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Valorization of the Orange Tree Pruning by Ethanol Process

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Abstract
Spain is the country with the largest citrus production in Europe, with about 5 million tons per year (2007), 3.5 million tons corresponding to oranges. Worldwide, only Brazil, China and the US have a higher annual production, with 21, 19, and 10 million tons, respectively. This pruned fraction of orange tree is an important lignocellulosic resource and consists of leaves, bark and young branches. The most obvious application is in combustion processes, but it is also feasible to use in conversion processes to products with high added value, including the production of cellulose pulp by alternative methods. The aim of this work is the systematic study of valorisation of orange pruning by applying an organosolv method (ethanol) for the production of cellulose pulps by assessing the effect of the most influential treatment variables on the characteristics of the pulps by means of factorial designs and statistical analysis of the results. The values estimated by the polynomial models reproduce the experimental results of the different dependent variables, with errors less than 22%, while by the neurofuzzy models the error are less than 20%. Although neurofuzzy models use a greater number of parameters than second order polynomial models, both types of models are equally effective for a typical experimental design of three variables of operation, with a total of 15 experiments.

1. Introduction

In addition to other treatments for lignocellulosic materials used to produce pulp, in recent decades organosolv processes have attracted significant interest. Both for legal reasons such as social pressure, the implementation of production processes pulp tends increasingly towards production methods that minimize environmental impact. At the same time, global demand for cellulosic products has increased significantly. Therefore, research on new methods and materials becomes urgent, in some scenarios (Muurinen, 2000).

In the scientific literature there is a wide variety of processes applicable to plant materials to leverage its components that try to avoid the environmental problems associated with current methods, particularly those associated with the Kraft process to be the most widely established throughout the world (Jiménez and Rodríguez, 2010). Within these methods are organosolv, producing the delignification of the raw material with the aid of organic solvents, often in softer conditions than the Kraft process itself, and that, moreover, do not use sulphur compounds in cooking with subsequent environmental benefits.

Spain is the country with the largest citrus production in Europe, with about 5 million tons per year (2007), 3.5 million tons corresponding to oranges. Worldwide, only Brazil, China and the US have a higher annual production, with 21, 19, and 10 million tons, respectively (FAOSTAT, 2012). For proper cultivation of orange trees and improving production, a periodic pruning must be conducted. Pruning is an operation which modifies the shape and natural development of the tree, having an immediate effect and also impacts in the medium and long term. Pruning process is related to the tree feeding. So, with fertile soil, fertilization is more balanced and the tree can support more number of branches, produces higher yields, and require subsequent lighter pruning. Apart from controlling the development and shape of the tree, pruning provides improved fruit quality by eliminating the remaining branches (unproductive), and increasing the insulation and ventilation in inland areas, where the fruits have high quality.

This pruned fraction of orange tree is an important lignocellulosic resource and consists of leaves, bark and young branches. The most obvious application is in combustion processes, but it is also feasible to use in conversion processes to products with high added value, including the production of cellulose pulp by alternative methods.

The aim of this work is the systematic study of valorisation of orange pruning by applying an organosolv method (ethanol) for the production of cellulose pulps by assessing the effect of the most influential treatment variables on the characteristics of the pulps by means of factorial designs and statistical analysis of the results.

2. Experimental

2.1. Raw Material, Analysis and Characterization

Orange tree pruning, specifically of the variety of *Citrus sinensis*, from Palma del Rio, Córdoba (Spain) were used as raw material. Since pruning is constituted of leaves and branches or different size, and in order to achieve a more homogeneous material, only stalks with a diameter larger than 1 cm were used in this work. Following drying at room temperature, the raw material was cold ground in a Retsch SM 2000 mill to avoid alterations in its components and stored in plastic bags before cooking processes.

The orange tree prunings were characterized as follows: holocellulose α-cellulose, lignin, ash and ethanol-benzene extractives according to TAPPI standards, namely T-9m54, T-203 os-61, T-222, T-211 and T-204, respectively.

2.2. Pulping

All the cookings experiments were performed in a 1.9 L cylindrical reactor operating in batch mode. Heating was provided by a system of electrical resistances surrounding the reactor. The reactor vessel is designed to achieve pressures higher than 30 kg/cm^2. The reactor is equipped with instruments for accurate measurement of pressure and temperature

A typical cooking experiment used 50 g (o.d.m.) of raw material at a liquid/solid ratio of 8:1. Pulps were beaten in a Sprout-Bauer refiner operating at 0.5% pulp consistency, using a disk spacing of 0.1 mm, and the fiberized material passing through a Sommerville screen model K134 to remove uncooked particles, while water was eliminated through centrifugation.

2.3. Pulp Characterization

The viscosity of the pulps was determined according to TAPPI T-230-om-94. Pulp yield was determined by weighing, and moisture determination, after removing the uncooked material.

2.4. Paper Sheets Formation and Characterization

Paper sheets with a grammage of 60 g/m^2 were obtained by using an Enjo-F39.71 (Metrotec) paper-sheet former according to TAPPI standard T-205 ps-95. The tensile index, burst index and tear index of paper sheets were determined according to TAPPI standards: T-494 om-96, T-403 om-97, T-414 om-98, respectively.

2.5. Experimental Design, Polynomial and Neural Fuzzy Model

A face centred factorial design was built and the corresponding cooking experiments performed. The design used consisted of a series of points (tests) around a central composition point (central test) and several additional points (additional tests) that were used to estimate the quadratic terms of a polynomial model and the constants or parameters of neural fuzzy model. The design met the general requirement that it allowed all parameters in the mathematical model to be estimated with a relatively small number of tests, (Montgomery, 1991). According to previous experimentation the ranges of variation of the independent variables were chosen as: temperature (T), 170-200°C; cooking time (t), 60-120 min; ethanol concentration in liquor (E), 60-80%.

Three parameters defined the experimental design used: k (number of variables); constant p (0 for k< 5 and 1 for k≥5); and number of central points, n_c. These parameters originate three sets of points:

- 2^{k-p} points constituting a factorial design
- 2*k axials points
- n_c central points

The total number of points (experiments) shall be given by the following expression:

$$n = 2^{k-p} + 2 * k + n_c \quad [1]$$

The total number of tests required for the three independent variables studied [*viz.* temperature (T), time (t) and ethanol concentration (E), were found to be 15.

The values of the independent variables were normalized to -1, 0 or +1 by using equation [2] in order to facilitate direct comparison of coefficients and expose the individual effects of the independent variables on each dependent variable:

$$X_n = 2\frac{X - \bar{X}}{X_{max} - X_{min}} \quad [2]$$

where X_n is the normalized value of T, t or E.

Experimental data were fitted to the following second-order polynomial

$$Y = a_0 + \sum_{i=1}^{n} a_i X_{ni} + \sum_{i=1}^{n} a_j X_{ni}^2 + \sum_{i=1, j=1}^{n} a_l X_{ni} X_{nj} \quad i<j \quad [3]$$

being *Y* a characteristic or property of the pulp (viscosity or yield) or paper sheets (tensile index, burst index or tear index), and coefficients a_0, a_i, a_j and a_l are unknown characteristic constants estimated from the experimental data.

Also, the relationships between the dependent variables (*viz.* pulp yield, viscosity, tensile index, burst index and tear index) and the independent (operational) variables were established by using a fuzzy neural model. This type of model combines the advantages of fuzzy logic systems (Zadeh, 1965) and neural networks (Works, 1989), and provides a powerful prediction tool based on the following equation (Jang, 1993) with three independent variables, the use of a singleton defuzzifier (a constant parameter) and a linear membership function for the independent variables:

$$Y_e = \frac{\sum_{l=1}^{8} c_l \left[\prod_{i=1}^{4} x_{ij} \right]_{j=1}^{2}}{\sum_{l=1}^{8} \left[\prod_{i=1}^{4} x_{ij} \right]_{j=1}^{2}} \quad [4]$$

where Y_e is the estimated value of the property to be modelled and c_l the defuzzifier of a fuzzy rule, x_i denoting the values of temperature (T), time (t) and ethanol concentration (E), and j being 1 or 2 in the equations:

$$x_{i1} = 1 - \frac{1}{(x_{high} - x_{low})}(x - x_{low})$$

$$x_{i2} = \frac{1}{(x_{high} - x_{low})}(x - x_{low}) \quad [5]$$

with x_{high} and x_{low} the extreme values of each variable.

With three independent variables, it is possible to establish the following 8 fuzzy rules (R_i) based on the extreme (high and low) values of such variables:

R_1: low T, low t and low E; R_2: low T, low t and high E ....R_7: high T, high t and low E; R_8: high T, high t and high E

With a Gaussian membership function with three levels (low, medium and high) for one of the variables and a linear membership function with two levels (low and high) for the other two, Eq. 4 would contain 12 terms in the numerator and another 12 in the denominator. The Gaussian membership function would be of the form:

$$x_i = \exp\left(-0.5\left(\frac{x - x_c}{L} \right)^2 \right) \quad [6]$$

being x the absolute value of the variable concerned; x_c its minimum, central or maximum value; and L the width of its Gaussian distribution.

The parameters and constants in the previous equation were estimated by using the ANFIS© (Adaptive Neural Fuzzy Inference System) Edit tool in the Matlab v. 6.5 software suite

3. Results and Discussion

3.1. Polynomial Model

By fitting the experimental data (table 1) to a second degree polynomial equation (Rodríguez *et al.*, 2011), the following equations were obtained:

$$YI = 45.55 + 1.87X_T^2 - 1.76X_t + 3.40X_E - 8.86X_T \quad [7]$$

$$VI = 434 + 50X_T^2 - 39X_E X_T + 64X_t - 74X_E + 84X_T X_t + 241X_T \quad [8]$$

$$TI = 16.83 - 0.75X_E + 1.00X_E X_T - 1.18X_E X_t - 1.31X_T X_t + 2.56X_T - 6.18X_T^2 \quad [9]$$

$$BI = 0.711 - 0.050X_T X_t + 0.060X_E X_T - 0.058X_E X_t + 0.103X_T - 0.271X_T^2 \quad [10]$$

$$TeI = 1.509 + 0.062X_t + 0.094X_E X_T - 0.116X_T X_t + 0.137X_T - 0.359X_T^2 \quad [11]$$

being; YI: pulp yield, VI: viscosity, TI: tensile index, BI: burst index, TeI: tear index, and X_T, X_t, X_E are the normalized values of temperature, time and ethanol concentration, respectively.

Table 2 shows the statistical parameters of the polynomial equations; it can be seen that the polynomial models provide a good fit to the experimental data. In addition, the values estimated by the previous equations reproduce the experimental results of the different dependent variables, with errors less than 5% (Yield), 10% (Viscosity), 12%

(Tensile index), 22% (Burst index) and 13% (Tear index).

Table 3 shows the optimal values of the dependent variables and the corresponding values of the operational variables, found using the More and Toraldo method (1989).

Analyzing equations 7 to 11 and the response surfaces when representing two independent variables keeping the other at its optimal value, it can be deduced which are the operational variables of greater and lesser influence on the characteristics of the pulps and paper-sheets.

Thus, the maximum variations values calculated for the yield, viscosity, tensile index, burst index and tear index, are presented in table 4. Also this table shows the maximum variations mentioned above expressed as percentage of deviation from their optimal values.

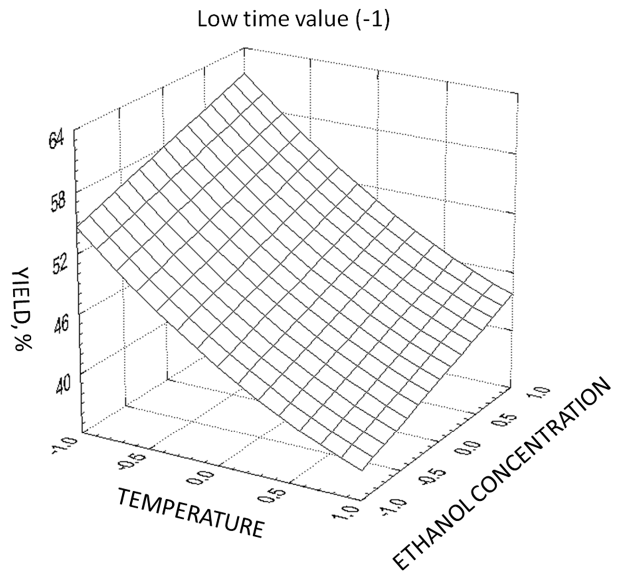

Figure 1. Variation of the yield depending on the temperature and the ethanol concentration, for a short time value.

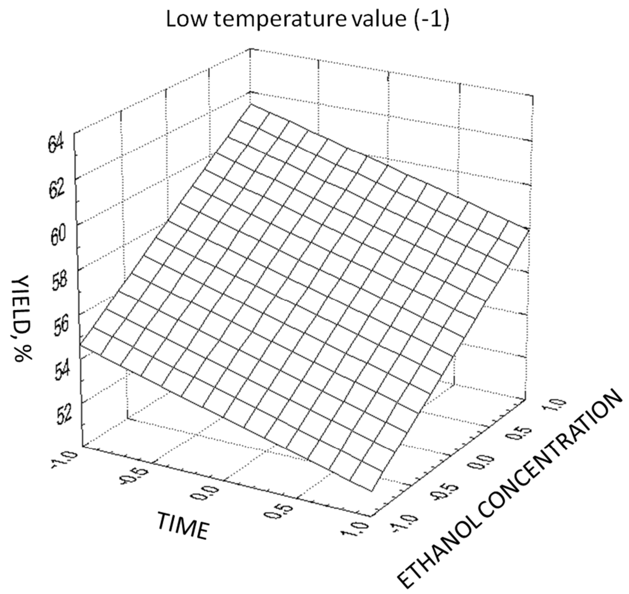

Figure 2. Variation of the yield depending on the ethanol concentration and of the time, for a low temperatura value

It follows from the data of table 4 and figures 1 and 2, that for the yield of the pulp, the most influential variable is the temperature and the less influential the pulping time. The figures also suggest that maximum yield is achieved when operating with low values of temperature and time and high ethanol concentration. By contrast, when operating in opposite conditions the yield is minimum.

Doing the same with the other dependent variables is possible to find that the most influential variable on the viscosity is the temperature and the less influential the ethanol concentration. For the tensile index the most influential variable is temperature and the less influential the process time. For the burst index the less influential variables are the time and the ethanol concentration. For the tear index the temperature is the most influential variable, while the ethanol concentration is the one that least affects.

3.2. Neural Fuzzy Model

The experimental data obtained (table 1) were adjusted to the equation of neurofuzzy model to estimate the parameters or constants (a_i) of this equation, with linear functions belonging to two different levels (high and low) for two variables of operation, and a Gaussian membership function at three levels (high, medium and low) for the other operational variable (tables 5 and 6).

Tables 7 and 8 show the estimated values of the dependent variables provided by the neurofuzzy models and the corresponding errors with respect to the experimental values (table 1). As shown, the predictions for the yield, viscosity, tensile index, burst index and tear index differ in less than 5, 8, 13, 8 and 20%, respectively, of their respective experimental values.

Neurofuzzy models allow to find the influence of each operational variable on the dependent properties. This can be illustrated easily with the results of the pulp yield. The values of parameters of the model for the estimation of this property are shown in table 5.

Through the application of the rules 1 and 2, table 5, it is revealed that, with low levels of the operational variables (rule 1), by increasing the ethanol concentration (rule 2), increases yield (from 57.1% to 63.1%). In addition, through comparison of rules 1 and 3 reveals that low values of the temperature and the ethanol concentration, an increase in processing time causes a decrease in the yield of the pulp, from 57.1% to the 49.6%. Finally, raise the temperature (rules 1 and 9) decreases the yield of the pulp from 57.1% to 36.1%. Based on the foregoing, the temperature is the most influential independent variable and the ethanol concentration the less influential.

In practice, can be considered two rules with identical levels of two operational variables and different values for the third operational variable, to find the influence of this last variable on the dependent variables considered.

Neurofuzzy and polynomial models were validated using the values achieved in two additional pulping experiments (columns 1 and 2 of table 9). This table also shows the values of the corresponding properties of the pulps and paper-sheets calculated using polynomial models (columns 3 and 4), and the proposed neurofuzzy (columns 5 and 6), as well as errors of predictions. These errors are similar to those found in the estimates for the various experiments of the considered experimental design (table 1), which confirm the validity of both models.

Table 1. *Values of the operational variables used in the experimental design used in ethanol pulping of the orange tree prunings, and values of the pulps and paper-sheets properties.*

Exp.	T,ºC	t,min	E,%	YI,%	VI,mL/g	TI,Nm/g	BI,kN/g	TeI,mNm^2/g
1	185	90	70	44.85	362	15.1	0.66	1.45
2	200	120	80	39.74	667	11.5	0.54	1.25
3	170	120	80	57.99	162	6.3	-	0.98
4	200	120	60	34.74	638	12.9	0.51	1.18
5	170	120	60	48.73	244	14.5	0.61	1.43
6	200	60	80	44.86	496	15.5	0.60	1.33
7	170	60	80	61.50	112	-	-	0.74
8	200	60	60	36.56	689	13.4	0.52	1.25
9	170	60	60	55.55	157	7.0	-	0.89
10	185	120	70	47.05	538	18.9	0.73	1.52
11	185	60	70	47.35	337	16.5	0.73	1.52
12	185	90	80	47.00	309	16.3	0.70	1.53
13	185	90	60	41.48	523	17.3	0.74	1.52
14	200	90	70	36.90	699	12.9	0.54	1.41
15	170	90	70	57.63	169	8.0	0.33	1.02

T, t, y E = Temperature, time and ethanol concentration, respectively
YI= yield; VI= viscosity; TI= tensile index; BI= burst index; TeI= tear index

Table 2. *Values of statistical parameters for the equations 7-11 in the pulping with ethanol of the orange tree prunings.*

Equation	Multiple-R	R^2	R-adjusted	p<	F>	t>
[7] Yield	0.98	0.97	0.96	0.0762	3.91	1.98
[8] Viscosity	1.00	0.99	0.99	0.0120	10.46	3.23
[9] Tensile index	0.98	0.97	0.94	0.0467	5.52	2.35
[10] Burst index	0.96	0.92	0.88	0.0524	4.99	2.23
[11] Tear index	0.95	0.91	0.86	0.0718	4.16	2.04

Table 3. *Values of the operational variables in the pulping with ethanol of the orange tree prunings, to obtain optimal values for the dependent variables.*

Dependent variable	Optimal values for the dependent variables	Values of the independent variables to obtain optimum values for the dependent variables		
		X_T	X_t	X_E
Yield, %	61.4	-1	-1	1
Viscosity, mL/g	987	1	1	-1
Tensile Index, Nm/g	18.8	0.02	1	-1
Burst Index, kN/g	0.81	0.4	-1	1
Tear Index, mNm^2/g	1.58	0.15	1	1

X_T, X_t, X_E = normalized values for temperature, time and ethanol concentration

Table 4. *Values of maximum variations in the dependent variables to vary an operational variable, kept the remaining in their optimal values in the pulping with ethanol of the orange tree prunings. (In brackets the percentages of these variations on the optimal values of the dependent variables).*

Equation	Variation of the dependent variables with operational variables		
	Temperature, ºC	Time, min	Ethanol concentration, %
YI, % [7]	17.70 (28.8%)	3.5 (5.7%)	6.8 (11.1%)
VI, mL/g [8]	728 (73.7%)	296 (29.9%)	228 (23.0%)
TI, Nm/g [9]	6.48 (34.5%)	2.33 (12.2%)	3.85 (20.5%)
BI, kN/g [10]	0.52 (64.3%)	0.16 (19.8%)	0.16 (19.8%)
TeI, mNm^2/g [11]	0.48 (30.4%)	0.09 (5.7%)	0.03 (1.9%)

Table 5. *Values of the constant a_i of the neural fuzzy model for the pulps properties, and values of R^2 in the pulping with ethanol of the orange tree prunings.*

Rule	T,ºC	t, min	E, %	Value of a_i for	
				YI, %	VI, mL/g
1	170	60	60	57.1	290
2	170	60	80	63.1	247
3	170	120	60	49.6	226
4	170	120	80	58.8	165
5	185	60	60	41.7	417
6	185	60	80	47.5	228
7	185	120	60	42.3	632
8	185	120	80	48.1	439
9	200	60	60	36.1	701
10	200	60	80	44.2	508
11	200	120	60	33.9	1001
12	200	120	80	38.9	763
9	170	90	60	-	-
10	170	90	80	-	-
11	200	90	60	-	-
12	200	90	80	-	-
R^2	-	-	-	0.98	1.00

T, t, and E = Temperature. Time and ethanol concentration. respectively;
YI = yield; VI = Viscosity

Table 6. *Values of the constant a_i of the neural fuzzy model for the paper-sheets properties, and values of R^2 in the pulping with ethanol of the orange tree prunings.*

Rule	T,ºC	t, min	E, %	Value of the c_i for		
				IT, Nm/g	IE, kN/g	ID,mNm^2/g
1	170	60	60	6.53	0.198	0.708
2	170	60	80	5.82	0.245	0.542
3	170	120	60	12.6	0.595	1.278
4	170	120	80	5.73	0.202	0.795
5	185	60	60	16.8	0.764	1.395
6	185	60	80	16.22	0.742	1.431
7	185	120	60	19.69	0.764	1.389
8	185	120	80	18.5	0.711	1.405
9	200	60	60	13.19	0.500	1.112
10	200	60	80	15.49	0.590	1.206
11	200	120	60	12.49	0.490	1.048
12	200	120	80	11.06	0.530	1.116
R^2	-	-	-	0.98	0.99	0.97

T, t, and E = Temperature, time and ethanol concentration, respectively;
TI= tensile index, BI= burst index, TeI= tear index

Table 7. *Estimated values of the dependent variables related with the pulps, using neural fuzzy models, and errors (percentage in brackets) with respect to the experimental values in the pulping with ethanol of the orange tree prunings.*

Experiment	T, ºC	t, min	E, %	YI, %	VI, mL/g
1	185	90	70	45.2 (0.8)	436 (0.4)
2	200	120	80	39.4 (0.9)	744 (0.7)
3	170	120	80	58.2 (0.3)	181 (3.5)
4	200	120	60	34.4 (1.0)	979 (1.3)
5	170	120	60	49.2 (0.9)	249 (2.4)
6	200	60	80	44.4 (1.0)	492 (0.8)
7	170	60	80	62.1 (1.1)	246 (2.4)
8	200	60	60	36.4 (0.4)	684 (0.7)
9	170	60	60	56.2 (1.1)	298 (1.9)
10	185	120	70	45.2 (3.9)	536 (0.4)
11	185	60	70	45.2 (4.6)	335 (0.4)
12	185	90	80	48.2 (2.5)	343 (1.9)
13	185	90	60	42.2 (2.5)	528 (1.1)
14	200	90	70	38.7 (4.8)	725 (3.6)
15	170	90	70	56.4 (2.1)	244 (7.4)

T, t, y E = Temperature, time and ethanol concentration ethanol, respectively
YI = yield; VI = Viscosity

Table 8. *Estimated values of the dependent variables related to the paper-sheets, using the neural fuzzy models, and errors (percentage in brackets) with respect to the experimental values in the pulping with ethanol of the orange tree prunings.*

Experiment	T, ºC	t, min	E, %	TI, Nm/g	BI, kN/g	TeI, mNm^2/g
1	185	90	70	17,0 (12,1)	0,710 (7,89)	1,358 (6,14)
2	200	120	80	11,5 (0,4)	0,541 (0,34)	1,133 (9,23)
3	170	120	80	6,5 (2,6)	0,232 (1,27)	0,831 (14,90)
4	200	120	60	12,9 (0,3)	0,507 (0,49)	1,068 (9,75)
5	170	120	60	13,0 (1,1)	0,605 (0,44)	1,285 (10,21)
6	200	60	80	15,5 (0,3)	0,599 (0,41)	1,219 (8,64)
7	170	60	80	6,4 (2,6)	0,275 (1,12)	0,595 (19,62)
8	200	60	60	13,4 (0,3)	0,515 (0,42)	1,129 (9,85)
9	170	60	60	7,1 (2,3)	0,232 (1,44)	0,749 (16,26)
10	185	120	70	18,1 (3,8)	0,707 (2,74)	1,360 (10,60)
11	185	60	70	15,8 (4,3)	0,713 (2,50)	1,356 (10,60)
12	185	90	80	16,5 (1,0)	0,690 (1,56)	1,363 (11,12)
13	185	90	60	17,5 (0,9)	0,729 (1,25)	1,353 (11,24)
14	200	90	70	13,3 (3,4)	0,540 (0,33)	1,137 (19,59)
15	170	90	70	8,3 (3,0)	0,336 (1,40)	0,865 (15,01)

T, t, and E = Temperature, time and ethanol concentration, respectively
TI = Tensile index; BI = Burst index; TeI = Tear index

Table 9. *Additional experiments to validate the polynomial and neural fuzzy models in the pulping with ethanol of the orange tree prunings.*

Parameter	1	2	3	4	5	6
T, ºC	180	190	180	190	180	190
t, min	75	105	75	105	75	105
E, %	65	75	65	75	65	75
YI, % (error %)	48.5	42.7	47.5 (1.9)	43.9 (2.8)	47.1 (2.9)	44.4 (4.0)
VI, mL/g (error %)	401	541	378 (5.7)	512 (5.2)	380 (5.3)	524 (3.2)
TI, Nm/g (error %)	16.8	15.8	15.5 (7.4)	16.3 (3.5)	14.5 (13.4)	16.5 (4.2)
BI,kN/g (error %)	0.60	0.63	0.64 (6.9)	0.70 (11.7)	0.62 (3.5)	0.67 (6.6)
TeI, mNm^2/g (error %)	1.31	1.63	1.40 (7.1)	1.55 (5.0)	1.23 (6.1)	1.32 (18.8)

T, t, and E = Temperature, time and ethanol concentration, respectively; YI = Yield; VI = Viscosity; TI = Tensile index; BI = Burst index; TeI = Tear index
Columns 1 and 2: Experimental data of the dependent variables in 2 experiments
Columns 3 and 4: Experimental data using polynomial equations found and deviations (%) vs experimental data of the columns 1 and 2, respectively
Columns 5 and 6: Experimental data using neural fuzzy equations found and deviations (%) vs experimental data of the columns 1 and 2, respectively

4. Conclusions

Although neurofuzzy models use a greater number of parameters than second order polynomial models, both types of models are equally effective for a typical experimental design of three variables of operation, with a total of 15 experiments. A neurofuzzy model offers physical interpretation of the constants (parameters) in so far as they represent averages of the properties values (dependent variables) destination under the conditions defined by the specific used fuzzy rule.

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