






Article

Preliminary Study of Recycled Aggregate Mortar for Electric Arc Furnace Dust Encapsulation

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Abstract: This article shows the preliminary results of a study carried out to determine the technical feasibility of encapsulating a high percentage of EAFD in cement-based mortars manufactured with the fine fraction of recycled concrete aggregates (RCA). Two families of mortars, with natural aggregate as a reference and with RCA, were studied. An incorporation rate by weight of two parts mortar to one part EAFD was tested. The mechanical strengths (compressive strength and tensile strength) before and after immersion in water, the rate of delinescence and the leaching behavior were studied. Mortars made with RCA showed similar mechanical strengths to the reference mortars made with natural aggregates; however, the incorporation of EAFD decreased the mechanical strengths. Encapsulation considerably reduced the leaching of heavy metals, although the Pb concentration remained above the hazardous waste limit. With this preliminary study, two wastes are managed together, and the results have shown that the use of RCA instead of natural aggregate is a viable alternative since it does not significantly impair the mechanical or leaching properties of the cement-based matrices used to encapsulate EAFD.

Keywords: Electric Arc Furnace Dust; recycled concrete aggregates; mechanical behavior mortar; leaching behavior mortar; encapsulation hazardous waste



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1. Introduction

The “Europe 2020” strategy aims to generate smart, sustainable and inclusive growth by opting for an efficient economy in the use of natural resources. The new paradigm of the “circular economy” has as its main objective the sustainable use of resources through the principle of “closing the lifecycle loop” of materials—that is, to reintroduce into the production process materials classified as “waste” whose destination was the landfill [1]. This practice reduces the volume of waste managed in landfills and reduces the amount of raw materials consumed [2].

One of the main focuses of action of the “circular economy” is the construction sector, since it is considered one of the main generators of waste and consumers of natural resources at the European level [3,4]. Within the construction sector, the mortar and concrete industry has a high impact due to its high consumption of non-renewable natural resources [5]. Construction and demolition waste (CDW) is one of the major waste streams globally; therefore, the use of recycled aggregates from CDW as raw materials in mortars and concretes is a practice to be promoted and is in line with the new paradigm of the “circular economy”.

The coarse fraction of recycled aggregates from CDW has been used successfully in the manufacture of structural and non-structural concrete [3,6,7]. The fine fraction of

these recycled aggregates (0/4 mm) has also been used successfully in the manufacture of masonry mortars [8–12] and structural concrete [13,14].

Furthermore, Spain is the third-highest steel producing country in the European Union [15]. More than 75% of its steel is produced through scrap recycling [16]. The scrap is melted in an electric arc furnace at temperatures above 1600 °C, and in this process occurs the volatilization of elements such as iron, zinc and lead, which react with oxygen and generate waste in the form of dust. This waste is known as Electric Arc Furnace Dust (EAFD), and it is collected and recovered in ventilation filters [17]. Although the composition of the EAFD depends on the scrap melted in the electric arc furnace, it is mainly composed of heavy metals such as zinc and lead [18].

From an environmental point of view, the EU Council Decision 2003/33/EC [19] establishes the criteria for classifying waste according to the concentration of heavy metals and anions detected in accordance with a conformity test (UNE EN 12457-4:2003). The levels of classification of waste based on its deposit in a landfill are inert, non-hazardous and hazardous. EAFDs are classified according to the EU Council Decision 2003/33/EC [19] as hazardous waste due to the high content of heavy metals that can leach [20–22]. Worldwide, around 70% of the EAFD produced is destined for landfilling after an encapsulation treatment, while the remaining 30% is destined for metal recovery [23–25].

Occasionally, scrap metal contains radioactive elements that may not be detected when entering the steel mill and then contaminate the steel mill dust during the manufacturing process [26]. Several radioactive incidents have been described in EAF steel mills in Italy, Mexico, Brazil, Thailand and Estonia [27]. In Spain, during the last decade, six incidents of this nature occurred in Andalusia (south of Spain) and the Basque Country (north of Spain). Because of this type of incident, it is necessary to encapsulate the EAFD with cement-based materials prior to transporting and subsequent depositing in storage installations for radioactive waste. The maximization of the ratio of EAFD/mortar is crucial to reduce the volume of storage necessary at this type of installation.

EAFD encapsulation techniques aim to create mechanically and chemically stable monolithic blocks. The use of conventional Portland cement in the encapsulation of EAFD is usually the most recommended technique due to the extensive knowledge of this material, its availability and its good long-term physical and chemical stability [28]. The verification of the adequate encapsulation of the EAFD by means of cement-based matrices must be carried out through the study of the mechanical properties of the monoliths formed by the setting of the cement and the leaching behavior of elements that are harmful to the environment [16]. There are successful studies in which cement-based matrices are used to encapsulate EAFD [29,30]. Even so, the immobilization of heavy metals from EAFD is not always chemically possible (adsorption) [31], it being necessary to develop matrices that allow the encapsulation of heavy metals from a physical point of view—for example, using denser matrices that do not allow the diffusion of contaminating elements [1,32]. The cement-based matrices used to date for the encapsulation of EAFD incorporate natural sand [31], and there are no studies on the feasibility of using recycled aggregates from CDW for the manufacture of encapsulation matrices. However, mortars made with recycled aggregates from CDW are more porous than those made with natural aggregates, which can impair the diffusion leaching phenomena of monolithic mortar blocks, hence the need to study the feasibility of using recycled aggregates from CDW in the manufacture of cement-based matrices for the immobilization of EAFD.

The objective of this study is to analyze the possibility of using the fine fraction of recycled concrete aggregates (RCA) for the encapsulation of EAFD in cement-based matrices. If the results of this research are favorable, two wastes can be reused together, reducing the consumption of natural resources and giving a second life to RCA- and EAFD-type waste, while promoting the new paradigm of the circular economy.

2. Materials and Methods

2.1. Characterization of Materials

A commercial siliceous sand (Natural aggregate: NA) was used as a reference, and a fine recycled aggregate from structural concrete waste (RCA) was used as an alternative. The RCA was collected from the 0/4 mm stockpile of a recycling plant located in Córdoba, Spain, where structural concrete waste from different sources was previously crushed and subsequently screened. Figure 1 shows the particle size distribution of NA and RCA calculated according to the standard UNE-EN 933-1:2006. In RCA, approximately 93% of the particles are less than 4 mm, while in NA, 100% are less than 4 mm. In RCA, more than 14% of the particles pass through the 0.063 mm sieve, and in NA, only 1.8% pass through this sieve. The RCA shows a more continuous particle size distribution, with higher compaction capacity than the NA, which presents a more uniform particle size distribution. In order that the results of this research can be applied on a real scale, the aggregates were used without altering their granulometric curves.

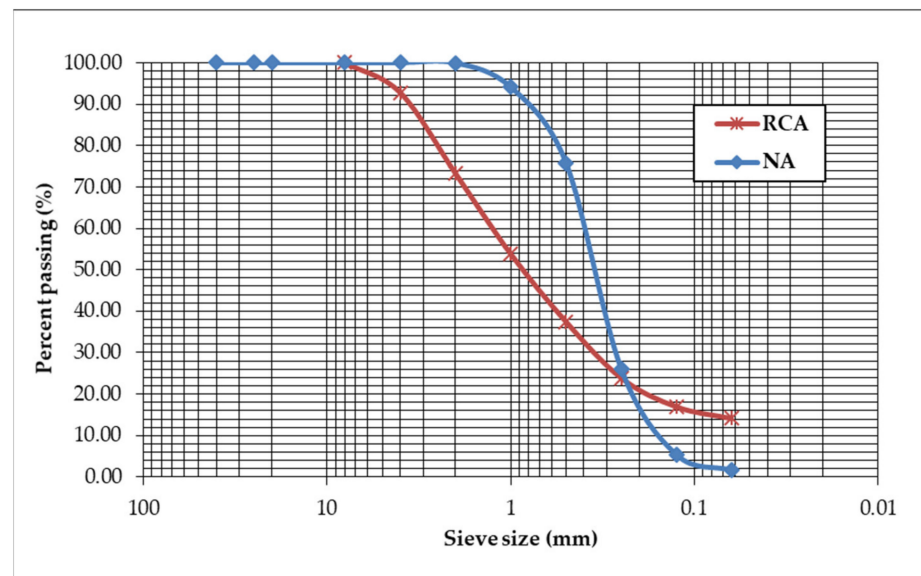


Table 1. Material chemical composition.

Component (% Mass)	SF	Cement	NA	RCA
SiO ₂	100.00	14.08	91.93	52.38
Al ₂ O ₃ %	–	3.20	3.23	7.92
Fe ₂ O ₃ %	–	4.56	1.07	2.81
SO ₃ %	–	4.23	–	–
CaO%	–	71.98	0.71	31.2
K ₂ O	–	0.96	3.06	1.82
MgO%	–	0.99	–	2.82
Na ₂ O	–	–	–	0.83
Cr ₂ O ₃	–	–	–	0.22

The EAFD was collected from an electric arc furnace steel mill located in the North of Spain (Zumárraga).

The specific surface area of EAFD, SF and cement was analyzed by the Brunauer–Emmett–Teller (BET) method, through the absorption of N₂ with Micromeritics ASAP 2010 equipment. The dry particle density was also determined according to standard UNE 80103:2013 (Standards used in the experimental work are shown in Appendix A). The results obtained are shown in Table 2.

Table 2. Physical properties of EAFD, SF and Cement.

	EAFD	SF	Cement
Specific surface area (m ² /g)	4.6	0.25	0.35
Dry particle density (g/cm ³)	3.81	2.60	3.14

The chemical composition of EAFD was determined using the EDAX technique using a Jeol scanning electron microscope with the following characteristics: model JSM-6300 with acceleration potential of 20 kV and working distance of 15 mm. The percentages by weight of the elements identified in the EAFD are indicated in Table 3.

Table 3. Chemical composition of EAFD.

Element (%)	EAFD
Zn	35.45
Fe	23.53
O	18.32
Ca	7.58
Cl	3.58
Pb	3.02
Mn	2.14
K	1.63
S	1.22
Mg	1.17
Si	1.05
Al	0.53
Ti	0.52
Cr	0.26
Total	100.00

The metallic elements that showed a higher proportion in the EAFD were Zn and Fe, and to a lesser extent Ca, Pb and Mn. These results were in accordance with those reported by other authors such as López and López-Delgado [33] and Sapiña et al. [34]. The composition of this type of waste varies depending on the quality of the molten scrap. Sofilic et al. [35] also identified Fe as the main element, and to a lesser extent Zn, Ca and Mn.

To evaluate the potential contaminating effect of the EAFD, a compliance test was carried out in accordance with the UNE EN 12457-4:2003 standard. This test is used to determine the concentration of heavy metals and anions in the leachate of a waste and allows classifying the waste in accordance with the criteria established by the EU Council Decision 2003/33/EC [19].

The compliance test procedure is described here: the dry mass to be tested was 0.90 kg. A quantity of leachate (deionized water) was added so that a liquid/solid ratio (L/S) of 10 L/kg was established. The mixture was stirred in a tumbler for 24 h at a speed between 5 and 10 revolutions per minute, and then the sample was filtered with 0.45 µm filters.

The liquids from the filtration were analyzed in an ICP-MS (Perkin Elmer ELAN DRC-e) equipped with a system for introducing diluted samples, argon plasma ionization and quadruple ion detection, which has a DRC cell to eliminate interferences. This analysis quantified the 12 heavy metals specified by the European Landfill Directive: Ni, Cr, Sb, Se, Mn, Hg, As, Pb, Cd, Cu, Ba and Zn. In addition, the sulphate, fluoride and chloride anion contents were obtained by ion chromatography according to the UNE-EN ISO 10304-1:2009 standard.

Table 4 shows the results obtained in the leaching test. Limit values for the environmental classification of waste according to the EU Council Decision 2003/33/EC [19] are also indicated.

Table 4. Leached concentrations of EAFD (mg/kg) and acceptance criteria (WAC, EU Council Decision 2003/33/EC).

Element (mg/kg) L/S = 10	EAFD	Criteria EU Landfill Directive 2003/33/EC		
		Inert	Non-Hazardous	Hazardous
Cr (Chromium)	1.970 ^(a)	0.5	10	70
Ni (Nickel)	0.053	0.4	10	40
Cu (Copper)	2.157 ^(a)	2	50	100
Zn (Zinc)	24.047 ^(a)	4	50	200
As (Arsenic)	<0.05	0.5	2	25
Se (Selenium)	2.762 ^(b)	0.1	0.5	7
Mo (Molybdenum)	20.494 ^(b)	0.5	10	30
Cd (Cadmium)	0.138 ^(a)	0.04	1	5
Sb (Antimony)	0.001	0.06	0.7	5
Ba (Barium)	6.935	20	100	300
Hg (Mercury)	0.180 ^(a)	0.01	0.2	2
Pb (Lead)	5483.866 ^(c)	0.5	10	50
Fluoride	65.8 ^(a)	10	150	500
Chloride	24,100 ^(b)	800	15,000	25,000
Sulphate	16,300 ^(a)	1000	20,000	50,000
Conditions of the Test Sample				
Conductivity (µS/cm)	8560			
Temperature (°C)	19.8			
pH	13.28			

^(a) Exceeds the inert waste limit; ^(b) Exceeds the non-hazardous waste limit; ^(c) Exceeds the hazardous waste limit.

In Table 4, it is necessary to highlight the high leaching of Pb in the EAFD sample, since with a pH higher than 12, the Pb is easily mobilized [36–39]. The EAFD showed a pH value of 13.28 during the UNE-EN 12457-4:2003 compliance test. The pH value of the EAFD depends on the raw material and the additives used in the production of steel; therefore, the pH values of EAFD are highly variable.

Leclerc et al. [40] found slightly lower pH values in five different types of EAFD: 8.2, 9.3, 10.4, 11.0 and 11.4. Ledesma et al. [16] studied the influence of pH on the leaching of metals from two EAFD. The pH dependence test confirmed minimal Pb release for pH values of 9–11, although when the pH was increased to values higher than 12 the concentration of Pb in the leachate increased significantly.

2.2. Experimental Design

Two families of mortars were tested using NA as a reference aggregate and RCA as an alternative aggregate. The reference mortar was dosed by weight with the following percentages of its components: 30% cement, 40% aggregates (natural or recycled depending on the family in each case) and 30% siliceous filler.

EAFD was incorporated into the two families of mortars in a mortar/EAFD weight ratio of 2:1. A previous study carried out by Ledesma et al. [31] was taken as a reference to choose the amount of waste to be incorporated into the mix, which determined the maximum amount of EAFD that could be mechanically encapsulated with cement-based mortars and limestone aggregates. The amount of water was added experimentally to achieve a liquid consistency of the mortar equal to 230 ± 10 mm on a shaking table (UNE-EN 1015-3:2000).

Table 5 shows the dosages used in each batch, as well as the nomenclature adopted for the different types of mortars tested.

Table 5. Mortar mix proportions.

Mortar Type	CEM (g)	NA (g)	RCA (g)	SF (g)	EAFD (g)	Water (g)	w/c	Consistency (mm)
NA-REF	1200	1600	-	1200	-	1050	0.88	236
NA-EAFD/2:1	800	1067	-	800	1333	1194	1.49	224
RCA-REF	1200	-	1600	1200	-	1233	1.03	234
RCA-EAFD/2:1	800	-	1067	800	1333	1628	2.04	236

2.3. Methodology

To evaluate the mechanical properties of the manufactured mortars, the compressive strength and tensile strength were measured before and after immersion, following the procedure described in the XP X31-212:2011 standard.

Cylindrical specimens of 80mm in height and 40 mm in diameter were used, following the methodology proposed by Ledesma et al. [31]. The mechanical strength data before immersion were determined after 28 days of mixing, and those corresponding to after immersion at 32 days, after the specimens had been immersed in deionized water for 96 ± 4 h at a temperature of 20 ± 5 °C, with L/S ratio = 10, as described in the XP X31-212:2011 standard.

The water in contact with the submerged specimens was passed through a $0.45 \mu\text{m}$ filter to determine the delitescence rate (XP X31-212:2011). The material retained on the filter was dried in an oven at a temperature of 105 ± 5 °C. The delitescence rate was obtained by the following expression (1):

$$t_d = 100 \times \frac{m_d}{m_{0s}} \quad (1)$$

where:

t_d : delitescence rate (%)

m_d : dry mass of solid particles retained on the $0.45 \mu\text{m}$ filter (g)

m_{0s} : dry mass of the specimen (g)

A total of 3 repetitions were carried out for the tests of compressive and tensile strength before and after immersion. To determine the delitescence rate, 3 of the 6 specimens that were submerged in deionized water were randomly taken, after which the eluate was filtered and the delitescence rate was calculated. Once the specimens were broken in the compressive strength test, before and after immersion, their dry bulk density was calculated according to the UNE-EN 1015-10:2000 standard.

To evaluate the concentration of heavy metals and anions that can leach from the encapsulated EAFD in the cement-based matrices, the method described in the XP X31-211:2012 standard was followed.

Two cylindrical samples of each type of mortar were immersed for 24 h in deionized water ($L/S = 10$) in continuous movement by means of a magnetic stirrer. The water in contact with the samples was passed through 0.45 μm filters, and the eluate from the filtering process was analyzed on an ICP-MS following the procedure described in the previous section. This method has been used in a previous study [31].

3. Results

3.1. Dry Bulk Density of Hardened Mortar

The dry bulk density of the hardened mortar was obtained after 28 days, and the results obtained are shown in Table 6.

Table 6. Dry bulk density of the hardened mortar: mean values and standard deviation.

Mortar	Dry Bulk Density (kg/m^3)
NA-REF	1563 \pm 0.006
NA-EAFD/2:1	1413 \pm 0.008
RCA-REF	1537 \pm 0.007
RCA-EAFD/2:1	1335 \pm 0.003

The dry bulk density decreased slightly when NA was replaced by RCA, attributed to the lower density of the RCA particles and the higher w/c ratio used [9]. The lower density of RCA was compensated by its more continuous particle size distributions, which gave the mortar specimens higher compactness [9,12].

The dry bulk density decreased when the EAFD content increased, attributed to the higher w/c ratio. The incorporation of EAFD causes a higher porosity of cement-based mortars [32] due to the interaction of the heavy metals contained in the EAFD with the cement hydration products, which also explains the lower dry bulk density of this type of EAFD mortar.

3.2. Mechanical Properties of Hardened Mortar

Figure 2 shows the results of compressive strength, tensile strength and rate of delitescence tests. The specifications that were taken into account to consider the mortar specimen a monolithic block were the same as previously considered by Ledesma et al. [31]: at 28 days of age, the compressive strength must be greater than 1 MPa, the tensile strength greater than 0.1 MPa, and the delitescence rate after immersion less than 10%. Lampris et al. [41] and Fernández et al. [42] also considered that a mortar reaches the monolithic block state when the compressive strength at 28 days is higher than 1 MPa, which is in accordance with what is indicated by the English Environmental Agency (EEA) [43].

All the analyzed samples showed a delitescence rate below the 10% established as the limit. A slight increase in the delitescence rate was observed with the incorporation of EAFD in the mix and with the use of RCA, which may be related to the lower dry bulk density and higher porosity of these specimens.

The loss of mechanical strength in cement-based materials with the incorporation of EAFD is common, mainly due to the high presence of heavy metals in the EAFD that affect the microstructure and hydration reactions of the cement [16,44].

Ledesma et al. [31] and Lozano-Lunar et al. [2] also found a notable decrease in the mechanical strengths of conventional and self-compacting mortars with the incorporation of EAFD. In this case, the decrease in compressive and tensile strength was higher than 50% in most of the mixtures. However, the compressive strengths obtained with EAFD were above 7 MPa and 0.5 MPa in tensile strength, which allowed classifying the mortar as a monolith block.

The substitution of natural sand for RCA showed good results in the manufacture of mortars, reaching replacement rates of up to 40% without being significantly affected by mechanical strength [9]. In the present study, a similar behavior of mortar with EAFD

was observed with NA and RCA, which contributes positively to jointly treat a hazardous waste (EAFD) and a recycled aggregate from CDW.

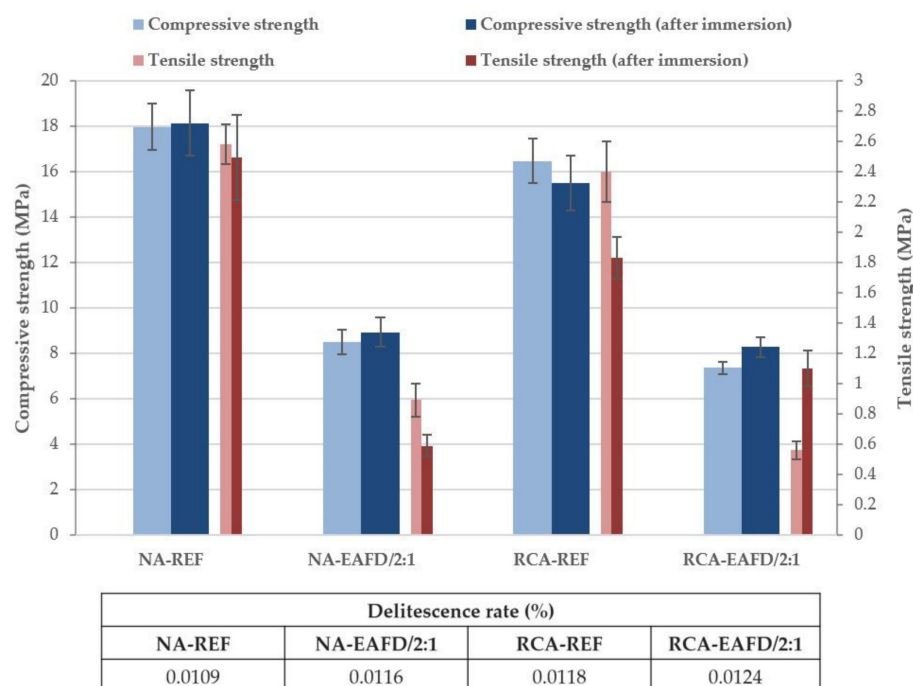


Figure 2. Results (mean values and standard deviation) of the mechanical properties.

3.3. Environmental Risk from Heavy Metal Leaching

Table 7 shows the release levels of elements obtained in the tank leaching test XP X31-211:2012 of the hardened mortars. The conditions during the test are also indicated conductivity, temperature and pH.

Table 7. Leaching behavior of monolithic samples of mortar (XP X31-211:2012).

Element (mg/kg) L/S = 10	NA-REF	NA-EAFD/2:1	RCA-REF	RCA-EAFD/2:1
Cr	0.013	0.029	0.110	0.131
Ni	0.010	0.009	0.023	0.022
Cu	0.003	0.012	0.013	0.029
Zn	0.070	7.630 ^(a)	0.167	10.035 ^(a)
As	<0.05	<0.05	0.012	<0.05
Se	0.003	<0.1	0.027	0.353 ^(a)
Mo	0.022	<0.5	0.020	<0.5
Cd	0.000	0.001	0.001	0.003
Sb	0.001	0.003	0.010	0.016
Ba	2.010	2.829	2.240	5.284
Hg	0.001	0.001	0.000	0.004
Pb	0.023	14.745 ^(b)	0.073	35.993 ^(b)
Fluoride	<10	<10	<10	<10
Chloride	7.463	3250 ^(a)	<10	4072.700 ^(a)
Sulphate	19.790	754.525	<50	105.650
Conditions of the Test Sample				
C (μS/cm)	711	2865	1284	2850
T ^a	20.7	19.2	30.5	28.9
pH	11.5	11.67	11.74	11.96

^(a) Exceeds the inert waste limit; ^(b) Exceeds the non-hazardous waste limit.

All elements were kept below the inert limit in the reference mortar of each family: NA-REF and RCA-REF. In the case of RCA-REF, this is of special interest since it is an inert waste.

Mortars made with EAFD, NA-EAFD/2:1 and RCA-EAFD/2:1, show similar element release results, which shows that the substitution of NA for RCA does not significantly impair leaching. In the case of NA-EAFD/2:1, Zn and chlorides exceeded the inert waste limit; however, Pb exceeded the limit of 10 mg/kg (14,745 mg/kg) established to classify the material as hazardous. In the RCA-EAFD mortar, in addition to Zn and chlorides, the inert limit was exceeded. Pb also exceeded the limit of 10 mg/kg (35,993 mg/kg). To reduce the amount of Pb in the leachate, further studies with less EAFD should be carried out until the optimal ratio of EAFD:mortar is achieved.

The leaching of Pb in the starting EAFD (Table 3) was 5483.866 mg/kg, an amount that exceeds the limits of the EU Council Decisión 2003/33/EC [19] for its deposit in landfill. With the encapsulation of the EAFD in cement-based mortars, using both NA and RCA it is possible to reduce this amount by 99.73% and 99.34%, keeping the release of Pb within the limits of the EU Council Decisión 2003/33/EC [19]. With the incorporation of EAFD, the resulting matrix is more porous, which favors the mobility of Pb [16,45]. The higher conductivity obtained in mortars with EAFD is in accordance with the higher presence of metals in the leachate.

4. Conclusions

In this preliminary study, the possibility of using recycled concrete aggregates (RCA) to encapsulate Electric Arc Furnace Dust (EAFD) in cement-based matrices was investigated. The following partial conclusions were drawn:

- (1) The EAFD showed a release of Pb that exceeds the limit for its classification as hazardous waste, favored by the high pH obtained in the leaching test.
- (2) The total substitution of NA for RCA reduced the dry bulk density of the hardened mortar and slightly decreased the compressive strength (8%) and the tensile strength (7%).
- (3) The incorporation of EAFD in a 2:1 weight ratio (EAFD: mortar) reduced the mechanical strength by around 50% both in mortars made with NA and with RCA, which may be due to the high presence of heavy metals in the EAFD and the highest w/c ratio necessary for its manufacture.
- (4) The weight ratio (EAFD: mortar) 2:1 used for the encapsulation of EAFD did not enable immobilizing the Pb below the hazardous waste limits, either in the mortars made with NA or in the mortars made with EAFD, which will require future studies to optimize the EAFD: mortar ratio.

In conclusion, the use of RCA instead of NA for the manufacture of encapsulation mortars for hazardous waste is a viable alternative since it does not significantly impair the mechanical or leaching properties of cement-based matrices. Pb has been identified as the most limiting element to encapsulate EAFD, with new studies being necessary to optimize the EAFD: mortar ratio and the leaching of Pb.

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Appendix A

Standards Used in the Experimental Work

UNE-80103:2013. Test methods of cements. Physical analysis. Actual density determination.

UNE-EN 1015-10:1999. Methods of test for mortar for masonry. Part 10: Determination of dry bulk density of hardened mortar.

UNE-EN 1097-6:2014. Test for mechanical and physical properties of aggregates. Part 6: Determination of particle density and water absorption.

UNE-EN 12457-4:2003. Characterization of waste. Leaching. Compliance test for leaching of granular waste materials and sludges. Part 4: One stage batch test at a liquid to solid ratio of 10 L/kg for materials with particle size below 10 mm (without or with size reduction).

UNE-EN 933-1:2006. Test for geometrical properties of aggregates Part 1: Determination of particle size distribution. Sieving method.

UNE-EN 933-11:2009. Test for geometrical properties of aggregates. Part 11: Classification test for the constituents of coarse recycled aggregate.

UNE-EN ISO 10304-1: 2009. Water quality—Determination of dissolved anions by liquid chromatography of ions—Part 1: Determination of bromide, chloride, fluoride, nitrate, nitrite, phosphate and sulphate (ISO 10304-1:2007).

XP X31-211:2012. Waste—test for the determination of the leachability of a solid waste material initially massive or generated by a solidification process.

XP X31-212:2011. Characterization of waste—determination of the massive solid characteristic.

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