	Redler	1	36.8	1.5	-	2.45	-
SS	Handling/Transport	Shovel loader	1	-	35.90	0.04	-	0.2
	Shutdown	Tap water	2	-	-	-	-	-
	Handling	Overband	1	3.68	145.75	-	0.02525	-
		Conveyor belt, 5 m	2	3.68	145.75	-	0.02525	-
	Screening	Vibrating screen	1	18.50	225	-	0.0822	-
pMRA								
	Handling/Transport	Shovel loader	1	-	-	0.02	-	0.1
		Shovel loader	1	-	100	0.01	-	0.05
	Handling	Overband	2	3.68	108.91	-	0.0338	-
		Blower	1	14	144.73	-	0.0967	-
		Vibrating plate	1	3	80	-	0.0375	-
		Conveyor belt, 15 m	1	7.36	148.51	-	0.0496	-
		Conveyor belt, 10 m	1	7.36	108.91	-	0.0676	-
		Conveyor belt, 5 m	5	4	108.91	-	0.0367	-
	Screening	Vibrating screen	4	22.08	250	-	0.0883	-
	Crushing	Jaw crusher	1	160	325	-	0.4920	-
		Impact mill	1	75	250	-	0.3000	-
		Ball mill	1	15	4	3	11.25	-
Water	Tap water (Europe without Switzerland), tap water production, conventional treatment, Cut-off, U (Ecoinvent v3.8).							
Transport								
CS distribution	Transport, freight, lorry 7.5-16 metric ton, EURO4 (RER), transport, freight, lorry 7.5-16 metric ton, EURO4, Cut-off, U (Ecoinvent v3.8).							-
QL distribution SSBA distribution BBA distribution BFA distribution SS distribution pMRA distribution	Transport, freight, lorry 7.5-16 metric ton, EURO4 (RER), transport, freight, lorry 7.5-16 metric ton, EURO4, Cut-off, U (Ecoinvent v3.8).							100
Water distribution	Transport, freight, lorry 7.5-16 metric ton, EURO4 (RER), transport, freight, lorry 7.5-16 metric ton, EURO4, Cut-off, U (Ecoinvent v3.8).							0.1
Execution								
Mixtures construction	Scarified	Rotavator	1	-	-	1	-	-
	Extended	Panning tractor	1	-	-	0.5	-	-
	Mixed	Rotavator	1	-	-	1	-	-
	Irrigation	Tank truck	1	-	-	0.09	-	-
	Leveling	Motor grader	1	-	-	0.5	-	-
	Compacted	Compactor	1	-	-	0.5	-	-

Data collected in the inventory were incorporated into the SimaPro software [48] and processed with the characterisation factors of the CML-IA baseline V3.07/EU25 method [49]. The impact evaluation was carried out for the categories: Abiotic depletion of elements (ADe), Abiotic depletion of fossil fuels (ADf), Global warming (GW), Ozone layer depletion (ODP), Photochemical oxidation (POF), Acidification (A) and Eutrophication (E). Initially, the environmental impacts generated by the production of materials, residues and by-products used in the formation of the mixtures were determined. Once the production phase of the materials was analysed, the environmental impacts associated with the stabilization of soils with different materials were determined.

6.3.- Results and discussion

This section shows the results obtained in the tests carried out during the four stages of the experimental programme shown above.

In addition, a discussion of the results was carried out to analyse the improvements in mechanical and geotechnical properties due to the addition of industrial by-products (IBP) and their combination with silica-based nanomaterials to stabilized soil.

Subsequently, the possible morphological and mineralogical changes produced after the addition of the different waste and by-products that may lead to the improvement in the geotechnical and mechanical properties were analysed.

Finally, an environmental assessment study was carried out by analysing the leachates produced by the waste and by-products applied and the stabilized soil mixtures used. In addition, the effects on the environment caused by the production of these materials and the execution of the stabilized soil mixtures were studied by means of a life cycle analysis.

6.3.1.- Effect of IBP and nanosilica on mechanical and geotechnical behaviour

The results obtained in phase 1 of the experimental program are shown in Table 6.5. Phase 1 aimed to analyse the effect of IBP both on the basic soil stabilization properties, such as plasticity, swelling and collapse, and on the engineering properties of the soil: compaction properties and bearing capacity determination.

Table 6. 5. Result of mechanical and geotechnical behaviour of stabilized soil mixtures

Mixture	Plasticity			Free swelling (%)	Collapse (%)	Maximum dry density (g/cm^3)	Optimum moisture content (%)	CBR 7d soaked (%)
	LL	PL	PI					
M0-CS	53.0	21.6	31.4	3.65	1.06	1.63	17.10	2.30
M1-1.5QL	48.5	30.9	17.6	0.08	0.31	1.58	19.30	17.50
M2-1.5QL-SSBA	43.1	33.9	9.6	0.06	0.39	1.58	19.00	20.90
M3-5BBA-1QL	45.0	30.4	14.6	0.29	0.54	1.59	19.90	15.47
M4-5BBA-0.5QL-SSBA	47.1	32.9	14.2	0.01	0.05	1.48	26.90	27.42
M5-5BFA-1QL	38.9	31.1	7.8	0.20	0.32	1.61	19.00	9.73
M6-5BFA-0.5QL-SSBA	43.1	35.9	7.2	0.04	0.14	1.50	23.40	29.33
M7-M5SS-1QL	41.6	32.9	8.7	0.29	0.19	1.63	17.70	24.26
M8-5SS-0.5QL-SSBA	43.7	42.7	1.0	0.04	0.09	1.62	18.10	29.66
M9-5pMRA-1QL	50.4	28.8	21.6	0.79	0.27	1.61	19.20	8.54
M10-5pMRA-0.5QL-SSBA	48.8	31.1	17.7	0.26	0.12	1.58	22.50	17.34

The effect of lime for stabilization of expansive and plastic clays has been extensively studied in the literature [50-52]. In the process of stabilization of clayey soils with lime, several stabilization mechanisms occur. The process is as follows:

After the initial mixing of the soil with the lime, calcium ions (Ca2+) from the lime are replaced by clay particles on the surface of the double tetrahedron layers, displacing water and other ions. In this process of cation exchange, the soil changes its macrotexture, improving its workability and compatibility.

With the change of macrotexture, a process of flocculation and agglomeration begins. In this phase, the plasticity index and the potential for swelling and collapse of the material decrease drastically and almost instantaneously.

Subsequently, a carbonation cementation takes place, in a time interval starting at 24-48 hours when the lime mixed with the soil hydrates. In this process, calcium oxide reacts with atmospheric carbon dioxide, precipitating calcium carbonate, improving the mechanical properties of the stabilized soils.

Finally, if the correct amounts of lime and water are added, a fast rise in soil pH above 10.5 occurs, allowing the clay particles to decompose and pozzolanic reactions to begin. Silica and alumina are released by reacting with the calcium in the lime to form calcium silicate hydrates (CSH) and calcium aluminium hydrates (CAH). CSH and CAH form the matrix that contributes to the strength of lime-stabilized soil layers. However, the alteration of the particle structure occurs slowly, depending on the type of clay present, starting at a usual maturation period of 1 to 4 days.

In the present work, the improvement of soil properties through the development pozzolanic reactions due to the interaction of IBP with the basic environment provided by lime were analysed.

Analysing the results shown in Table 6.5 for the determination of the Atterberg limits, it was observed that the untreated clayey soil presented a high plasticity, with a PI of 31.4%. For the development of this study, a value of 1.50% lime has been established in the control mix M1-1.5QL, in order to analyse the effect of the additions by reducing this percentage and adding IBP and nanomaterials. All the mixes were analysed at an age of 7 days in order to evaluate the possible effects of pozzolanic reactions that occur in the mixes made with IBP and SSBA.

The M1-1.5QL mixture had a PI of 17.6%, indicating a reduction of half over the untreated soil. This reduction was mainly due to an increase in the plastic limit and a slight reduction in the liquid limit.

As can be seen in Table 6.5, the PI of the rest of the mixtures where IBP were applied without interaction with nanomaterials, showed a reduction in PI. This reduction was mainly due both to the interaction of the percentage of lime added and to the physical modification of the soil by the addition of a percentage of 5% IBP, which did not present plasticity. Similar behaviour has been reported by different authors who have studied the stabilization of expansive soils and improvement of properties by the application of steel ash, biomass bottom ash and pulverised construction and demolition waste [35, 53-55].

Analysing mixtures in which IBP were combined with QL and SSBA, a further reduction in PI was observed, mainly due to an increase in PL. The addition of nanosilica had contradictory effects on the consistency limits of soils, showing in the literature its increase or reduction of plasticity depending on the percentage of nanomaterial added and binder or by-product applied in combination [56].

This reduction may be due to a stabilization mechanism by pozzolanic reactions between the calcium in the lime, the aluminosilicates present in the IBP, which decomposed in the basic environment due to the soil-lime interaction, and the highly reactive silica added in the SSBA, resulting in an overall increase of the plastic limit and a lowering of the PI [28].

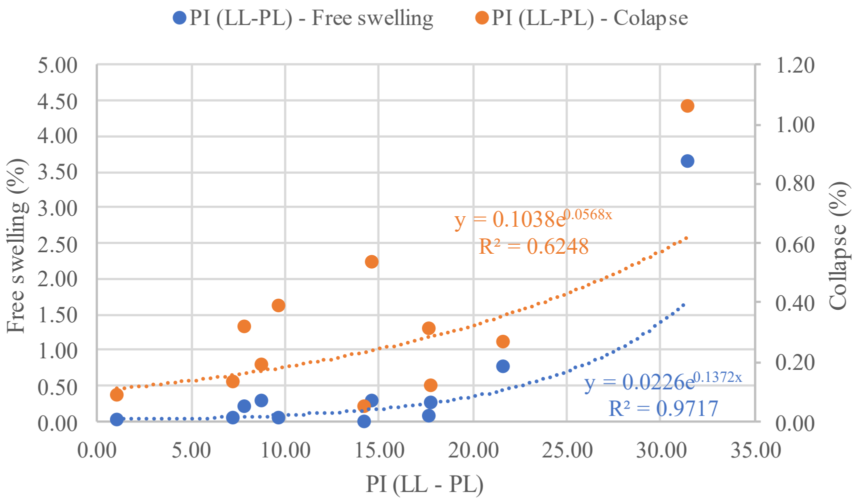


Fig. 6. 1. Correlation between Plastic index and swelling / collapse

As shown in Table 6.5, the untreated soil, M0-CS, had a high expansivity, with a free swelling value of 3.65%. However, the control mix M1-1.5QL had an expansion index of 0.08%, allowing almost total control over expansion.

For the rest of the samples, as shown in Figure 6.1, a consistent exponential relationship between the plasticity index and the free swelling were found, due to the mechanisms involved in the reduction of plasticity being the same as those involved in the reduction of expansivity.

However, between the collapse potential and the plasticity index a possible exponential relationship was observed, but it was not clear from the data analysed, due to the different factors involved in the development of collapse in soils, both at the micromechanical and mechanical level [57].

Analysing the data shown in Table 6.5 and represented in Figure 6.2, the results obtained in the mechanical behaviour tests of the soils are shown.

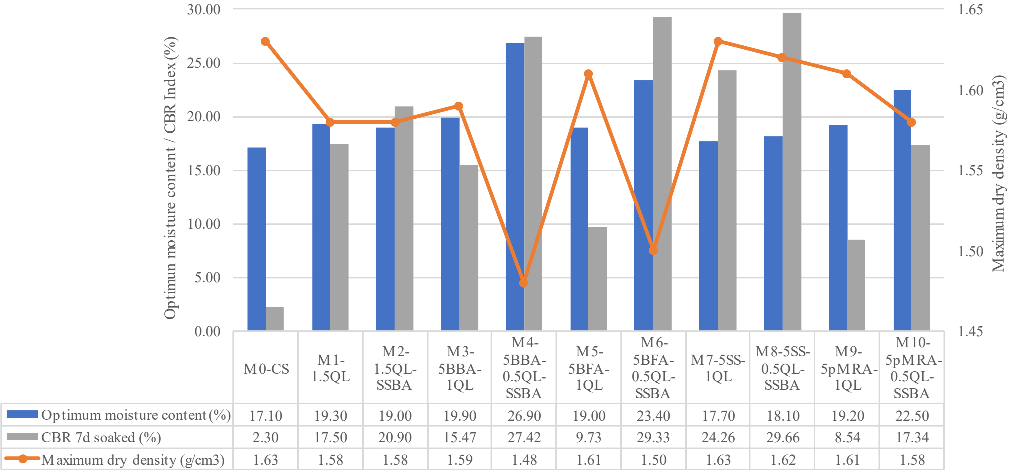
The untreated soil M0-CS had the lowest OMC with a value of 17.10% and the highest MDD,1.63 g/cm3. For these compaction parameter values, the untreated soil presented a bearing capacity measured in CBR index of 2.30%. This value indicated a practically null bearing capacity, classifying the soil as marginal or disposable, according to the Spanish standard. In addition, the Spanish standard requires a minimum value of 6% in CBR index for its application in embankment fillings, and a value of 12% for its application in road sub-bases [58].

The M1-1.5QL mixture showed a drop in MDD to a value of 1.58 g/cm3, and an increase in OMC to 19.30%. This behaviour is typical of expansive clayey soils stabilized with lime and has been described extensively in the literature [28, 29, 52, 59]. This change in compaction properties implied an increase in the CBR index to a value of 17.50%, giving the lime- stabilized soil sufficient geotechnical and mechanical properties for use in road layers.

Analysing the effect of the addition of silica-based nanomaterials in combination with the lime, M2-1.5QL-SSBA mixture, it was observed that it had a negligible effect on the OMC and MDD values. Regarding the bearing capacity, an increase of the CBR index up to a value of 20.9% was observed, an increase probably produced by the promotion of the reactions derived from the reactivity of the nanosilica. These results were consistent with different studies where nanosilica was applied in combination with cement [60].

Regarding the compaction properties of the mixtures combining IBP without nano, as shown in Figure 6.2 and Table 6.5, they presented in general an increase in the OMC and a slight drop in the MDD. This behaviour was due to the combined effect of the addition of 1% lime and the modification of the physical characteristics derived from the addition of 5% of each IBP. The same behaviour has been reported by different authors who have applied biomass ash, fly ash, activated steel slag and CDW to stabilize expansive soils [35, 54, 61, 62].

When analysing the results shown for the blends combining IBP with nanosilica, it was observed that the mixtures M4-5BBA-0.5QL-SSBA and M6-5BFA-0.5QL-SSBA showed a more pronounced drop in maximum density, to 1.48 and 1.50 g/cm^3, respectively, as well as a rise in OMC to 26.90 and 23.40%. The mixtures M8-5SS-0.5QL-SSBA and M10-5pMRA-0.5QL-SSBA, showed a slight drop in MDD, with values of 1.62 and 1.58 g/cm^3, respectively, and an increase in OMC to 18.10 and 22.50%.



	M0-CS	M1-1.5QL	M2-1.5QL-SSBA	M3-5BBA-1QL	M4-5BBA-0.5QL-SSBA	M5-5BFA-1QL	M6-5BFA-0.5QL-SSBA	M7-5SS-1QL	M8-5SS-0.5QL-SSBA	M9-5pMRA-1QL	M10-5pMRA-0.5QL-SSBA
Optimum moisture content (%)	17.10	19.30	19.00	19.90	26.90	19.00	23.40	17.70	18.10	19.20	22.50
CBR 7d soaked (%)	2.30	17.50	20.90	15.47	27.42	9.73	29.33	24.26	29.66	8.54	17.34
Maximum dry density (g/cm3)	1.63	1.58	1.58	1.59	1.48	1.61	1.50	1.63	1.62	1.61	1.58

Fig. 6. 2. Results of mechanical properties of stabilized clayey soil mixtures

This behaviour was partly explained, as in mixtures without addition of SSBA, by the modification of the physical properties of the soil, although the increase in OMC may be due to the development of pozzolanic properties, which generated a higher affinity to water [63].

Analysing the effect of the addition of IBP on the bearing capacity of the soils, it was observed that all the mixtures increased their CBR index to an acceptable value for their application in civil engineering works according to Spanish standards.

However, the addition of nanomaterials allowed to increase the bearing capacity of the mixes, thus extending their range of application to road sub-bases by reducing the lime content by up to 66% over the M1-1.5QL mix.

As can be seen in Figure 6.3, BFA showed a higher reactivity to the addition of nanomaterials, with a relative increase of 300%, followed by pMRA with a relative increase of 200% and BBA, with an increase of 175%. SS shows a relatively low increase after the addition of SSBA, with an increase from 24.26% to 29.66%, showing an improvement in the bearing capacity.

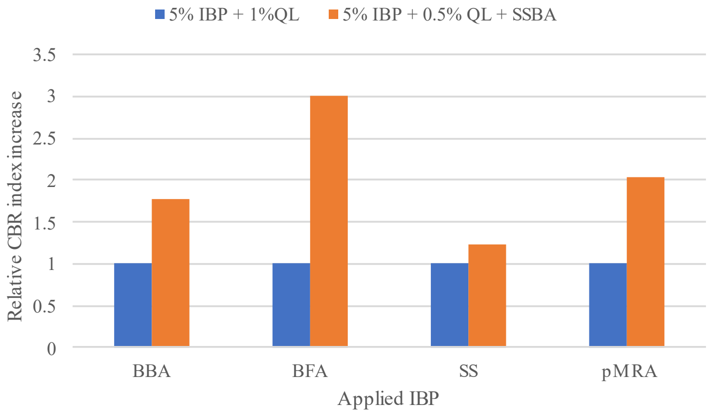


Fig. 6. 3. Relative CBR Index evolution in stabilized soil mixtures with and without nanosilica combined with IBP

6.3.2.- Morphological and mineralogical assessments

In order to evaluate the products of the hydration or pozzolanic reactions produced by the interaction of lime, IBP and nanomaterials, a study of the phases found was carried out by XRD and observation of the morphology by SEM microscopy of the mixtures made in the laboratory 28 days after their manufacture.

Figure 6.4 shows the diffractograms with the phases analysed for the 10 stabilized soil mixtures. In Figure 6.5, the selected images of the mixtures M1, M2, M3, M5, M7 and M9 are shown together with the result of the semi-quantitative analysis associated with each image.

The XRD results shown in Figure 6.4 show the phases composing the ten stabilized soil mixtures. Quartzite and calcite phases were in the majority in all the analysed mixtures. A Montmorillonite phase, a clay mineral belonging to the smectite group, which was responsible for the high plasticity and swelling of the soil.

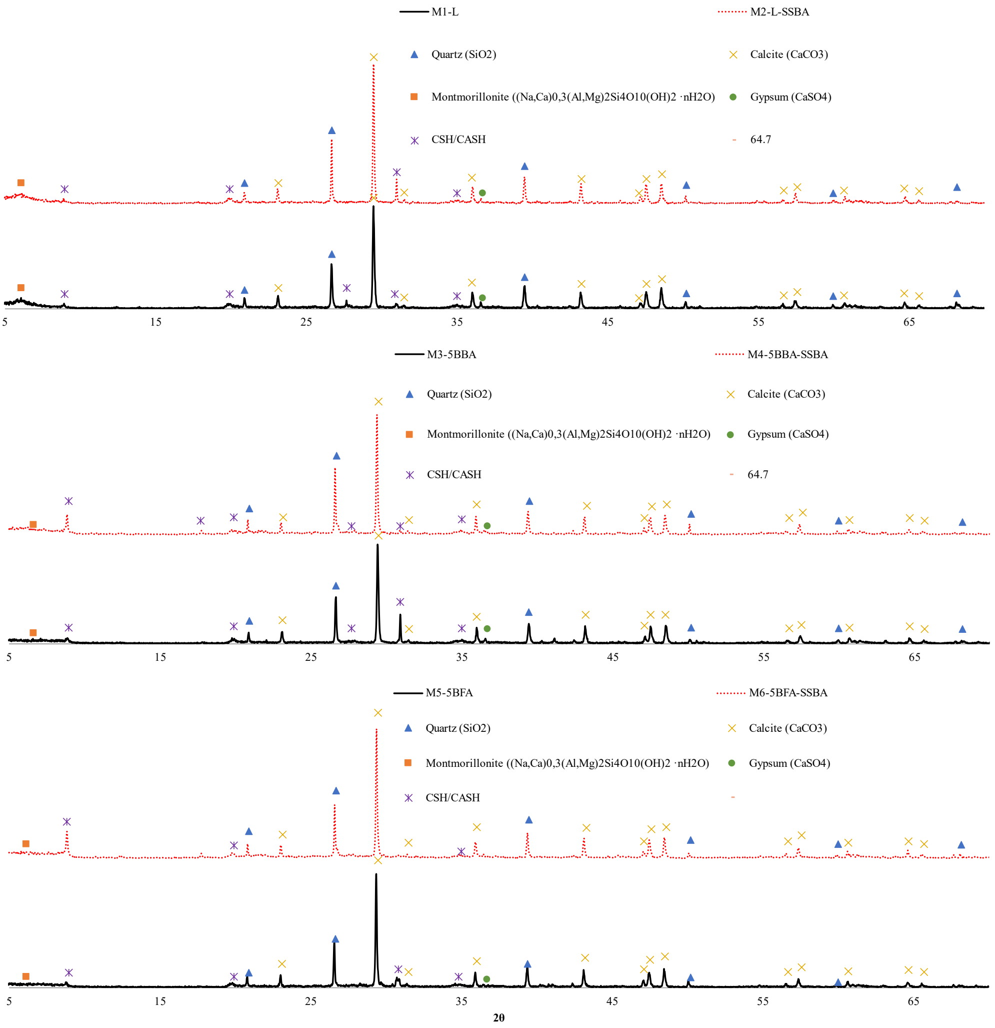


Fig. 6. 4. Results of DRX of stabilized soil mixtures (Part 1)

The compounds analysed after the stabilization process of the samples were: Calcium carbonate, present both naturally in the soil and because of the interaction process of added CaO and CO_2 from the air, hydrated calcium silicate and hydrated calcium aluminosilicate, coming from the development of pozzolanic reactions of lime with clay minerals and components of IBP.

The phases of CSH and CASH gels found in the 2θ angles coincided with those reported by other authors in the analysis of compounds from hydration processes of clayey soils with different wastes, industrial by-products or geopolymers obtained by alkaline activation processes [61, 64-66].

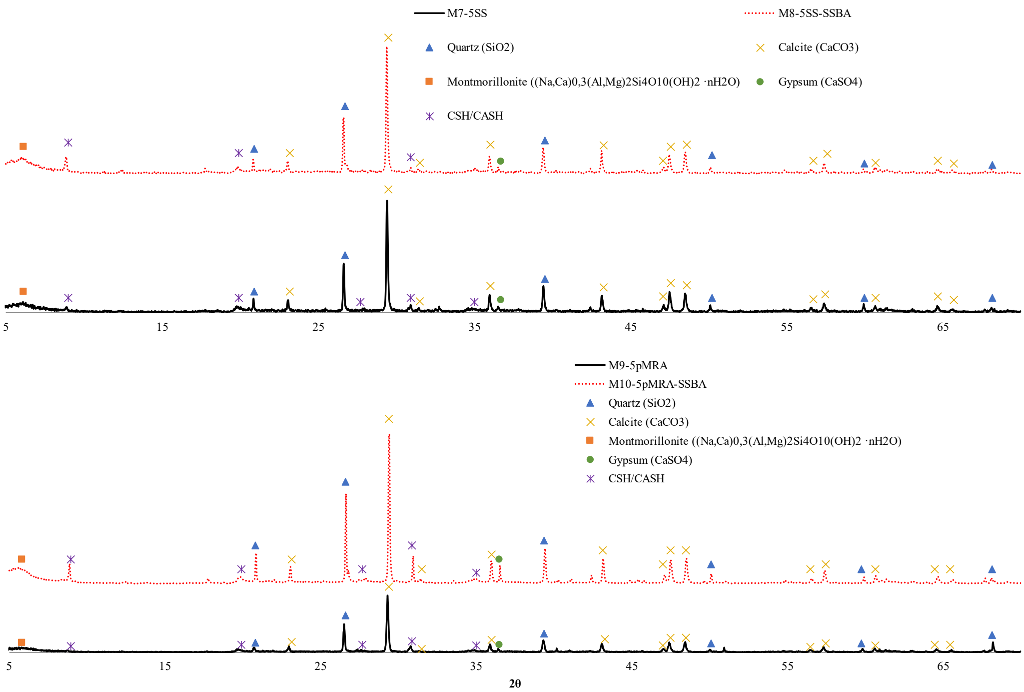


Fig. 6.4. Results of DRX of stabilized soil mixtures (Part 2)

Although all the mixtures presented peaks where CSH/CASH gels were found, analysing the mixtures where IBP were combined with nanomaterials and lime, a slight increase in the intensity of these could be observed; however, it was difficult to quantify the increase in the amount of these phases using this technique. On the contrary, since these elements presented a notably amorphous morphology, quantify the percentage of crystalline and amorphous phases in the sample analysed was possible, as shown in Table 6.6.

Table 6. 6. Crystallinity and amorphous percentage of stabilized soil mixtures

	M1-QL	**M2-QL-SSBA**	**M3-5BBA**	**M4-5BBA-SSBA**	**M5-5BFA**	**M6-5BFA-SSBA**	**M7-5SS**	**M8-5SS-SSBA**	**M9-5pMRA**	**M10-5pMRA**
% Amorphous	26.4	27.5	24.5	32.7	24.8	32.5	26.2	30.8	23.0	27.7
% Crystallinity	73.6	72.5	75.5	67.3	75.2	67.5	73.8	69.2	77.0	72.3

The crystallinity data of the ten stabilized soil samples analysed presented the same pattern of behaviour as those shown in the analysis of mechanical properties, measured in terms of CBR bearing capacity, indicating a higher increase in the percentage of amorphous phases found in those samples where IBP were combined with nanomaterials.

This analysis allowed, in a qualitative way, to correlate the interactions of nanomaterials and IBP in a basic environment offered by the lime reaction with improvements in soil bearing capacity and geotechnical properties, as well as to evaluate indirectly the high reactivity of nanomaterials, which has been widely exposed in different studies [67-70].

Finally, SEM microscopy had been used to study the morphology of the stabilized mixtures. As mentioned above, CSH/CASH gels were found in all ten samples analysed, however, it was difficult to quantify the increase of these compounds by optical techniques.

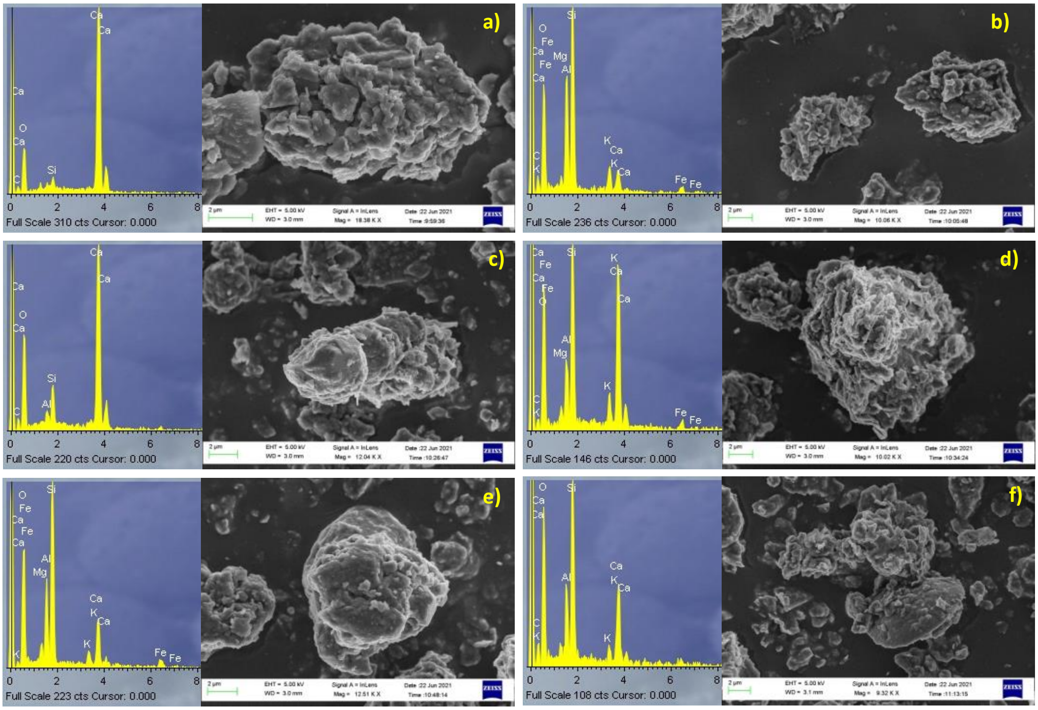


Fig. 6. 5. SEM microscopy and semi-quantitative analysis of the stabilized mixtures: a) M1; b) M2; c) M3; d) M5; e) M7; f) M9

For this reason, 6 images corresponding to the mixtures M1, M2, M3, M5, M7 and M9 were selected, and a semi-quantitative analysis was carried out at the indicated points to corroborate the results using the XRD technique.

As seen in the images, sheet-like clay minerals, calcite minerals and quartzite were found. In all the images, amorphous CSH/CASH type gels were found, which was consistent with the XRD results. Comparing figures, a) and b), quantify which of the mixtures presents a higher percentage of CSH/CASH gels was not possible, however, the presence of these compounds validates the results presented.

6.3.3-. Environmental assessments of leached

The leachate concentrations according to the compliance test are shown in Table 6.7. This test was performed on the raw materials to obtain their classification according to the limit values regulated by the Landfill Admission Directive 2003/33/EC and to identify the polluting potential of each raw material or IBP.

Unresolved numbers presented a concentration of heavy metals below the inert limit (inert material). Yellow numbers presented a concentration exceeding the limit for inert (non-hazardous material). Numbers in red exceed the limit for non-hazardous material (hazardous material). To be considered as inert a material must have a concentration within the inert range for all heavy metals analysed.

Figure 6.6 shows a graphical representation in terms of relative values with the concentrations of heavy metals of each analysed material expressed in relation to the limits imposed by the European directive.

As expected, clay soil and lime were classified as inert materials. This was due to the natural origin of clays, which can be applied as a waterproofing material in landfills [71] and the composition, mainly CaO, presented by the lime.

pMRA was classified as inert because, as shown in the compliance test results, the concentrations of all the heavy metals analysed were below the limit for inert material. Different studies have analysed recycled aggregates coming from the same plant [36, 39] showing similar results, indicating the suitability of these aggregates to be applied in road layers as granular material. However, Del Rey et al. [72] showed in their study the contaminant potential of recycled aggregates, concluding that in general, it should be treated as a non-hazardous material due to the Cr levels obtained. The disparity of results was due to the heterogeneity of recycled aggregates, whose composition in the same plant could vary depending on the construction and demolition waste that were processed for its production.

Table 6. 7. Results of leaching compliance test of raw materials (mg/kg)

	CS	*QL*	*BBA*	*BFA*	*SS*	*pMRA*
Cr	0.01	0.31	0.65	36.09	3.42	0.30
Ni	0.03	0.01	0.18	0.04	0.00	0.01
Cu	0.19	0.00	1.72	0.32	0.00	0.03
Zn	0.06	0.05	0.23	0.19	0.05	0.03
As	0.09	0.00	0.35	4.43	0.08	0.00
Se	0.02	0.04	0.22	2.58	0.78	0.02
Mo	0.07	0.47	0.72	2.76	12.67	0.09
Cd	0.00	0.00	0.00	0.01	0.01	0.00
Sb	0.04	0.00	0.04	0.48	0.00	0.02
Ba	0.12	2.11	0.09	0.46	18.34	0.33
Hg	0.00	0.00	0.00	0.00	0.00	0.00
Pb	0.00	0.30	0.01	1.10	0.06	0.00

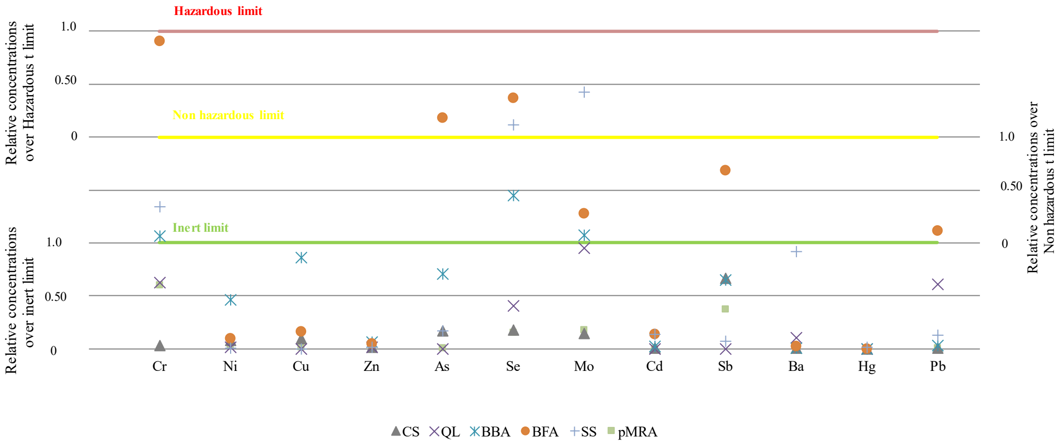


Fig. 6. 6. Graphical representation of relative values of leaching compliance test of raw materials

According to the results obtained, BBA were classified as non-hazardous material, due to exceeding the directive limit for the elements Cr, Se and Mo. BFA were classified as hazardous materials, due to exceeding the limits for the elements Cr, As, Se, Mo, Sb and Pb.

Both types of ash, BFA and BBA, came from the burning of the same biomass, which was the reason polluting metals were the same. However, BFA was a more refined product of combustion, collected in the gas expulsion filters together with the volatile elements, which was why the concentration of these elements was higher, and new heavy metals appear, exceeding the limits of the regulations.

In addition, numerous studies have investigated the polluting potential of BA from olive biomass, [39, 73-75] Cr, Se and Mo have been found to be problematic elements. The concentration of these heavy metals were associated with the high pH of the AB, which contributed to the dissolution of metals from basic metal salts, oxides and carbonates [76].

Steel Slag was classified as a hazardous material due to exceeding the directive limit for the elements Cr, Se and Mo. The presence of different leached elements in steel slag from electric arc furnace depends both on the manufacturing process and on the cooling and stabilization of the slag. Cr, Ba and V, were typically problematic elements in steel slags from electric arc furnaces studied by different authors [77, 78].

After analysing the results obtained in the compliance test, it was concluded that 3 of the 4 IBP were classified as non-hazardous or inert materials, which would prevent their application as raw materials. However, in the present study, these materials were applied in a percentage of 5% by weight on the stabilized soil mass, so it was necessary to analyse the polluting potential in combination with the soil and other stabilizing materials, such as lime and/or nanomaterials.

Table 6.8 shows the heavy metal concentration results obtained for the clay soil and for the 10 stabilized soil mixtures analysed. In addition, a graphical representation of the values obtained in relative terms is shown in Figure 6.7.

Table 6. 8. Results of leaching percolation test of stabilized soil mixtures (mg/L)

	M0-CS	**M1-1.5QL**	**M2-1.5QL-SSBA**	**M3-5BBA-1QL**	**M4-5BBA0.5QL-SSBA**	**M5-5BFA-1QL**	**M6-5BFA-0.5QLSSBA**	**M7-5SS-1QL**	**M8-5SS-0.5QL-SSBA**	**M9-5pMRA-1QL**	**M10-5pMR -0.5QL SSBA**
Cr	0.001	0.023	0.018	0.025	0.029	3.099	1.386	0.069	0.041	0.005	0.006
Ni	0.001	0.001	0.000	0.002	0.005	0.001	0.000	0.008	0.001	0.002	0.002
Cu	0.021	0.047	0.039	0.068	0.056	0.029	0.034	0.064	0.007	0.021	0.023
Zn	0.020	0.014	0.008	0.034	0.009	0.003	0.076	0.008	0.011	0.019	0.018
As	0.008	0.003	0.002	0.011	0.005	0.002	0.031	0.002	0.001	0.008	0.007
Se	0.002	0.001	0.003	0.002	0.003	0.110	0.069	0.007	0.007	0.003	0.003
Mo	0.014	0.059	0.054	0.073	0.056	0.260	0.179	0.097	0.127	0.028	0.031
Cd	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Sb	0.002	0.002	0.001	0.004	0.000	0.002	0.003	0.004	0.001	0.002	0.002
Ba	0.140	0.333	0.280	0.234	0.076	1.453	0.135	0.088	0.068	0.131	0.149
Hg	0.000	0.000	0.000	0.000	0.000	0.001	0.001	0.000	0.000	0.000	0.000
Pb	0.003	0.004	0.001	0.006	0.001	0.000	0.001	0.000	0.000	0.004	0.004

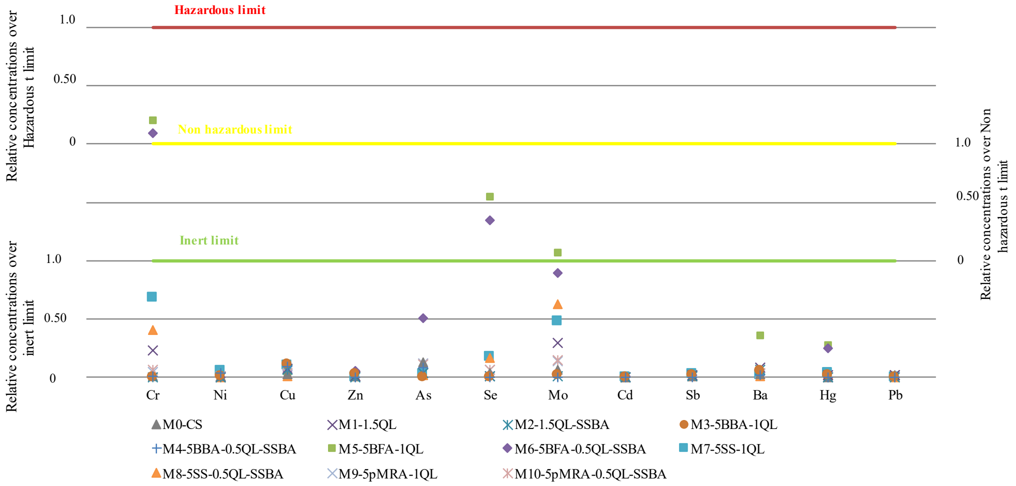


Fig. 6. 7. Graphical representation of relative values of leaching percolation test of stabilized soil mixtures

Analysing the data shown in Table 6.8 and represented in Figure 6.7, a clear reduction was observed in all heavy metals for which the limit was exceeded in the compliance test. The mixtures where BBA, SS and MRA were applied presented concentration values of heavy metals below the inert limit, and their application did not represent a risk for the environment. However, the mixtures M5 and M6, where BFA was applied, although a large drop in the relative concentration above the limits was observed, comparing figure 6 and Figure 6.7, very high values were obtained for the elements Cr, Se and Mo.

This reduction was due to two distinct mechanisms: reduction due to physical mixing of IBP with soil and immobilisation of heavy metals due to the formation of CSH gels, which captured heavy metals by adsorption or co-precipitation [38, 65].

6.3.4.- Life cycle assessment of stabilized clay soil

The characterisation values associated with the production of 1 tonne of materials are listed in Table 6.9. The results showed that the production of SSBA and QL generated the highest environmental impacts in all categories.

Specifically, the manufacture of 1 tonne of SSBA required 15.18 g of Sb eq., 4603.9 MJ, 2696.7 g of SO2 eq. and 566.9 g of PO_4 eq., impacts associated with ADe, ADf, A and E categories. For these categories, the loads occurred as a consequence of the consumption of mineral resources (tellurium, gold, copper) and fossils (gas/natural, coal/hard, oil/crude) as well as air emissions (sulfur dioxide, nitrogen oxides, ammonia) and water discharges (phosphate, chemical oxygen demand).

Likewise, during the manufacture of 1 tonne of QL, 861.1 kg of CO_2 eq., 0.04 g of CFC-11 eq. and 130 g of C2H4 eq. were emitted, associated substances with the categories GW, ODP and POF. These values were originated due to air emissions of carbon dioxide/fossil, methane/fossil, methane and carbon monoxide. These emissions derived from the manufacturing process off the kiln, as a consequence of the particular chemical composition of the raw materials and fuels used [79]

For SSBA production, the second highest impact values were reached in categories ADe and E, as a consequence of the landfill disposal of the non-recovered part, which spills consumption of mineral resources (tellurium) and Phosphate discharges into water.

Regarding the loads associated with the production of IBP (BBA, BFA, pMRA and SS), the values obtained were lower, due to the fact that the recovery of these materials requires minimal processing through loading and storage machinery.

Finally, the extraction of CS generated very low impact values since the material could be obtained in the trace of the work with minimal processing.

Table 6. 9. Results obtained in the LCA by impact categories for material (per 1t)

Material	*Impact category*						
	ADe	***ADf***	***GW***	***ODP***	***POF***	***A***	***E***
	g Sb eq.	MJ	kg CO_2 eq.	g CFC-11 eq.	g C_2H_4 eq.	g SO_2 eq.	g PO_4 eq.
CS	0.000016	0.42	0.03	0.000005	0.01	0.10	0.02
QL	0.111120	3253.17	861.15	0.042414	130.04	757.62	91.45
BBA	0.000085	2.25	0.16	0.000028	0.04	0.53	0.12
BFA	0.099882	18.70	1.72	0.000090	0.71	11.96	3.89
SS	0.006205	21.10	1.72	0.000182	0.45	7.24	3.15
pMRA	0.018622	56.95	4.80	0.000358	1.22	33.43	7.62
SSBA	15.182838	4603.91	451.34	0.022252	117.42	2696.70	566.99

The characterisation results of the soil mixtures analysed are listed in Table 6.10. The untreated soil (M0) showed the lowest impact values in all categories since only CS and water are included in its dosage. Regarding the soil mixtures treated with QL, the lowest impact values were found in the M1 mixture for ADe and E categories, and in the M4 mixture for the rest of the categories. The highest values were observed in M2 mixture for ADf, GW, ODP and POF categories, M6 mixture for ADe, and M9 mixture for A and E categories.

Table 6. 10. Results obtained in the LCA by impact categories for stabilized soil mixtures

Mixture	*Impact category (per fuctional unit: experimental section)*						
	ADe	***ADf***	***GW***	***ODP***	***POF***	***A***	***E***
	g Sb eq.	MJ	kg CO_2 eq.	g CFC-11 eq.	g C_2H_4 eq.	g SO_2 eq.	g PO_4 eq.
M0	0.15	1040.96	79.85	0.01123	18.83	290.04	99.61
M1	1.34	21339.55	5099.73	0.27432	775.02	5057.65	729.70
M2	4.58	22377.22	5199.49	0.27982	800.40	5644.26	852.78
M3	2.82	20673.40	3854.46	0.26134	580.84	5013.46	879.60
M4	5.34	14030.56	2126.64	0.16745	331.76	3783.21	764.58
M5	4.78	21232.76	3931.48	0.26567	600.74	5291.20	960.76
M6	7.19	14459.52	2178.56	0.17044	347.00	4018.69	831.34
M7	3.00	21512.61	3977.73	0.27054	602.51	5253.37	952.55
M8	5.91	15531.53	2342.08	0.18476	366.98	4203.26	862.39
M9	3.21	21971.82	3990.97	0.27085	610.74	5705.94	1032.83
M10	6.02	15906.00	2349.21	0.18410	374.38	4624.72	941.22

Specifically, the M1 mixture stabilized with 1.5% QL presented a consumption of 1.34 g of Sb eq. in the ADe category and emitted 729.7 g of PO4 eq. in E category. Likewise, the soil mixture treated with BBA, QL and SSBA (M4) required a consumption of 14030.5 MJ for ADf, and emitted 2126.6 kg of CO2 eq., 0.167 g of CFC-11 eq., 331.7 g of C2H4 eq. and 3783.2 g of SO2 eq., corresponding to the GW, ODP, POF and A categories respectively.

Likewise, the mixture of soil treated with 1.5% QL and SSBA (M2) generated the highest loads in several categories highlighting a consumption of 22377.2 MJ for ADf, and the air emissions of 5199.4 kg of CO2 eq. for GW, 0.279 g of CFC-11 eq. for ODP, and 800.4g of C2H4 eq. for POF. The incorporation of SSBA in the mixture treated with BFA and QL (M6) caused the highest impact in the ADe category with a value of 7.19 g of Sb eq. Finally, the mixture treated with pMRA and 1% QL (M9), generated up to 5705.9 g of SO2 eq. and 1032.8 of PO4 eq. in categories A and E respectively.

The soil mixtures are represented comparatively in Figure 6.8. As can be seen, in general, the SSBA-treated mixes showed the highest impacts in the ADe category. For the rest of the categories, the highest impacts were reached in the mixtures treated with QL at 1.5% and 1%, although SSBA also had an influence in category E. By contrast, the mixtures treated with 1.5% and 1% lime showed less impact in the ADe category, with reductions that ranged between 16% and 81%. In the ADf, GW, ODP, POF and A categories, the impacts were reduced between 2% and 59% in the mixtures treated with IBP, SSBA and QL at 0.5%. And for the E category, the loads were reduced between 7% and 29% for the mixtures treated with IBP and 0.5% QL, and the mixture treated with 1.5% QL.

Figure 6.9 shows the contribution to the impact in each category of the processes that constitute the stabilized soil mixtures (materials, transportation and execution). The contribution for each of them is detailed below.

In the M0-CS mixture, the execution of the untreated soil generated the highest impacts in the ADf, GW, ODP, POF and A categories, with contributions ranging from 49% in category A to 67% in ODP. For categories ADe and E, the highest impacts were generated by the use of water, varying between 58% and 75%. CS's contribution ranged from 4% in ADe to 18% in ODP, while transportation of materials caused from 1% in POF to 4.5% in ADe.

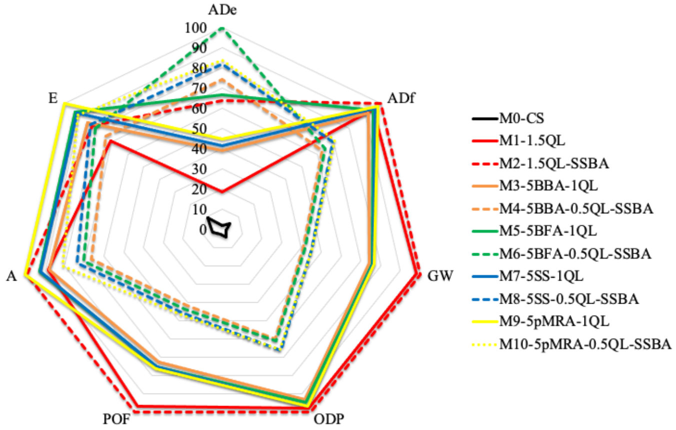


Fig. 6. 8. Comparative chart of characterization results of stabilized soil mixtures.

For the M1-1.5QL mixture, the manufacturing of the QL was responsible for the highest impacts across all categories, ranging from 47% (ADe) to 96% (GW). Next, the transport of materials generated the second highest impact in all categories, between 2% (POF) and 42% (ADe). The contribution to the impact from the use of water was up to 9%, execution works caused up to 4% (E) and the use of CS generated the least impact with contributions of less than 1% in all categories.

In the M2-1.5QL-SSBA mixture, the contribution to the impact of the processes was similar to the M1 mixture, although slight differences were observed due to the incorporation of SSBA, which generated the highest contribution in the ADe category (70%), and between 1% (ODP) and 14% (E) in the rest of the categories.

In the M3-5BBA-1QL mixture, the QL presented the highest contribution in five of the seven impact categories, ranging from 57% (A) to 85% (POF and GW). For the ADE and E categories, the highest contribution corresponded to the transport processes, being 80% and 48%, respectively. The water contributed with impacts of less than 7%, to the execution of the mixture generating loads of less than 4%, and 1% for CS. It is noteworthy that the incorporation of BBA in the mixture contributed with impacts of less than 0.3% in all categories.

In the M4-5BBA-0.5QL-SSBA mixture, the QL contribution was the highest in the GW (72%) and POF (70%) categories. For the ADe category, the highest contribution was 57%, which corresponded to SSBA, and for the rest of the categories (ADF, ODP, A AND E) the charges were associated with transport processes, around 45%. The water contributed with impacts of less than 11% and the execution of the mixture generated loads of less than 5%. Regarding the contribution of CS and BBA, it constituted percentages lower than 1% and 0.3%, respectively.

In the M5-5BFA-1QL mixture, the QL presented the highest contribution in five of the seven categories with values ranging from 55% (A) to 84% (GW). For the ADe and E categories, the highest contribution corresponded to transportation processes, up to 47%. Water generated impacts of less than 7%, the mix execution processes contributed with values less than 3%, and for CS they were less than 1%.

In the M6-5BFA-0.5QL-SSBA mixture, the contribution of the QL was the highest in the GW (71%) and POF (67%) categories. For the ADe category, the highest contribution was 42%, which corresponded to SSBA, and for the rest of the categories (ADf, ODP, A y E) between 39% and 45% that were associated with transport processes. BFA contributed up to 25% in ADe, water contributed less than 8% and the execution of the mixture generated loads less than 4%. Regarding the contribution of CS, it constituted percentages of less than 1%.

In the M7-5SS-1QL mixture, the highest contributions corresponded to QL in the ADf, GW, ODP, POF y A categories, with values ranging between 56% (A) and 84% (POF). For the rest of the categories (ADe and E), the highest contributions were generated by transport processes, that oscillated between 45% (ADe) and 76% (E). SS caused contributions of up to 6.5%, for water it was less than 6.4%, and for execution processes and CS were 3% and 1% respectively.

In the M8-5SS-0.5QL-SSBA mixture, transport processes generated the highest contribution in ADf, ODP, A and E categories, from 40% in A to 46% in E. QL contributed up to 68% in POF and 71% in GW, and SSBA was responsible for contributions of up to 56% in ADe. Water and SS caused an impact lower than 7% in all categories, and the execution processes and CS contributed with loads lower than 4% and 1% respectively.

In the M9-5pMRA-1QL mixture, QL was responsible for the highest contributions in five of the seven categories, with values ranging from 51% in A to 83% in GW. Transportation processes generated the highest impacts in the ADe and E categories, 71% and 41% respectively. pMRA incorporation was responsible for up to 14%, water contributed loadings less than 6%, execution of the mixture 3%, and CS 0.8%.

In the M10-5pMRA-0.5QL-SSBA mixture, the incorporation of SSBA generated the highest contribution in the ADe category, with 53%. By decreasing the amount of QL in the mix dosage with respect to the M9 mix, its contribution was the highest in the GW (69%) and POF (65%) categories. Likewise, transportation processes generated the greatest impacts in the rest of the categories, with contributions between 36% (A) and 43% (ODP). The contributions of the rest of the processes were less than 15% for pMRA, 7% for water, 4% for the execution of the mixture and 1% for CS.

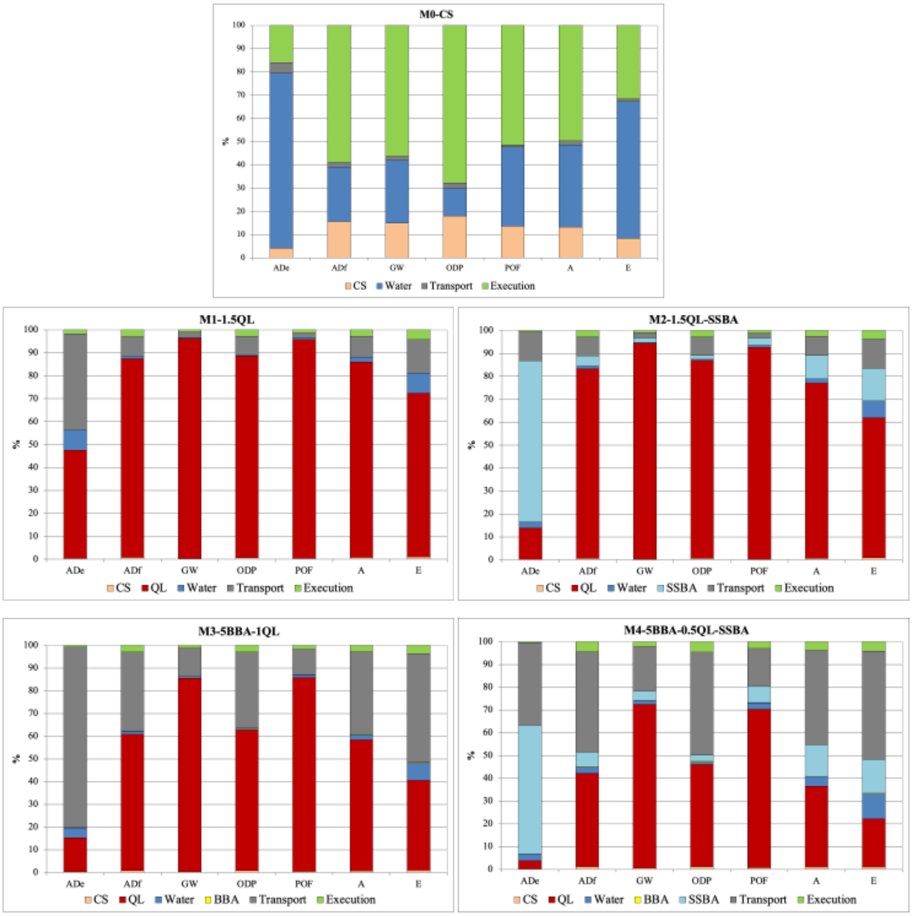


Fig. 6. 9. Contribution of processes to the impact of soil mixtures. (Part 1)

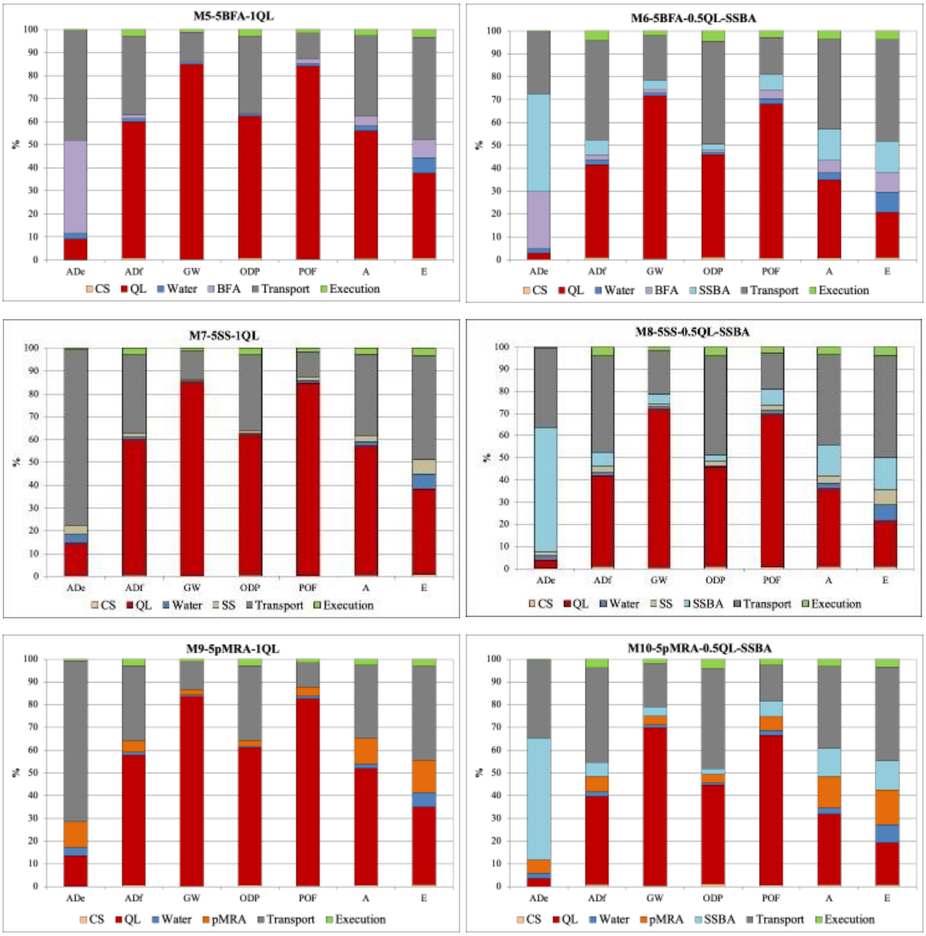


Fig. 6. 9. Contribution of processes to the impact of soil mixtures. (Part 2)

6.4.- Conclusions

The present study analysed the effects of applying different industrial by-products (IBP) in combination with lime and/or nanomaterials to stabilize an expansive and plastic clayey soil. The medium-term mechanical and geotechnical properties of ten stabilized soil samples were evaluated, along with the mineralogical variations resulting from the application of IBP. Additionally, an environmental analysis of the leachates produced by the different IBP and the stabilized soil mixtures was conducted. Finally, a life cycle analysis (LCA) was performed to assess the potential environmental improvements resulting from the application of IBP.

The following conclusions can be drawn from this study:

In terms of the geotechnical properties (plasticity, free swelling, and collapse) of the stabilized mixtures with IBP analyzed, all the mixtures exhibited improvements. This can be attributed to the interaction between lime and the soil, as well as the addition of IBP (at a 5% range), which modified the physical properties.

The interaction of nanomaterials with IBP led to an increase in the plastic limit of the soil, resulting in a significant reduction in swelling and collapse in the stabilized soils.

Analyzing the bearing capacity of the developed stabilized soil mixtures, it was observed that a combination of 5% IBP and 1% lime allowed for sufficient bearing capacity in all cases to be applied in road layers. This is due to the effect of lime and the improvement of the soil's physical properties resulting from the addition of IBP.

By combining 5% IBP, 0.5% lime, and SSBA, a substantial improvement in loading capacity was observed in the blends. The combination of BFA, lime, and SSBA exhibited a 300% increase, while BBA and pMRA, lime, and SSBA showed around a 200% increase. The blend with SS, lime, and SSBA demonstrated a 122% improvement.

The results obtained in the mineralogical study confirmed the formation of CSH/CASH gels in all mixtures where IBP were combined with nanomaterials. There was a correlation between the reduction in crystallinity of the mixtures when IBP were combined with SSBA and the improvement in mechanical and geotechnical properties, indicating the formation of gels derived from pozzolanic reactions.

The environmental impact study of leachates demonstrated the polluting potential of BBA, BFA, and SS. pMRA was classified as inert, but its potential as a non-hazardous material was indicated by scientific experience and production variability.

The application of BBA, SS, and pMRA did not pose a risk to the environment due to the application rate and the immobilization of heavy metals in the stabilized mixtures. However, mixtures containing BFA exhibited high levels of hazardous properties, discouraging its use in the analysed percentage.

The production of SSBA and QL resulted in the highest environmental loads due to mineral and fossil resource consumption, air emissions, and water discharges. Conversely, the production of IBP (BBA, BFA, SS, and pMRA) required minimal processing by machinery, resulting in low environmental impacts.

Regarding the stabilized soil mixtures, the incorporation of IBP reduced the environmental loads in almost all categories compared to the mixture stabilized with QL at 1.5%. The combined use of IBP and SSBA further reduced the impact values by decreasing the proportion of lime in the mixture dosage (from 1% to 0.5%). However, the incorporation of nanomaterials led to higher impact values in the ADe category due to the consumption of mineral resources necessary for their manufacture.

In conclusion, after analysing four types of industrial by-products, evidence was found for the formation of CSH/CASH gels resulting from the interaction between waste and nanomaterials under a basic environment facilitated by lime. This interaction improved the mechanical and geotechnical properties of the soils. Additionally, the leachate impacts were reduced, and CO2 emissions were mitigated through lime reduction.

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CAPÍTULO VII

FEASIBILITY OF USING NANOSILANES IN A NEW HYBRID STABILISED SOIL SOLUTION IN RURAL AND LOW-VOLUME ROADS

Díaz-López, J.L., Cabrera, M., Marcobal, J.R., Agrela, F., Rosales, J.

applied sciences

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Article

Feasibility of Using Nanosilanes in a New Hybrid Stabilised Soil Solution in Rural and Low-Volume Roads

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Abstract: The application of new materials for soil stabilisation is a growing field of study in recent years. In this work, the effect of two types of silica-based nanomaterials combined with binders (quicklime and cement) are studied to stabilise soils and form structural layers for rural and low volume roads. The physical and chemical properties of the materials have been determined, as well as the mechanical behaviour of the stabilised soil. Three hybrid stabilised soil sections have been designed using a multilayer elastic model, executed at full scale and measuring the evolution of their properties in the medium to short term. The results show that the application of silica-based nanomaterials and two types of binders on the tread layers provide high structural stability and good behaviour of the sections.

Keywords: hybrid stabilisation; mechanical behaviour; real scale application; nanomaterials

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1. Introduction

Over several decades, a large primary and secondary network of pavement roads and highways has been introduced by all developed countries to connect urban centres, metropolis and industrial areas with each other. This connectivity, combined with marine and air modes of transport, has generated a great socioeconomic impact derived from the transport of goods and passengers [1].

However, there are many rural areas, small cities and, especially, developing countries where it is not always possible to build this great network of roads due to economic, accessibility or constructability issues, as well as the fact that other modes of transport have a lower level of development and, therefore, cannot even be implemented. In this way, rural and low volume roads are a major opportunity to improve the connectivity and socioeconomic development of these areas [2].

Rural roads have different functions according to the level of development of the country where they are built. In developed countries, rural roads are usually designed to connect towns with low populations and agricultural and livestock areas with a low volume of vehicles. On the contrary, in developed countries, rural roads are designed to meet the socioeconomic needs of the rural population, connecting remote areas to basic health services, education and markets [3].

These types of road can be composed of a subgrade and a thin asphaltic layer or, most commonly, by compaction of an unpaved unbound granular material [4] or even compaction of stabilised soils that are found in the location of the road [5].

However, in most cases, these soils, especially clayey soils, present geotechnical problems, such as a lack of bearing capacity, high plasticity or swelling potential, that prevent their use. In order to improve the properties of soils and increase their range of

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VII

FEASIBILITY OF USING NANOSILANES IN A NEW HYBRID STABILISED SOIL SOLUTION IN RURAL AND LOW-VOLUME ROADS

Díaz-López, J. L., Cabrera, M., Marcobal, J. R., Agrela, F., & Rosales, J. (2021). Feasibility of using nanosilanes in a new hybrid stabilised soil solution in rural and low-volume roads. Applied sciences, 11(21), 9780.

Abstract

The application of new materials for soil stabilisation is a growing field of study in recent years. In this work, the effect of two types of silica-based nanomaterials combined with binders (quicklime and cement) are studied to stabilise soils and form structural layers for rural and low volume roads. The physical and chemical properties of the materials have been determined, as well as the mechanical behaviour of the stabilised soil.

Three hybrid stabilised soil sections have been designed using a multilayer elastic model, executed at full scale and measuring the evolution of their properties in the medium to short term. The results show that the application of silica-based nanomaterials and two types of binder on the tread layers provide high structural stability and good behaviour of the sections.

Keywords

hybrid stabilisation; mechanical behaviour; real scale application; nanomaterials

7.1.- Introduction

Over several decades, a large primary and secondary network of pavement roads and highways have been introduced by all developed countries to connect urban centres, metropolis and industrial areas with each other. This connectivity, combined with marine and air modes of transport, has generated a great socio-economic impact derived from the transport of goods and passengers [1].

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These types of road can be composed of a sub-grade and a thin asphaltic layer or most commonly by compaction of an unpaved unbound granular material [4] or even compaction of stabilised soils which are found in the location of the road [5]. However, in most cases these soils, especially clayey soils, present geotechnical problems such as lack of bearing capacity, high plasticity or swelling potential that prevent their use. In order to improve the properties of soils and increase their range of use in civil engineering applications, soil stabilisation for application in road layers has become widespread in recent years [6,7].

Soil stabilisation is defined as the improvement of the shear strength, durability stiffness and reduction of the plasticity and swelling potential of soils achieved by mechanical means or the addition of stabilising products, such as hydraulic binder, fly and rice husk ash, chemical stabilisation, recycled waste and by-products, etc, [8].

Among all the stabilising products, stabilisation with binders (commonly lime and cement) have been widely used by numerous authors in recent decades [9-12]. Nevertheless, the production of these traditional materials generates a negative environmental impact due to the use of raw material resources and the high CO_2 emissions involved in their production.[13]

For the reason mentioned above, in recent years several studies that analyse the possible stabilisation of soils with industrial by-products or recycled materials, such as biomass fly and bottom ash, [14-17] phosphogypsum, [18,19], steel slag [20,21] and magnesia oxides [22,23], among many others, have been increased.

In addition, in recent years an alternative to conventional stabilising products and wastes and by-products has emerged: nanomaterials.

Nanomaterials are particles with a typical size of between 1-100 nm, with a very high specific surface area, which implies very high reactivity [24], achieving soil improvements with very low dosages. Nanomaterials commonly used in soil stabilisation are composed of simple oxides, such as SiO_2, TiO_2 or $CaCO_3$ or carbon nanotubes [25,26].

Although a large number of nanoparticles has been developed, the pozzolanic capacity of Nano-SiO_2 and its reactivity with lime and cement to form calcium silicate hydrate (CSH) compounds have led most studies to focus on it [27-31].

Kulanthaivel et al. [27] studied the effect of using synthesised Nano-SiO2 by a sol–gel process together with cement to stabilise clay soil, concluding that a 7% addition of nano-SiO2 improves the unconfined compressive strength in a ratio of 5.24 and reduces permeability in the range of 0.01976 cm/sec to0.01198 cm/sec of the soil which is consistent with previous studies [28].

Ghasabkolaei et al. [30] and Bahmani et al. [31] studied soil stabilisation with cements and nanosilane with dosages lower than 1% by weight of Nano-SiO2, observing an increase in unconfined compressive strength in all mixtures with nanoparticulate additives and a high formation rate of calcium silicate hydrate (CSH), which implies an improvement in soil properties.

The present study shows new structural solutions for rural and low volume roads based on soil stabilised with organosilanes and small amounts of lime and cement. The design parameters of the new sections which were obtained through laboratory tests were tested in a real scale application to verify the feasibility use of the developed solutions. In addition, a medium-short-term performance study of road sections built was carried out to check their durability.

7.2.- Research purpose and experimental programme

This work is a continuation of the one carried out by Rosales et al. [32] in which a conventional control section was made according to Spanish specifications [33] and two alternative experimental sections based on nanomaterials and quicklime were performed in order to reduce the total thickness of the treated layers.

In the present work, three new trial sections are proposed with a hybrid stabilisation process in which nanomaterials and small amounts of quicklime and cement are used to reduce their treated thickness with respect to the control section by 40% and improve the mechanical properties and durability.

To carry out the trial sections in a successful manner, the following phases were followed in the present work:

Phase 1. Conceptualisation and pre-design of alternative sections based on the study conducted by Rosales et al. [32]

Phase 2. Physicochemical characterisation of the materials involved in the work, namely expansive clayey soil, sandy soil which is a rejection of the production of crushed gravel, lime, cement, and nanomaterials.

Phase 3. Laboratory study to obtain the design parameters of the sections. In this phase, the percentages of soil for the mixtures were defined, as well as the quantities of binders and organosilanes. The California Bearing Ratio (CBR) index and unconfined compressive strength (UCS) were obtained.

Phase 4. Trial sections structural design using Everstrees 5.0 software. From the CBR index and simple compressive strength, the elastic modulus of the layers composing the sections were determined. Once the elastic parameters of the materials had been defined, the different section options were evaluated by means of a multilayer elastic analysis in the software.

Phase 5. Construction and survey of the experimental sections. In this phase, the road sections were built, and short- and long-term checks of the bearing capacity of the soil were performed by non-destructive tests such as load plate and falling weight deflectometer (FWD).

7.3.- Phase 1. Section pre-design

According to the results obtained by Rosales et al. [32], three alternative sections composed of two layers were proposed, one of lime stabilised expansive clayed soil (CS) and another upper one of a mixture of CS and sandy soil (SS) stabilised with cement.

In the first pre-design stage, layer thicknesses and soil proportions were not defined; however, the sections and their construction process were defined in a qualitative manner, as shown in Figure 7.1.

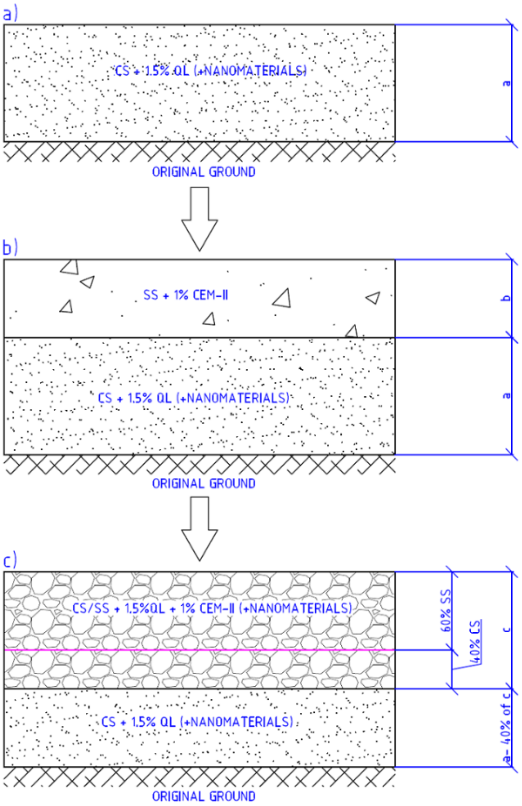


Fig. 7. 1. Manufacturing process scheme of the hybrid stabilised sections. a) Step 1: Pre-compaction of CS; b) Step 2: Spreading and mixing of SS and cement; c) Step 3: Compaction of CS+SS

As shown in Figure 7.1 the process of construction of the hybrid stabilised sections was carried out in three steps:

Step 1: Spreading, mixing and pre-compaction of the quicklime (or quicklime and nanomaterial) stabilised clayey soil layer on the original ground. The nanomaterials were added, according to the dosage shown in the following sections, into the mixing water. The curing time of the layer was one day.

Step 2: After one day, spreading and mixing of the sandy soil layer on top of the compacted quicklime (or quicklime and nanomaterial) stabilised clayey soil layer and spreading of the cement.

Step 3. Compaction of the layer composed of a percentage of compacted quicklime (or quicklime and nanomaterial) stabilised clayey soil plus sandy soil and cement. The result is called hybrid stabilised soil.

The following sections analyse the physicochemical properties of the materials involved as well as the mechanical properties of the mixes to be defined to optimise both the thickness of the mixes and the percentages of binders.

7.4.- Laboratory tests

7.4.1.- Phase 2: Materials and physicochemical characterisation

In this section, the materials used during the research for the development of the tests and construction of trial sections are shown.

7.4.1.1.- Soils: Expansive clayey soil and sandy soil

In this work, two types of soils have been analysed for the subsequent stabilisation and construction of the layers of the trial section. An expansive clayey soil (CS) and a quarry reject from the production of crushed gravel called sandy soil (SS).

The clayey soil (CS) comes from the plot where the real-scale sections are built, in Villacarrillo (Jaén), Andalusia.

The sandy soil comes as rejects from a crushed gravel production quarry located near Villacarrillo. The rejects are subjected to a sieving process to achieve a particle size within the parameters of Spanish regulations [34]. Table 7.1 shows the physicochemical properties of the two soils analysed and Figure 7.2 shows the granulometric curves of the soils.

Table 7. 1. Physical and chemical properties of CS and SS

Properties	CS	SS	Standard Test
Atterberg limits			UNE-EN ISO 17892-12:2019
Liquid Limit (%)	53.0	16.1	
Plastic limit (%)	21.6	11.9	
Plasticity index (%)	31.4	4.2	
Grain size distribution			UNE-EN ISO 17892-4:2019
Gravel (> 4 mm) (%)	0.0	77.1	
Sand (0.063-4mm) (%)	5.4	17.4	
Silt and clay (<0.063 mm) (%)	94.6	5.5	
Maximun dry density (kg/m^3)	1.55	2.28	UNE 103501:1994
Organic matter content (%)	0.31	0.02	UNE 103204:2019
Water-soluble sulphate (% SO_3)	0.05	0.07	UNE-EN 1744-1:2010+A1:2013
Main components XRF (%)			
P	0.08	0.03	
Si	25.57	3.32	
Ca	14.16	31.2	
Al	2.69	0.83	
S	0.03	0.05	
K	1.66	1.74	
Mg	1.37	16.2	
Fe	2.42	1.06	

In contrast, the sandy soil has a continuous grain size, composed mainly of particles larger than 4 mm with a maximum aggregate size of 32 mm and a percentage of fines of less than 6%. The plasticity index of SS is 4.2, which indicates that it is not a very plastic material.

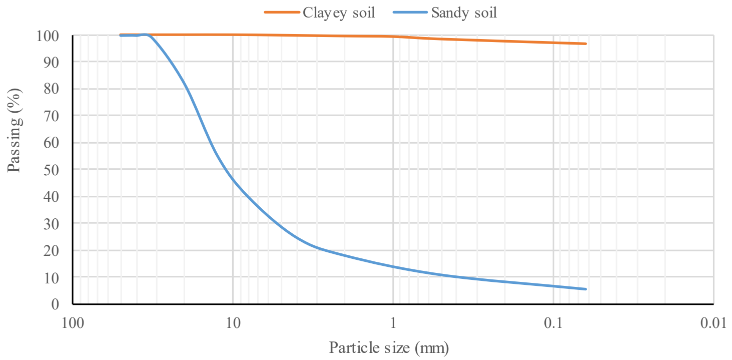


Fig. 7. 2. Particle size distribution of clayey soil and sandy soil

Both materials have low percentages of organic matter content and water-soluble sulphates, which make them suitable for use in road layers in accordance with Spanish regulations.

Regarding the composition of the soils, an analysis of major compounds by X-ray fluorescence (XRF) and mineralogical analysis by X-ray diffraction (XRD) was carried out, as shown in Table 7.1 and Figure 7.3 and 7.4.

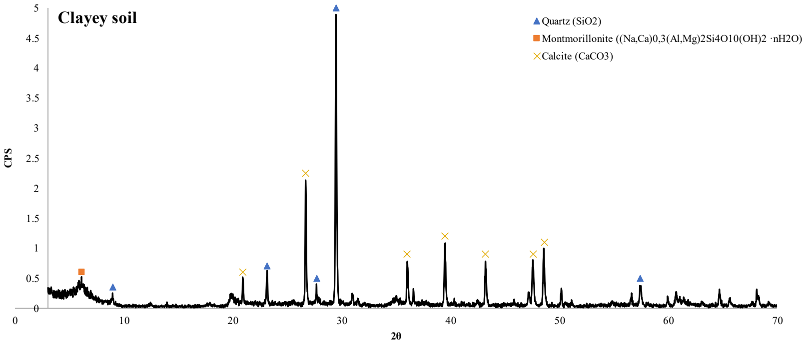


Fig. 7. 3. XRD of Clayey soil

The clayey soil presents a main composition of silicon, calcium and aluminium, typical composition of clays, which is observed in a mineralogy composed of quartz, calcite and montmorrollite type clay minerals.

The sandy soil has a main composition of calcium and magnesium, typical composition of dolomite minerals, as shown in the XRD of Figure 7.4

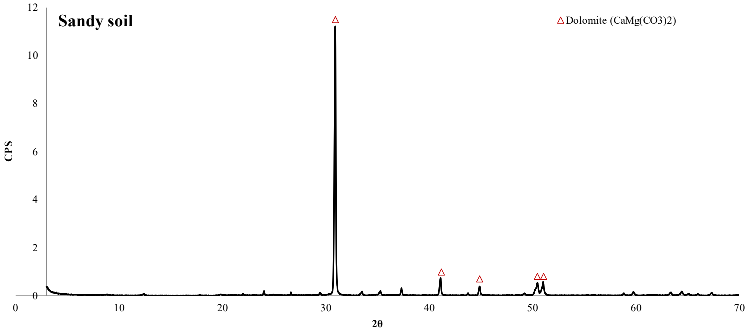


Fig. 7. 4. XRD of sandy soil

7.4.1.2.- Binders: quicklime and cement

Binders are materials that react chemically with water, forming cementitious compounds which can bind and improve the properties of soils, among other functions.

In the present work, two binders have been applied, commercial quicklime (QL) type CL 90-Q and a commercial ordinary Portland cement (OPC) type CEM II/B-L 32.5R. Table 7.2 shows the composition in the form of oxides of both binders.

Table 7. 2. Composition of binders

Composition (%) / Binder	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O
CEM II/B-L 32.5R	16.20	3.83	2.75	60.41	0.84	2.64	0.64
CL 90-Q	2.37	1.47	0.21	57.26	13.78	0.72	0.07

7.4.1.3.- Nanomaterials

Three types of silica-based nanomaterials were used in this study, named N1, N2 and N3.

N1 is a concentrated liquid solution of sodium silicate for soil stabilisation together with binders such as lime or cement. N2 is organo-silane and N3 is a silica-based acrylic co-polymeric in aqueous solution form, intended to be used together in a 1-1 ratio.

For the three nanomaterials, its chemical composition was obtained by X-ray fluorescence (XRF) analysis and a thermogravimetric analysis (TGA) were performed. Table 7.3 shows results of XRF analysis and Figure 7.5 shows the results of TGA.

As can be observed in Table 7.3, the composition of N1 was mainly silicon and sodium, elements that form the nano-sized sodium silicates that make up this material. N2 and N3 were composed only of silicon as the main element and traces of the other elements. This sodium is dissolved in the form of organ-silanes in N2 and in the form of acrylic co-polymer in N3.

Table 7. 3. Main composition XRF (%) of nanomaterials

Main Composition XRF (%)	N1	N2	N3
Si	24.200	40.600	38.400
Ca	0.279	0.014	0.097
Al	0.180	0.086	0.531
S	0.020	0.014	0.051
K	0.026	-	0.053
Mg	0.150	-	0.093
Fe	0.057	0.050	0.488
Na	12.100	-	0.207

The results of the TGA analysis are shown in Figure 7.5. In N1, a progressive weight loss was observed of up to 200°C, which is due to the loss of water in the nanosilane. This nanomaterial showed a high silica content of 32%; N2 showed a higher weight loss, the silica content at 400°C is 17%; N3completely loses its mass at a temperature of 400ºC.

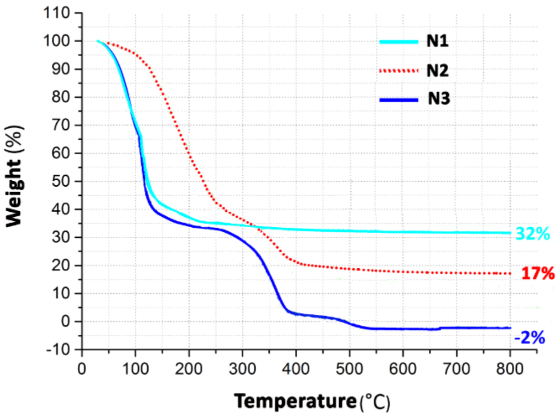


Fig. 7. 5. TGA curves of nanomaterials

The higher stability of silica in the form of sodium silicate compared to other solutions such as organo-silanes or acrylic co-polymers were observed, organosilane compounds lost 83% of their mass and acrylic compounds did not retain mass after the test.

7.4.2.- Phase 3: Mechanical behaviour of soil mixtures

In this section the requirements for thickness layers design are determined. Modified Proctor compaction test, California Bearing Ratio (CBR), unconfined compressive strength and shear test are performed and shown. Figure 7.6 shows photographs of experimental methods and materials applied in this work.

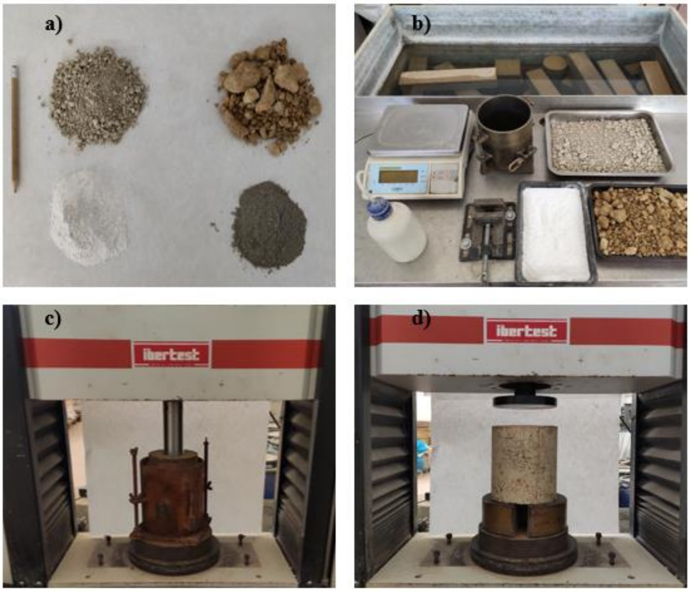


Fig. 7. 6. Experimental methods and materials photographs. a) Soils and binders; b) Modified Proctor test; c) CBR index test; d) Unconfined compressive strength test.

7.4.2.1.- Mix design

Six mixes of soil with binder and/or nanomaterials were defined to design the three alternative innovative sections studied in this article. Table 7.4 shows the mixtures analysed in laboratory, the dosage of each material and its designation.

The dosages of mixtures shown in the table above are expressed in weigh (kg) per cubic metre of material.

Table 7. 4. Dosages of the mixtures

Designation	**Materials (kg/m^3)**						
	CS	**SS**	**Quicklime**	**OPC**	**N1**	**N2**	**N3**
CS + 1.5%QL	1590.00	-	23.85	-	-	-	-
CS + 1.5%QL +N1	1590.00	-	23.85	-	0.910	-	-
CS + 1.5%QL +N2&N3	1590.00	-	23.85	-	-	1.000	1.000
AM-1	660.00	1380.00	9.90	20.40	-	-	-
AM-2	660.00	1380.00	9.90	20.40	0.910	-	-
AM-3	660.00	1380.00	9.90	20.40	-	1.000	1.000

Each material was added emulating the real process of road construction:

Quicklime was added to the dry mass of CS in all mixtures. OPC was added to the total dry mass of soil (CS and SS) and nanomaterials were added to the dry volume of CS.

Finally, AM-1, AM-2, and AM-3 were composed of 0.40 m3 of CS and 0.60 m3of SS per one cubic metre of the total mix.

The AM mixes were made with a special manufacturing process, which is explained below:

Step 1. 40% in volume of CS was mixed with an addition of 1.5 w% of quicklime (AM-1), 1.5 w% of quicklime plus N1 (AM-2) or 1.5 w% of quicklime and N2 plus N3 (AM-3). The mixture was compacted according to the Modified Proctor compaction test and stored in a wet chamber with a minimum moisture of 90%.

Step 2. One day after compaction the mixture was decompressed into particles with a size smaller than 25 mm.

Step 3. The stabilised CS was mixed with 60 v% of SS and 1 w% of OPC compared to the total dry mass of soil. The mixture was compacted according to the Modified Proctor test and stored in a wet chamber (compressive strength and shear tests) or curing tank (CBR test), before the samples were tested.

7.4.2.2.- Compaction test: Modified Proctor

The level of compaction is a key parameter in road layer construction. A proper compaction ensures the strength and durability of the road; conversely, if the road has not been adequately compacted, it becomes unstable which can result in a possible differential settlement. Settlement in road implies pavement deformation and cracking that, added to storm water infiltration, causes serious impact on traffic and road safety [35].

Compaction is studied through the moisture-dry density relationship, which is determined according to the Modified Proctor Test, UNE EN 103501: 1994 standard.

The moisture-dry density relationship, which is shown in Figure 7.7, is a very useful tool for understanding compacted soil behaviour. Maximum dry density to optimum moisture content is obtained, which allow the samples made to achieve optimal mechanical behaviour. Also, the curve shape indicates the sensitivity of the soil to water addition. If the curve is flatter, the maximum dry density is less affected by moisture changes; however, if the curve is sharper small changes in moisture content greatly affect the maximum dry density [36], as can be observed in Figure 7.7.

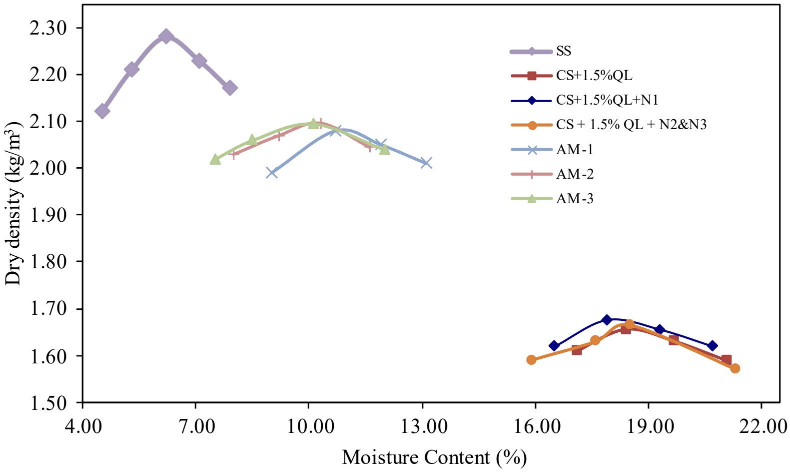


Fig. 7. 7. Moisture-dry density curves

Analysing Figure 7.7, SS shows the highest maximum dry density, 2.28 kg/m3, and minimum optimum moisture content 6.2%, likely due to its physical properties, such as a higher quantity of coarse particles and its higher density. However, CS stabilised with lime and quicklime plus nanomaterials show values contrary to SS due to the clay gradation, which is composed mainly of fine particles and its great absorption of water, which is a typical characteristic of expansive soil.

As can be observed, the addition of nanomaterials in the amount used in this article (0.056w% N1 and 0.12% N1&N2) shows a low effect on compaction parameters, especially in dry density which is consistent with the results obtained in previous studies of soil stabilisation with nanoparticles [29].

However, Alireza et al. [37] showed a decrease in the maximum dry density and an increase in the optimum compaction humidity in a soil stabilised with 5% lime from nano-SiO_2 additions greater than 1%.

Finally, the AM-1, AM-2 and AM-3 mixtures have an intermediate behaviour among the materials that compose them. A slight increase in the maximum dry density due to the addition of nanomaterials is observed in the AM-2 and AM-3 mixtures, likely due to the interaction of the stabilised soils with nanomaterials and the OPC.

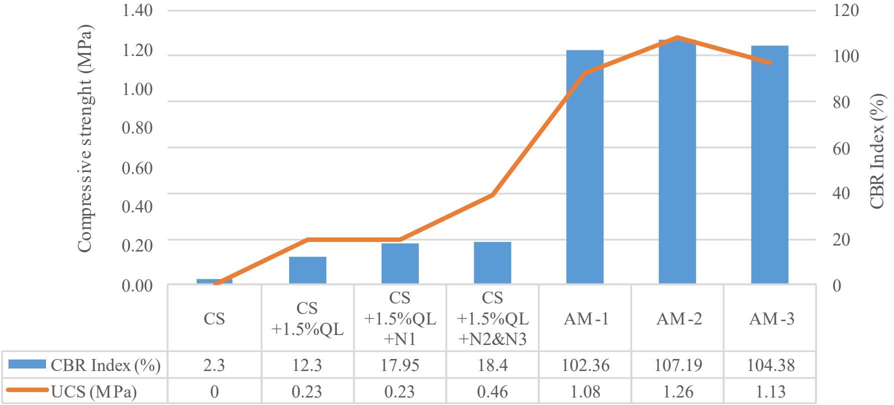
7.4.2.3.- Design parameters: California Bearing Ratio (CBR) and Compressive Strength

The structural behaviour of road layers is determined by the load bearing capacity. According to the type of road layer executed, this bearing capacity is measured according to the CBR (California Bearing ratio) or unconfined compressive strength.

The CBR index measures the bearing capacity of soils and compacted aggregates used in the construction of road bases or sub-bases. The CBR index depends on the density and moisture conditions of the samples. In this study, the samples were compacted according to the optimum moisture obtained in the Modified Proctor Test to reach maximum dry density and the highest possible bearing capacity. The CBR value is carried out in accordance with UNE 103-502

Unconfined compressive strength (UCS) was performed according to the NLT-305/90 standard in specimens 177.8 mm high and 152.4 mm in diameter. UCS measured the resistance in cohesive soil or cement treatment soil or granular materials. Like CBR samples, the samples were manufactured under the optimal compaction conditions obtained in the Modified Proctor Test.

Spanish specifications [34] establish that lime stabilised soil with lime must comply with the minimum CBR index; however, cement treated soil must exceed a minimum value of unconfined compressive strength. The results obtained in laboratory test are shown in Figure 7.8.



	CS	CS +1.5%QL	CS +1.5%QL +N1	CS +1.5%QL +N2&N3	AM-1	AM-2	AM-3
CBR Index (%)	2.3	12.3	17.95	18.4	102.36	107.19	104.38
UCS (MPa)	0	0.23	0.23	0.46	1.08	1.26	1.13

Fig. 7. 8. CBR Index and compressive strength

Analysing the results shown in Figure 7.8, the use of different nanosilica improve the bearing capacity of soils.

The addition of 0.056 w% of N1 and 0.112 w% of N2&N3 increase the CBR index by 31.5% and 33.2% respectively.

Similar increases in the CBR index have been observed in previous studies [38] with similar dosages, probably due to the reactions of these nanomaterials with cement and the soil minerals themselves, leading to pozzolanic cementitious reactions. This behaviour has been observed in previous studies, with higher increases in bearing capacity or simple compressive strength, due to the higher addition percentages of the nanomaterials, between 1-7% [37,39].

AM-1, AM-2 and AM-3 mixtures show similar CBR index values, in the order of 100% of CBR index with slightly higher values in the mixtures with nanomaterials, which shows an excellent bearing capacity. At the same time, unconfined compressive strength values of between 1.08-1.23 MPa are obtained, relatively high values for mixes with a total of 1 w%

cement, being normal values for stabilisation of a minimum of 3% according to Spanish specifications [34].

7.5.- Phase 4: Trial sections structural design

In this chapter, the design of three trial sections based on results obtained in laboratory tests are shown.

Three alternative sections (AS) were designed to reduce the thickness of the control section analysed by Rosales et al. [32] while the mechanical and durability properties of the current section were maintained or improved.

To guarantee the adequate structural performance of the alternative sections, a calculation process was carried out using EverStress5.0 software. The maximum load capacity of the control section, measured in maximum number of standard axles of 13-ton heavy vehicles, was determined and compared with the alternative sections, which must present a value equal to or greater than the control section.

To determine the maximum number of equivalent single axle loads of 13 tons, the following methodology was followed:

1) Determination of the elastic modulus (E) of the layers from CBR index or compressive strength according to the type of materials and its mechanical behaviour

2) Calculation of vertical deformations in the subgrade in Everstress 5.0 Software

3) Calculation of the number of equivalent axles according to the fatigue law described by the Spanish specifications [40] according to the expression (1).

$$\boldsymbol{\varepsilon_v = 2.16E - 2 * N_d^{-0.28}} \tag{1}$$

$\boldsymbol{\varepsilon_v}$ Unit vertical deformation in the subgrade

$\boldsymbol{N_d}$ Number of equivalent axles of 13 ton

In the first place, the calculation of materials with a reduced CBR, such as soil from the construction site or stabilised or granular materials that do not present significant compressive strength, were calculated by the Transport and Road Research Laboratory method [41] which is in accordance with the study of different bibliographies that indicate that the application of this method is appropriate for materials with CBR of less than 10% and without unconfined compressive strength.

The formula of Powell et al. [41] for stabilised materials and unbound granular materials was applied according to the expression (2).

$$\boldsymbol{E(MPa) = 17.6\ CBR^{0.64}} \tag{2}$$

Furthermore, with soils in which the CBR results were increased, a compressive strength test was carried out beforehand. The elastic modulus of these soils that present CBR values greater than 20% and compressive strength greater than 0.2 MPa was calculated by the Molenaar equation. This equation considers the unconfined compressive strength as the main modulus calculation parameter.

The formula of Molenaar [42] for materials treated with binders was applied according to the expression (3).

$$\boldsymbol{E(MPa) = 1435\ UCS^{0.885}} \tag{3}$$

Table 7.5. shows the elastic modulus of the analysed mixtures according to the expression shown above.

Table 7. 5. Elastic Modulus (MPa) of soils or mixtures

Soil or mixture	**Design parameter**		**Elastic modulus (MPa) by calculation**
	CBR (%)	**UCS (MPa)**	
CG*	60.05	-	242
CS + 1.5%QL	12.3	-	88
CS + 1.5%QL+N1	17.95	-	112
CS+1.5%QL+N2&N3	18.4		114
AM-1	-	1.08	1536
AM-2	-	1.26	1761
AM-3	-	1.13	1599

*Obtained from Rosales et al. [32]

Once the elastic modulus of the materials that make up each layer were determined, they were entered into the software together with the thicknesses of each layer and with a stress of 800 kPa, in accordance with Spanish regulations.

First, the conventional section was analysed, which presented a bearing capacity of approximately 75,000 equivalent axels.

Subsequently, three series of alternative sections (AS) AS-1, AS-2 and AS-3 of 45 cm, 50 cm, and 55 cm, maintaining the proportion of 40% CS and 60% SS of the design mixes were analysed.

Table 7.6 shows the results obtained for the vertical deformation of the subgrade and the number of equivalent axels for each solution.

Table 7. 6. Results of Vertical deformations in subgrade obtain in multilayers analysis and number of axles obtained by calculus

Section	Vertical deformations in the subgrade (ε_v) (10^{-6})	Number of equivalent axles of 13 ton
Conventional solution	**932.96**	**74,737.32**
AS-1-45cm	1138.72	36,678.91
AS-2-45cm	1049.18	49,140.69
AS-3-45cm	1095.34	42,136.58
AS-1-50cm	**880.26**	**91,986.36**
AS-2-50cm	**810.96**	**123,284,21**
AS-3-50cm	**850.40**	**104,052.13**
AS-1-55cm	750.50	162,585.39
AS-2-55cm	645.06	279,193.29
AS-3-55cm	678.72	232,814.61

As is observed in table 7.6, the 45 cm section series structural capacity was insufficient and was therefore rejected.

The 55 cm series exceeded by three times the required capacity of 75,000 equivalent axels and was therefore rejected in order not to oversize the section.

Therefore, the thickness of the section was 50 cm, due to a higher structural capacity than the control solution and a reduction in the section thickness of 30 cm.

7.6.- Phase 5: Construction and section in-situ tests

7.6.1.- Trials sections execution

After the design phase, the three trial sections of 50 cm total thickness were constructed, with a length of 100 m for alternative sections 1 and 2 (AS-1 and AS-2) and a length of 50 m for alternative section 3 (AS-3).

The three trial sections were built near the newly-built road in Villacarrillo, Jaén, Spain. [32] and the performance of the AS were compared with the control section which were used along the layout of the road. Figure 7.9 shows the control section and the location of the trial section.

Fig. 7. 9. General location of trial sections

The final solutions were as follows:

-Alternative section 1 (AS-1): 30 cm of CS (40%) stabilised with 1.5% lime and SS (60%) stabilised with 1% CEM-II over the total soil (CS+SS) carried out according to the process described in Figure 7.1, over a 20 cm layer of CS stabilised with 1.5% quicklime.

-Alternative section 2 (AS-2): 30 cm of CS (40%) stabilised with 1.5% lime and 0.056% N1 and SS (60%) stabilised with 1% CEM-II over the total soil (CS+SS) carried out according to the process described in Figure 7.1, over a 20 cm layer of CS stabilised with 1.5% lime and 0.056% N1.

-Alternative section 3 (AS-3): 30 cm of CS (40%) stabilised with 1.5% lime and 0.12% N2&N3 and SS (60%) stabilised with 1% CEM-II on the total soil (CS+SS) made according to the process described in Figure 7.1, on a 20 cm layer of CS stabilised with 1.5% lime and 0.12% N2&N3.

Figure 7.10 shows a scheme of the developed sections:

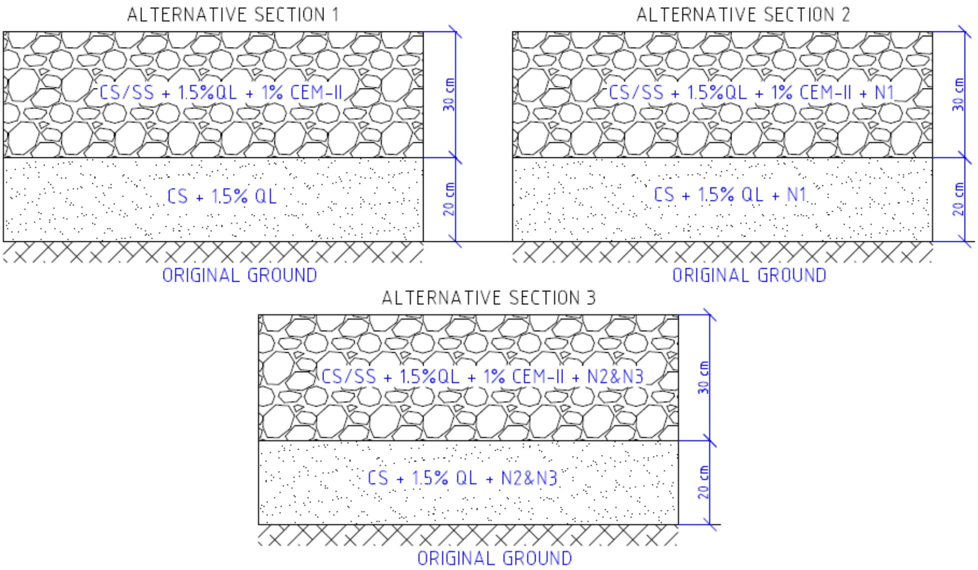


Fig. 7. 10. Trial sections scheme

7.6.2.- Trial sections survey

According to Spanish specifications [33] three categories of subgrades according to the equivalent elastic modulus are stabilised: low quality, 60-120 MPa, medium quality, 120-300 MPa and high quality, greater than 300 MPa.

For rural and low volume roads, a low-quality subgrade with a modulus between 60 and 120 MPa is structurally valid; however, due to the control section being designed as a medium quality subgrade, this criterion was maintained for the test sections, setting a minimum of 120 MPa equivalent modulus.

To determine and compare the equivalent modulus value, the following methods were analysed.

-Analysis of the theoretical deflection produced by a 500 kPa applied on a 300 mm diameter plate in a multilayer elastic model, the method specified in Spanish regulations [40]. The theoretical equivalent modulus value per section was obtained.

-Analysis of the deflections measured in the falling weight deflectometer (FWD) test. The average equivalent modulus value of the section was obtained.

-Analysis of the second load cycle in plate bearing test. A point value of equivalent modulus per section was obtained.

Table 7.7 shows the result obtained in the Everstress 5.0 program analysing the multilayer elastic model with a stress of 500 kPa applied on a 300 mm diameter plate. Applying the formula (4), the equivalent modulus of compressibility of each section, Ev, was determined.

$$\boldsymbol{E_v(MPa) = 13.150/d_0(mm/100)}\ [40] \tag{4}$$

Table 7. 7. Theorical deflection and modulus of compressibility obtained in Everstress 5.0

Everstress results	AS-1	AS-2	AS-3
Theorical deflection (mm/100)	78.02	73.51	75.63
Theorical modulus of compressibility, Ev (MPa)	168.55	178.89	173.87

As can be observed in table 7.7, the three alternative sections present a sufficient theoretical equivalent modulus to be considered medium quality subgrade.

Once the theoretical equivalent modulus values for each section have been obtained, they are verified with the values obtained in the in-situ tests, FDW and plate bearing test.

As Figure 7.11 shows, the climatology of the area shows two distinct annual seasons, with 6 months with abundant rainfall and 6 dry months [43]. Therefore, two in-situ test campaigns were carried out: the first in

July 2020, in the dry season, and the second in March 2021, in the wet season.

Once the data for both periods had been obtained, the average annual value of equivalent modulus was obtained for each of the methods.

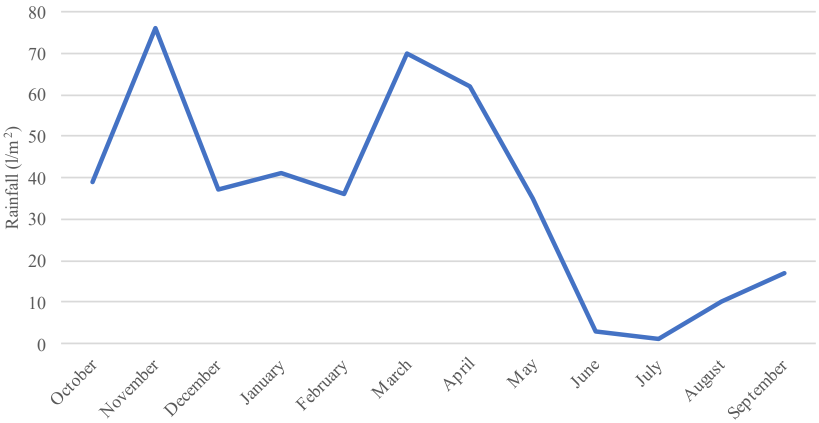


Fig. 7. 11. Annual rainfall distribution in Villacarrillo, Spain

7.6.2.1.- Deflection measurements by FWD

The deflection measurement enables characterising the structural capacity of the formed subgrade as well as its layers along the road layout. A Dynatest HDW 8081 falling weight deflectometer (FWD) was used.

A pressure of 850 kPa was applied through 300 mm diameter plate. The surface deformation due to the application of this load was measured by seven geophones located at 0-300-450-600-900-1200-1500 mm from the application of the load.

Surface deformation measurements were taken every 10 m in sections AS-1 and AS-2 and every 5 m in section AS-3 in both lanes, obtaining the average per section.

Figure 7.12, Figure 7.13 and Figure 7.14 show the mean deflection values measured in the dry and wet seasons for each section.

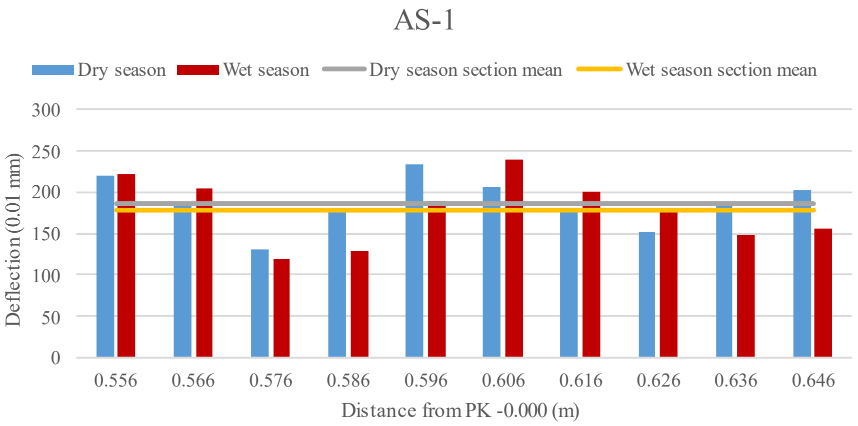


Fig. 7. 12. Deflection measurement in AS-1

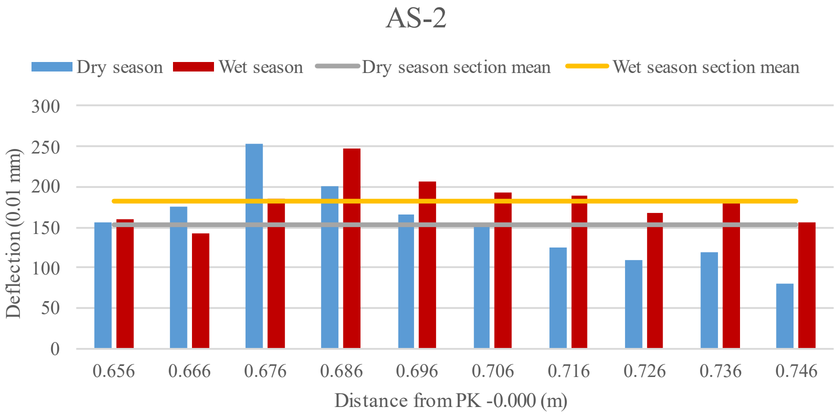


Fig. 7. 13. Deflection measurement in AS-2

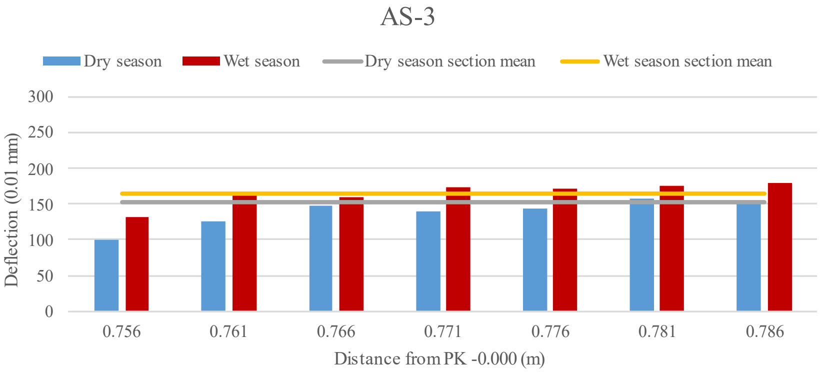


Fig. 7. 14. Deflection measurement in AS-3

Analysing Figure 7.12, Figure 7.13 and Figure 7.14, two patterns of behaviour were observed: firstly, the addition of nanomaterials, both sodium silicates and organo-silanes together with acrylic co-polymers, improved the bearing capacity of the soil, reducing the deflections measured during the test.

Secondly, the sections with nanomaterials showed a drop in bearing capacity due to rainfall, in contrast to the AS-1 section, which remained practically constant after a year's weather.

However, the AS-1 section showed greater dispersion in the data, which could be attributed to problems during the execution of the work, presenting over-compacted areas that reduce the average deflection of the section.

Table 7.8 shows the average annual deflection of each section, as well as the weighted deflection for a load of 500 kPa, which enables calculating the elastic modulus through equation (4).

Table 7. 8. Modulus of compressibility mean in each section

Trial section	Anual average deflection (0.01 mm)	weighted deflection (0.01 mm)	Ev (MPa)
AS-1	183	108	122
AS-2	168	99	133
AS-3	159	93	142

As is shown in Table 7.8, the average values of the sections show a reduction in the average annual deflections and an increase in the equivalent modulus of the sections made with nanomaterials.

Comparing the deflection results obtained for the new hybrid stabilised sections, a better behaviour is observed than the alternative section analysed by Rosales et al. [32], and a slight drop is observed when compared to the 80 cm thickness control section. However, the section reduction of approximately 40% and a similar mechanical behaviour confirm the suitability of the proposed method of hybrid stabilised solutions for road layers.

All the sections can be classified as medium category, although with lower values than those obtained theoretically, due to the irregularities of the terrain and construction peculiarities of the linear works.

7.6.2.2.-Plate bearing Test

The plate load test is an in-situ test used to measure the final bearing capacity of the subgrade built from the sections designed through its compressibility modulus.

The plate bearing test was performed according to UNE 103808:2006 and it consists in measuring the settlement of a rigid circular plate resting on the ground, subjected to different loads in a staggered manner, called load cycle. This circular-shaped plate has a surface area of 700 cm^2 (diameter 298.5 mm) and the measurements enable determining the compressibility modulus in the first load cycle (Ev1) and in the second load cycle (Ev2).

The plate bearing test enables the calculation of the punctual behaviour of the section in the place where the load was applied, so its result was not as representative as those obtained in FDW; however, it enables verifying the behaviour of the subgrade and analysing its evolution over time.

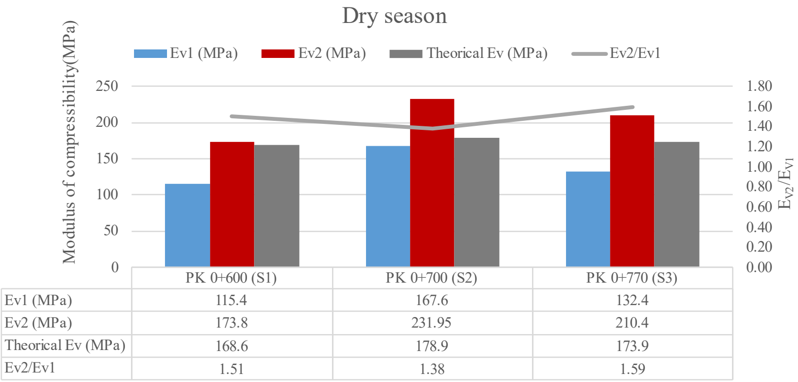
The results of plate load test are shown in Figure 7.15, Figure 7.16 and Figure 7.17.

As is shown in Figure 7.15, all sections presented a medium quality, which verifies adequate structural behaviour in relation to the volume of loads the sections will support during their useful life.

An improvement in the quality of subgrade AS-2 and AS-3 was observed due to the addition of nanomaterials, similar to that observed in FDW results.

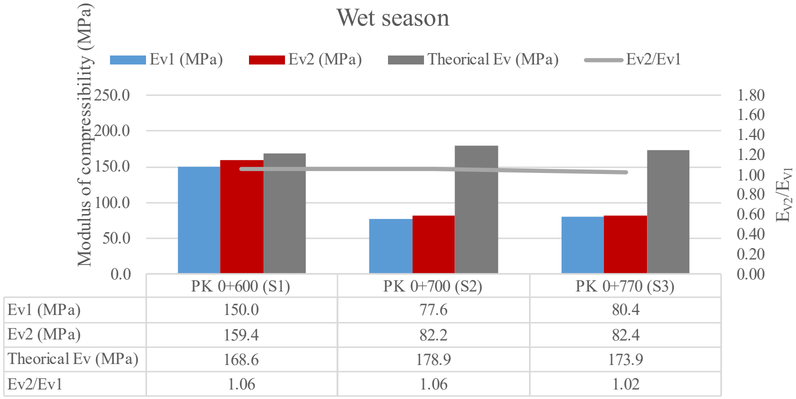
Analysing Figure 7.16, a drop in the equivalent modulus was observed in all three sections due to the rainy period; however, this drop was greater in sections AS-2 and AS-3 than AS-1.

This sharp drop may be due to a lack of bearing capacity in some of the layers that make up the section because of the rain, or perhaps a specific problem associated with the uncertainty of the load plate test.



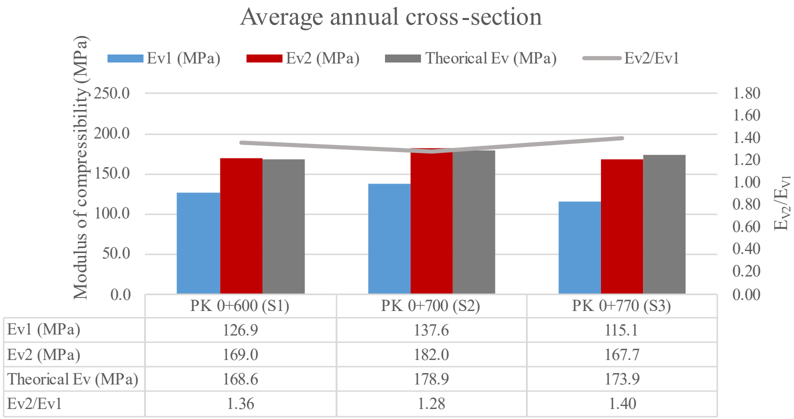
	PK 0+600 (S1)	PK 0+700 (S2)	PK 0+770 (S3)
Ev1 (MPa)	115.4	167.6	132.4
Ev2 (MPa)	173.8	231.95	210.4
Theorical Ev (MPa)	168.6	178.9	173.9
Ev2/Ev1	1.51	1.38	1.59

Fig. 7. 15. Plate bearing test results in dry season



	PK 0+600 (S1)	PK 0+700 (S2)	PK 0+770 (S3)
Ev1 (MPa)	150.0	77.6	80.4
Ev2 (MPa)	159.4	82.2	82.4
Theorical Ev (MPa)	168.6	178.9	173.9
Ev2/Ev1	1.06	1.06	1.02

Fig. 7. 16. Plate bearing test results in wet season



	PK 0+600 (S1)	PK 0+700 (S2)	PK 0+770 (S3)
Ev1 (MPa)	126.9	137.6	115.1
Ev2 (MPa)	169.0	182.0	167.7
Theorical Ev (MPa)	168.6	178.9	173.9
Ev2/Ev1	1.36	1.28	1.40

Fig. 7. 17. Annual average plate bearing test results

Finally, Figure 7.17 shows the average annual behaviour of the sections. Despite the large drop observed in the rainy weather for sections AS-2 and AS-3, an adequate mechanical behaviour was observed, being practically identical to that obtained in the theoretical analysis using the multilayer elastic model, which confirms the validity of the multilayer elastic method used and the validity of the sections constructed.

Comparing the results with those obtained by Rosales et al. [32], an increase in the modulus of compressibility of the current hybrid sections is observed. This increase confirms the adequate behaviour of the new solutions constructed with a reduction of the input materials.

Additionally, the evolution of the EV_2/EV_1 ratio is shown, limited by Spanish specifications to 2.2, a limitation that is met by all sections in both dry and wet seasons.

7.7.- Conclusions

In this work, the construction of trial sections based on hybrid stabilised soils with small amounts of lime, cement and nanomaterials has been studied. For this purpose, an in-depth laboratory study was carried out to determine the physicochemical properties of the materials and the mechanical behaviour of the mixtures of stabilised soil. A structural design of the trial sections was carried out through a multilayer elastic model. Finally, the sections were built in a real scale application and their short and medium-term in situ properties were monitored.

The following conclusions have been drawn from this study:

- The stabilisation of soils with nanosilica slightly increases the maximum dry density of the samples and slightly reduces their optimum water content. A reduction in optimum water content implies a reduction in the consumption of natural resources (water, fuel, less CO2 emmissions…) to achieve same or higher degree of compaction.

- The addition of nanosilica improves the bearing capacity of the stabilised soils as measured by the CBR index. The nanomaterial N1, composed of sodium silicate, shows a higher reactivity in combination with cement, increasing the unconfined compressive strength.

-The proposed hybrid stabilisation solution reduces the thickness of the control section by 37.5% and increases the bearing capacity measured in number of equivalent axles by up to 25%.

-The sections built with N1 and N2&N3 show an improvement in the annual mean equivalent modulus of compressibility, with these sections showing a greater drop in bearing capacity during the rainy season.

-The results support the application of the developed alternatives solutions with a hybrid stabilisation process due to their increased bearing capacity for rural and low volume roads. However, due to the typical climatic conditions in southern Spain, there is no evidence of effectiveness in other conditions. In countries with high humidity and large temperature differences the solution should be verified

As a general conclusion, the use of nanomaterials in percentages between 0.06-0.12% improves the mechanical behaviour of stabilised soils and allows a reduction in the thickness of the road layer, improving its general structural capacity, although there is a drop in capacity during rainy periods, which will be the subject of future studies.

Author Contributions

Conceptualization, F.A. and J.R.M.; methodology, J.R. and F.A.; investigation, J.L.D.-L. and M.C.; resources, F.A. and J.R.M.; data curation, J.L.D.-L. and J.R.; writing—original draft preparation, J.L.D.-L. and M.C.; writing—review and editing, M.C., J.R. and J.L.D.-L.; supervision, F.A.; All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest

The authors declare no conflict of interest.

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CAPÍTULO VIII

CONCLUSIONES
CONCLUSIONS

VIII CONCLUSIONES / CONCLUSIONS

8.1.- Conclusiones

De acuerdo con los resultados obtenidos a lo largo de la investigación desarrollada en la presente Tesis Doctoral, y expuestos en los Capítulos IV-V-VI-VII, las siguientes conclusiones son extraídas:

En relación con las propiedades físicas y químicas de los ARM:

1. Los áridos reciclados mixtos, sometidos al tratamiento adecuado en planta, presenta una distribución granulométrica continua. Esta distribución granulométrica está dentro de los rangos de aplicación como zahorra compactada en capas de carreteras.
2. Los ARM procedentes de RCD, sometidos a un procesamiento en planta menos exigente, presentan un mayor porcentaje de yeso en su composición, lo que implica un elevado porcentaje de sulfatos solubles. Este hecho limita la aplicación de estos materiales debido a problemas ambientales y técnicos.

En relación con las propiedades físicas y químicas de los subproductos analizados, CFB, CVB y EBA:

1. Las CFB presentan una granulometría continua, presentando partículas desde los 80 mm hasta las pocas micras. Es un material altamente friable, pudiendo reducirse el tamaño de partícula con cargas mecánicas moderadas.
2. Analizando la composición obtenida, las CFB se componen de porcentajes elevados de calcio, sílice y potasio. Mineralógicamente, se componen de cuarcita, calcita, dolomita y aluminosilicatos. Esta composición, indica un potencial reactivo bajo condiciones de pH básicos, fomentando así las reacciones puzolánicas.
3. Las CVB, debido a su definición, presentan una distribución granulometría compuesta de partículas de muy reducido tamaño, con un porcentaje del 66% en peso inferior a 63 micras. Esta elevada finura aumenta su reactividad con los suelos. Su composición, si bien incluye aluminosilicatos, es mayoritaria en carbonatos y sulfatos potásicos.
4. La EBA se caracteriza principalmente por presentar una gran proporción de partículas finas y una composición rica en sílice,

aluminio y calcio. Estas características hacen que sea un material con gran potencial estabilizar de suelos, debido su reactividad hidráulica y puzolánica.

En relación con las propiedades físicas y químicas de los nanomateriales:

1. Tres nanomateriales han sido analizados en la presente Tesis Doctoral. El primero, con una composición mayoritaria de sodio y sílice, siendo una solución de sodio silicato. El segundo y tercer nanomateriales, denominados organosilano y solución acrílica de sílice, están compuestos por sílice con trazas de otros elementos.
2. Los nanomateriales analizados se presentan como soluciones líquidas de sílice nanométrico, con una densidad similar a la del agua, con objeto de añadirlo disuelto en este elemento.
3. Debido a su tamaño nanométrico, presentan una alta superficie específica, reduciendo así el porcentaje de dosificación, siendo del orden entre 0.06-0.12% en peso sobre la masa de suelo seco a estabilizar.

En relación con la aplicación de ARM con diferentes procesamientos en planta como zahorra compactada:

1. Los ARM provenientes de RCD con un proceso de recogida selectiva en origen, presentan una composición con menor porcentaje de yeso y otros materiales no deseados, lo que implica una mejora en las propiedades físicas con tratamientos menos complejos, resultando en beneficios técnicos y ambientales.
2. Los ARM presentan una densidad máxima de compactación inferior a los áridos naturales, este hecho facilita el procedimiento constructivo de las bases de carretera, debido a los menores requerimientos de medios mecánicos para alcanzar los parámetros de compactación deseados.
3. La capacidad portante de los ARM es de una excelente calidad, incluso en los áridos obtenidos mediante tratamientos de RCD menos exigentes, permitiendo su aplicación de acuerdo con criterios técnicos. Los ARM procedentes de RCD con recogida selectiva alcanzan valores de capacidad portante similar a zahorras compactadas convencionales.

En relación con la estabilización de suelos arcillosos expansivos con nanomateriales:

1. La capacidad portante del suelo arcilloso estabilizado con cal y nanomateriales presenta un aumento de aproximadamente el 30% sobre la mezcla sin nanomateriales, medida de acuerdo con el índice CBR.
2. La adición de nanomateriales base sílice implica una mejora en las propiedades geotécnicas de los suelos, reduciendo la plasticidad, hinchamiento libre y potencial de colapso. Sin embargo, no implica un aumento significativo de la conductividad hidráulica a los 7 días con respecto a las adiciones sin nanomateriales.

En relación con la estabilización de suelos arcillosos expansivos con los subproductos analizados, CFB, CVB, EBA, pARM y/o nanomateriales.

1. Al realizar un análisis con distintas dosificaciones de cenizas de biomasa como material estabilizador de suelos, se observa que la adición de **CFB** en un porcentaje en peso del 15% sin otro material estabilizador no implica una mejora sustancial en las propiedades de compactación, estabilización o aumento de capacidad de carga. De cualquier modo, existe un aumento de las propiedades analizadas debido a la modificación física del suelo.
2. La adición de **CVB** en un porcentaje del 15% sin otro material estabilizador implica mejoras muy significativas en las propiedades geotécnicas y mecánicas del suelo. Esto es debido a las modificaciones físicas de la adición, así como los procesos de floculación y endurecimiento por carbonatación asociados a las cenizas volantes.
3. La combinación de un 5% de cenizas de biomasa (CFB o CVB) con 1% de cal, elimina la plasticidad, hinchamiento y colapso del suelo arcilloso analizado. Asimismo, mejora la capacidad portante de los suelos hasta valores superiores a los límites de la normativa española para categorizarse suelo estabilizado tipo S-EST2.
4. Los ensayos obtenidos a través del ensayo triaxial consolidado no drenado realizado sobre las mezclas de suelo estabilizado con cenizas de biomasa indican la idoneidad de uso para formación de terraplenes y formación de taludes, ampliando el rango de aplicación de los nuevos materiales estabilizadores reciclados.
5. Analizando las propiedades geotécnicas (plasticidad, hinchamiento libre y colapso) de las mezclas estabilizadas con EBA y pMRA

analizadas, se observa que todas las mezclas exhibieron mejoras. Esto puede atribuirse a la interacción entre la cal y el suelo, así como a la adición de subproducto (en un rango del 5%), que modificó las propiedades físicas.

6. La interacción de los nanomateriales con los cuatro subproductos analizados condujo a un aumento del límite plástico del suelo, resultando en una reducción significativa de la plasticidad, del hinchamiento y del colapso en los suelos estabilizados.
7. Analizando la capacidad portante de las mezclas de suelo estabilizado desarrolladas, se observó que una combinación de 5% de los subproductos analizados y 1% de cal permitía en todos los casos una capacidad portante suficiente para ser aplicada en capas de carretera. Esto se debe al efecto de la cal y a la mejora de las propiedades físicas del suelo resultante de la adición de cualquiera de los subproductos.
8. Al combinar 5% de subproducto industrial, 0,5% de cal y nanomateriales, se observa una mejora sustancial de la capacidad de carga en las mezclas. La combinación de CVB, cal y nanomaterial exhibe un aumento del 300%, mientras que CFB y pARM, cal y nanomaterial mostraron alrededor de un aumento del 200%. La mezcla con EBA, cal y nanomaterial obtiene una mejora del 122%.
9. Los resultados obtenidos en el estudio mineralógico confirmaron la formación de geles CSH/CASH en todas las mezclas en las que se combinaron subproductos con nanomateriales. Hubo una correlación entre la reducción de la cristalinidad de las mezclas cuando se combinaron subproductos con nanomaterial y la mejora de las propiedades mecánicas y geotécnicas, lo que indica la formación de geles derivados de reacciones puzolánicas.

En relación con la construcción de un tramo de carretera a escala real:

1. La construcción de un tramo experimental a escala real de carretera, permitió verificar el aumento en los parámetros técnicos medidos en las secciones donde se aplican nanomateriales, permitiendo una reducción del 37.5% del espesor con respecto una solución convencional.
2. Las secciones de carretera construidas donde se aplican nanomateriales muestran una mejora en el módulo de compresibilidad anual medido. Sin embargo, estas secciones exhiben una caída en el módulo medido tras la estación lluviosa.

En relación con el impacto ambiental causado por lixiviación:

1. Los ARM con porcentaje en peso de yeso superior al 5% presentan problemas de lixiviación por sulfatos, de acuerdo con la directiva de admisión a vertederos 2003/33/EEC. Sin embargo, se observa una falta de legislación específica acerca de la contaminación ambiental por sulfatos en capas de carreteras por aplicación de ARM. Los limites propuestos en la presente Tesis Doctoral permitirían la ampliación del rango de utilización de los ARM garantizando un adecuado desempeño estructural sin comprometer el medio ambiente.
2. Los ARM procesados y aplicados como material estabilizador de suelos no presentan problemas de lixiviación de metales pesados, debido al porcentaje de aplicación (5% en peso).
3. Los subproductos analizados (CFB, CVB y EBA), presentan problemas de lixiviación de metales pesados. Las CFB, son catalogadas como material no peligroso, al superar los límites permitidos en Cr, Se y Mo. Las CVB y EBA, son clasificados como material peligroso. Las CVB exceden el límite en Cr, así como en As y Se. Las EBA presentan valores muy altos en Mo.
4. La aplicación de CFB y EBA en un porcentaje del 5% en peso, como material estabilizador no presenta riesgos medioambientales por lixiviación de metales pesados, clasificándose las mezclas como material inerte de acuerdo con el Test de Percolación en columna. Los porcentajes aplicados junto con la inmovilización de metales pesados en el proceso de estabilización de suelos garantizan su aplicación en condiciones de seguridad.
5. El uso de CVB como material estabilizador de suelos presenta problemas ambientales al encontrar una concentración de cromo lixiviado mayor a la establecida en la normativa. La aplicación de un 5% de CVB implica que la mezcla de suelo estabilizado sea clasificada como material peligroso.

En relación con el análisis de ciclo de vida:

1. El uso de ARM en lugar de árido natural como zahorra compactada en la construcción de bases de carreteras, implica un enorme beneficio ambiental. En términos de emisiones de CO_2 a la atmosfera, la aplicación de ARM obtenido a partir de RCD con recogida selectiva en origen implica una reducción de aproximadamente el 75% de emisiones de gases de efecto invernadero a la atmosfera, garantizando un adecuado comportamiento técnico y evitando problemas debidos a la producción de lixiviados.

2. La producción de nanomateriales y cal generan elevadas cargas ambientales debido al consumo de recursos minerales y fósiles, las emisiones a la atmósfera producidas en su fabricación y los vertidos de agua. Sin embargo, comparando los porcentajes recomendados de aplicación de nanomateriales (0.06-0.12%) y de cal (2% mínimo de acuerdo al PG-3), el uso de nanomateriales implica una reducción de las emisiones en procesos de construcción de carreteras.
3. La producción de los subproductos analizados (CFB, CVB, EBA y pMRA) requiere un procesamiento mínimo en comparación con la producción de cal y nanomateriales, implicando una gran reducción en la carga ambiental.
4. La incorporación de subproductos industriales para la estabilización de suelos arcillosos reduce las cargas ambientales en casi todas las categorías del ACV en comparación con la mezcla estabilizada con cal al 1,5%. El uso combinado de subproductos industriales y nanomateriales reduce aún más los valores de impacto al disminuir la proporción de cal en la dosificación de la mezcla (de 1% a 0,5%).

Finalmente, y en base a la investigación realizada durante el desarrollo de la presente Tesis Doctoral, se pueden extraer las siguientes conclusiones generales:

Los áridos reciclados, cenizas de fondo y volantes de biomasa, escorias blancas de acería son subproductos industriales con un alto valor añadido, y un gran potencial como material alternativo a aplicar para la ejecución de capas de carreteras, reduciendo así el uso de materiales estabilizadores convencionales, tierras de préstamo y depósito de suelos marginales a vertedero.

El estudio de estos subproductos ha demostrado su validez para ser aplicados en base de carretera (ARM como zahorra compactada) y como materiales estabilizadores de suelos in situ. Además, la combinación de estos materiales reciclados con nanomateriales permite alcanzar propiedades técnicas que superan en ciertas medidas a los materiales convencionales, reduciendo los lixiviados de metales pesados y así como las emisiones de CO_2 y las afecciones al medio.

En definitiva, la aplicación de estos materiales es técnicamente viable, así como ambientalmente sostenible, fomentado el crecimiento económico dentro de un modelo de economía circular.

8.2.- Conclusions

In light of the research conducted in this PhD Thesis, encompassing Chapters 5-8, the following conclusions are drawn:

Regarding the physical and chemical properties of Mixed Recycled Aggregates (MRA):

1. Mixed recycled aggregates, after undergoing appropriate plant treatment, exhibit a continuous grain size distribution within the optimal range for use as a compacted sub-base in road construction.
2. MRA derived from Construction and Demolition Waste with less rigorous plant processing demonstrate a higher gypsum content in their composition, indicating a substantial presence of soluble sulfates.

Regarding to the physical and chemical properties of the analyzed by-products, Biomass Bottom Ash (BBA), Biomass Fly Ash (BFA), and Steel Slag (SS):

1. BBA showcases a continuous particle size distribution, ranging from 80 mm down to a few microns. It is a friable material, with particle size reduction occurring under moderate mechanical stress.
2. Compositional analysis reveals that BBA is rich in calcium, silica, and potassium. From a mineralogical perspective, it predominantly consists of quartzite, calcite, dolomite, and aluminosilicates, indicating a potential for reactivity under basic pH conditions and facilitating pozzolanic reactions.
3. BFA, inherently fine-grained, features approximately 66% by weight with a particle size less than 63 microns. This fine texture enhances its reactivity with soils. Although it contains aluminosilicates, its primary constituents are carbonates and potassium sulfates.
4. SS is characterized by a significant proportion of fine particles and a composition rich in silica, aluminum, and calcium. These attributes render it a material with substantial potential for soil

stabilization, owing to its hydraulic and pozzolanic reactivity.

Regarding the physical and chemical properties of nanomaterials:

1. This PhD Thesis examined three nanomaterials. The first, primarily composed of sodium and silica, is a sodium silicate solution. The second and third nanomaterials, referred to as organosilane and silica acrylic solutions, are predominantly silica with trace amounts of other elements.
2. These nanomaterials are presented as nanometric silica liquid solutions, with a density similar to water, intended for dissolution in water.
3. Their nanometric size results in a significantly high specific surface area, thereby reducing the required dosage percentage to a range of 0.06-0.12% by weight of the dry soil mass for stabilization.

Regarding the application of Mixed Recycled Aggregates (MRA) with different in-plant processing as compacted aggregates:

1. MRA derived from CDW with a selective collection process at the source exhibit compositions with lower gypsum and undesirable material percentages. This implies improved physical properties with less complex treatments, yielding both technical and environmental benefits.
2. MRA possess a lower maximum compaction density compared to natural aggregates, which facilitates the construction of road bases by requiring fewer mechanical means to achieve desired compaction parameters.
3. The bearing capacity of MRA is of excellent quality, even in aggregates obtained from less demanding CDW treatments. They meet application criteria and demonstrate bearing capacity values similar to those of conventionally compacted aggregates when derived from separately collected CDW.

Regarding the stabilization of expansive clay soils with nanomaterials:

1. Stabilizing clayey soil with lime and nanomaterials enhances its bearing capacity, as measured by the CBR index, by

approximately 30% compared to the mixture without nanomaterials.

2. The addition of silica-based nanomaterials implies an improvement in the geotechnical properties of the soils, reducing plasticity, free swelling and collapse potential. However, it does not imply a significant increase in hydraulic conductivity at 7 days compared to additions without nanomaterials.

Regarding the stabilization of expansive clayey soils using the analyzed by-products (BBA, BFA, SS), pMRA, and/or nanomaterials:

1. When analyzing different dosages of biomass ash as a soil stabilizer, it is evident that adding 15% BBA as a sole stabilizer does not result in a significant improvement in compaction, stabilization, or load-bearing properties. However, improvements are observed in these properties due to the physical modification of the soil.
2. Adding 15% BFA as the sole stabilizing material leads to substantial improvements in the geotechnical and mechanical properties of the soil. These improvements are a result of physical modifications and the flocculation and carbonation hardening processes associated with fly ash.
3. Combining 5% biomass ash (BBA or BFA) with 1% lime eliminates plasticity, swelling, and collapse in the analyzed clayey soil. It also enhances the bearing capacity of the soil to levels exceeding Spanish regulations, categorizing it as stabilized soil type S-EST2.
4. Undrained consolidated triaxial tests performed on the biomass ash-stabilized soil mixtures demonstrate their suitability for embankments and slope formation, expanding the range of application for these new recycled stabilizing materials.
5. All SS and pMRA stabilized mixtures tested exhibited improvements in geotechnical properties (plasticity, free swelling, and collapse). These enhancements can be attributed to the interaction between lime and soil, as well as the addition of the by-product (at a 5% rate), which altered the physical properties.

6. The interaction of nanomaterials with the four analyzed by-products led to an increase in the plastic limit of the soil, resulting in a significant reduction of swelling and collapse in the stabilized soils.
7. Evaluating the bearing capacity of the developed stabilized soil mixtures, it was observed that a combination of 5% of the analyzed by-products and 1% lime consistently provided sufficient bearing capacity for use in road layers. This is due to the effects of lime and the improved physical properties of the soil resulting from the addition of any of the by-products.
8. By combining 5% of the analyzed by-products, 0.5% lime, and nanomaterials, substantial improvements in load-carrying capacity are observed in the mixtures. The combination of BFA, lime, and nanomaterials exhibits a 300% increase, while BBA and pMRA, lime, and SSBA show approximately a 200% increase. The mixture with SS, lime, and nanomaterials shows a 122% improvement.
9. Mineralogical analysis results confirm the formation of CSH/CASH gels in all mixtures when by-products are combined with nanomaterials. A correlation is observed between the reduction in crystallinity in blends when IBA are combined with nanomaterials and the improvement in mechanical and geotechnical properties, indicating the formation of gels resulting from pozzolanic reactions.

Regarding to the construction of a full-scale road section:

1. The construction of a full-scale experimental road section validated the increased technical parameters in sections where nanomaterials were applied, allowing for a 37.5% reduction in thickness compared to conventional solutions.
2. Road sections constructed applying nanomaterials show an improvement in the measured annual compressibility modulus. However, these sections exhibit a drop in the measured modulus after the rainy season.

Regarding the environmental impact caused by leaching:

1. MRA with a gypsum content exceeding 5% by weight may lead to sulphate leaching issues, as per the landfill directive 2003/33/EEC. However, specific regulations concerning sulphate-related environmental pollution in road layers due to MRA application are lacking. The limits proposed in this Doctoral Thesis aim to expand the utilization of MRAs, ensuring structural performance while safeguarding the environment.
2. The pMRA, processed and applied as a soil stabilizer material, does not pose heavy metal leaching concerns due to the limited application percentage (5% by weight).
3. The analyzed by-products (BBA, BFA, and SS) are associated with heavy metal leaching problems. BBA is categorized as non-hazardous material but exceeds permitted limits for Cr, Se, and Mo. Both BFA and SS are classified as hazardous materials, with BFA exceeding limits in Cr, As, and Se, and SS exhibiting notably high values for Mo.
4. The application of BBA and SS as stabilizing materials at a 5% weight percentage does not present environmental risks due to heavy metal leaching, classifying the mixtures as inert material based on column percolation tests. The applied percentages, coupled with the immobilization of heavy metals in the soil stabilization process, ensure safe application.
5. The use of BFA as a soil stabilizer material is not recommended. Mixtures developed with 5% BFA show Cr values exceeding regulatory limits.

Regarding the life cycle analysis:

1. Substituting natural aggregates with MRA as compacted aggregates in road base construction carries significant environmental benefits. In terms of CO_2 emissions, applying an MRA obtained from CDW with selective source collection reduces greenhouse gas emissions by approximately 75%, ensuring technical performance and mitigating leachate production issues.

2. The production of nanomaterials and lime generates substantial environmental burdens due to resource consumption, emissions during manufacture, and water discharges. However, comparing the recommended application rates of nanomaterials (0.06-0.12%) and lime (2% minimum according to PG-3), the use of nanomaterials implies a reduction of emissions in road construction processes.
3. The production of the analyzed by-products (BBA, BFA, SS, and pMRA) involves minimal processing compared to the production of lime and nanomaterials, resulting in a substantial reduction in environmental impact.
4. Incorporating industrial by-products for clay soil stabilization reduces environmental loads across various LCA categories compared to a 1.5% lime-stabilized mixture. The combined use of industrial by-products and nanomaterials further reduces impact values by reducing the proportion of lime in the mixture dosage from 1% to 0.5%.

Based on the research conducted during the development of this Doctoral Thesis, the following general conclusions can be drawn:

Recycled aggregates, bottom ash, biomass fly ash, and white steel slag represent industrial by-products with high added value and significant potential as alternative materials for road construction. This potential allows for a reduction in the use of traditional stabilizing materials, the need for borrowing earth, and the disposal of marginal soils in landfills.

The study of these by-products has demonstrated their suitability for use in road base applications (such as Mixed Recycled Aggregates for compacted gravel) and as in-situ soil stabilizing materials. Furthermore, when these recycled materials are combined with nanomaterials, it becomes possible to achieve technical properties that surpass those of conventional materials, while concurrently reducing heavy metal leachates, CO_2 emissions, and overall environmental impact.

In summary, the application of these materials is both technically feasible and environmentally sustainable. This approach promotes economic growth within a circular economy model.

8.3.- Futuras líneas de investigación

La realización del presente trabajo responde a los objetivos planteados acerca de la posible aplicación de materiales reciclados y nanomateriales para su aplicación en distintas tipologías de capas de carreteras. Del conocimiento adquirido se plantean nuevas líneas de investigación mostradas a continuación:

Probada la viabilidad de uso de los distintos subproductos en combinación con pequeñas cantidades de cal y nanomateriales para su aplicación en la estabilización de suelos arcillosos expansivos, se plantea la posibilidad de producir un producto terminado compuesto por un subproducto, un activador alcalino sostenible y nanomateriales. De esta forma se podría desarrollar un producto terminado que no dependiera de un material como la cal para ofrecer el ambiente básico necesario para el desarrollo de las reacciones puzolánicas. La posible comercialización de un producto sostenible de bajo coste para estabilización de suelos facilitaría su implementación en el mercado de la construcción.

Ampliación del rango de utilización de las soluciones obtenidas para le mejora de suelos a otros campos como en la edificación. La mejora de suelos para cimentaciones de edificación es un mundo poco explorado en la aplicación de materiales sostenibles. Estudios de comportamiento dinámico para los suelos mejorados mediante la adición de nuevos productos estabilizadores sostenibles serían necesarios para garantizar el correcto desempeño estructural del conjunto suelo / estructura.

Estudio de un paquete integral de carreteras aplicando subproductos industriales disponibles en la zona de construcción. En la presente Tesis Doctoral se ha estudio la ejecución de subbases y bases de carreteras aplicando materiales reciclados provenientes de diferentes subproductos. Se plantea estudiar un paquete integral compuesto por subbase de suelo estabilizado o mejorado, base de carretera de zahorra reciclada y pavimento ejecutado con hormigón sostenible aplicando un sustitutivo al cemento fabricado a partir de subproductos industriales. Estudio de comportamiento del conjunto del paquete de carreteras a largo plazo mediante auscultación estática y dinámica.