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Short Communication

Production of pulp and energy using orange tree prunings

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The aim of this work was to chemically characterize orange tree prunings and use it in pulping and combustion processes. Soda-anthraquinone pulping of the main fraction of orange pruning (stems with a diameter >0.5 cm) was simulated with polynomial and neurofuzzy models, that predicted pulp properties as a function of operating variables (155–185 °C, 40–90 min, soda concentration, 10–16%) with errors less than 20%. The heating values (16,870 kJ/kg), the flame temperature (1150–2150 °C) and dew point temperature of fuel gas (47–53 °C) for the residual fraction from orange pruning (stems diameter <0.5 cm and leaves) was determined and compared with other non-wood lignocellulosic materials. As a consequence the price of kJ obtained by combustion of this residual fraction is less than other lignocellulosic materials, much lower than those of fossil fuels.

1. Introduction

Spain alone produces more than 5,000,000 tons of orange tree prunings per year (Rodríguez et al., 2010) that could potentially be converted to value-added products. The woody fraction can be used for the production of pulp and various chemicals (Dogaris et al., 2009; Alfaro et al., 2009; Ziaie-Shirkolaee et al., 2008). The remaining fraction consisting mainly of leaves, bark, pith and young stems with relatively low cellulose contents could potentially be used in combustion processes (Arvelakis and Koukios, 2002; Ozturk and Bascetinlik, 2006; Overend and Wright, 2008).

Therefore, the aim of this study was to evaluate the optimal use of the two fractions derived from orange tree prunings. The main fraction was pulped with soda–anthraquinone and the influence of operating variables on the properties of pulps and paper was determined. The residual fraction was used as fuel, and heating values, flame temperature and dew point temperature of the combustion gases were measured.

2. Experimental

2.1. Material

Orange tree prunings were separated into tstems with a diameter of >1 cm and a fraction consisting leaves and stems with diameter less than 1 cm.

The following analytical procedures were used: moisture (Tappi T11m-59), holocellulose (Tappi T9m-54), lignin (Tappi T13m-59), extractives (Tappi T6m-59), ash (Tappi T15m-58), volatile (UNE-32019), fixed carbon (difference between 100 and the sum of ashes plus volatiles) an elemental analysis (Dumas method with a Eurovector EA 3000).

2.2. Pulping and sheetsmaking

Pulp was obtained by using a 15-L batch cylindrical rotatory reactor, under the conditions listed in Table 2. Paper sheets were prepared on an ENJO-F-39.71 sheet machine according to the TAPPI 220 standard method.

2.3. Characterization of the pulp and paper sheets

The pulp and paper obtained were characterized according to the following standard methods: yield (gravimetrically), viscosity (Tappi T230om-94), Kappa number (Tappi T236cm-85), Lignin (Tappi T222), breaking length (Tappi T494om-96), burst index (Tappi T403om-97), tear index (Tappi T414om-98) and brightness (Tappi T525om-92).

2.4. Experimental design and modeling used

To quantify the effects of operational variables on the dependent variables, a 2^n factorial design was used (Montgomery, 1991).

Experimental data (operational and dependent variables) were fitted to a second-order polynomial model. The values of the

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operational variables were normalized to values from −1 to +1 by using the following equation:

$$X_n = \frac{2(X - \bar{X})}{X_{\max} - X_{\min}} \quad (1)$$

where X_n is the normalized value of T (temperature), t (processing time) and S (soda concentration); X is the actual experimental value of the variable concerned; $\bar{X}$ is the mean of $X_{\max}$ and $X_{\min}$; and $X_{\max}$ and $X_{\min}$ are the maximum and minimum value, respectively, of such a variable.The constant parameters in the polynomial equation were estimated by using the BMDP© program.

The application of neural fuzzy model involves using the following equation (Jang et al., 1997):

$$Y_e = \frac{\sum_{l=1}^{8} c_1 R_1}{\sum_{l=1}^{8} R_1} \quad (2)$$

where Y_e is the estimated value of the property to be modeled (dependent variable), c_l a constant parameter and R_l a fuzzy rule.

With three independent variables one can establish the following eight fuzzy rules according to the extreme (high and low) values of such variables (x_{1i}, low and x_{2i} high):

R_1: low T, low t and low S: $R_1 = x_{1T} \cdot x_{1t} \cdot x_{1S}$
R_2: low T, low t and high S: $R_2 = x_{1T} \cdot x_{1t} \cdot x_{2S}$
. . ..
R_7: low T, high t and high S: $R_7 = x_{1T} \cdot x_{2t} \cdot x_{2Si}$
R_8: high T, high t and high S: $R_8 = x_{2T} \cdot x_{2t} \cdot x_{2S}$

The values low (x_{1i}) and high (x_{2i}) are obtained by following equation (linear membership function):

$$X_{1i} = 1 - \frac{(xi - x_{\text{low } i})}{x_{\text{high } i} - x_{\text{low } i}} \quad (3)$$

$$X_{2i} = \frac{(xi - x_{\text{low } i})}{x_{\text{high } i} - x_{\text{low } i}} \quad (4)$$

$x_{\text{high } i}$ and $x_{\text{low } i}$ denoting the extreme values of the operational variable and x_i the absolute value of T, t or S.

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. (2) would include 12 terms in the numerator and 12 in the denominator. The values x_{ij} (low -x_{1i}-, medium -x_{2i}- and high -x_{3i}- of operational variable (T, t and t) are obtained by following equation (Gaussian membership function):

$$X_{ji} = \exp[-0.5((x_i - x_{ci}/L_i))^2] \quad (5)$$

where x_i denotes the absolute value of the variable concerned; x_{ci} its minimum, central or maximum value; and L_i the width of its Gaussian distribution.

The constant parameters in the Eq. (2) were estimated by using the ANFIS (Adaptative Neural Fuzzy Inference System) Edit tool in the Matlab© 6.5 software suite.

2.5. Heating value

The gross calorific values (constant volume) were determined according to EN/TS 14918:2005 (E) Solid biofuels – method for the determination of the heating value, and UNE 164001 EX standards by using a Parr 6200 Isoperibol Calorimeter.

3. Results and discussion

3.1. Analysis of the orange tree pruning

The results of the elemental analysis and the contents of cellulose, lignin, ethanol–benzene extractable, ash, volatile and fixed carbon for the two fractions of orange tree pruning are presented in Table 1. These values are the averages of three trials for each of the variables measured. These results were similar to those found in the literature (Jiménez et al., 1991; Jiménez and González, 1991; Ververis et al., 2009) for other lignocellulosic materials (wheat straw, sunflower stalks, vine shoots, cotton stalks, olive tree pruning, olive wood, etc.).

Table 1
Elemental analysis and components analysis of orange tree pruning.

Analysis	Components	Main fraction	Residual fraction
Elemental	Carbon (%)	45.45	42.21
	Hydrogen (%)	6.16	5.92
	Nitrogen (%)	0.41	1.29
	Sulfur (%)	0.01	0.02
	Holocellulose (%)	73.20	63.39
Components	Lignin (%)	19.95	14.78
	Extractives (%)	3.57	6.49
	Ash (%)	3.37	14.84
	Volatile (%)	78.79	78.30
	Fixed carbon (%)	17.84	6.86

Standard deviation for all variables is less than 2% in all cases.

The percentage of sulfur was very low, so the combustion gases of these lignocellulosic materials were poor in SO_2, compared to gases from the combustion of fossil fuels (Jiménez and González, 1991).

The ash content of residual fraction of the orange tree prunings (14.84%) was higher than that of wheat straw (6.49%) and sunflower stalks (9.57%); the ash content of the main fraction of the orange tree prunings (3.37%) was similar to that the olive prunings (3.26%) and higher than that of cotton stalks (2.17%), vine shoots (2.66%) and olive wood (1.18%) (Jiménez and González, 1991; Jiménez et al., 1991).

3.2. Soda–anthraquinone pulping

The soda–anthraquinone pulping has been applied to various non-wood materials (Mahiuddin et al., 2005; López et al., 2005; Abrantes et al., 2007; Labidi et al., 2008; Jiménez et al., 2009a), but not yet to orange tree pruning. Table 2 shows the results of the pulping under various conditions.

3.2.1. Polynomial models

By fitting the experimental data to an polynomial equation the following equations were obtained:

$$\text{YI} = 40.56 - 3.73X_T - 1.95X_t - 4.06X_S + 1.38X_t^2 \quad (R^2 = 0.95; p < 0.7777; t > 1.97) \quad (6)$$

$$\text{KN} = 47.62 - 12.98X_T - 10.71X_t - 16.99X_S + 14.85X_S^2 \quad (R^2 = 0.87; p < 0.0197; t > 2.77) \quad (7)$$

$$\text{Li} = 7.66 - 2.62X_T - 1.80X_t - 2.73X_S + 1.74X_S^2 \quad (R^2 = 0.90; p < 0.0524; t > 2.20) \quad (8)$$

$$\text{VI} = 7.99 + 87X_T + 67X_t + 53X_s - 143X_T^2 + 59X_t^2 - 154X_S^2 - 44X_TX_t - 149X_TX_S \quad (R^2 = 0.96; p < 01843; t > 1.50) \quad (9)$$

$$\text{TE} = 16.67 + 1.30X_T + 2.90X_S + 2.63X_TX_S^2 \quad (R^2 = 0.84; p < 0.0283; t > 2.52) \quad (10)$$

$$\text{BI} = 0.65 + 0.11X_S + 0.10X_TX_S \quad (R^2 = 0.72; p < 0.0043; t > 3.52) \quad (11)$$

Table 2
Values of operating variables used in the experimental design applied in the cooking of the main fraction of pruning orange free with soda–anthraquinone and values of dependent variables.

Experiment	*T* (°C)	*t* (min)	*S* (%)	YI (%)	KN	Li (%)	VI (mg/L)	TE (N m/g)	BI (kN/g)	TI (mN m^2/g)	BR (%)
1	170	65	13	40.87	45.0	7.50	894	16.0	0.65	1.13	22.8
2	185	90	16	34.10	25.5	2.82	548	21.0	0.73	1.13	29.9
3	155	90	16	38.04	39.8	6.27	782	17.0	0.69	1.10	26.8
4	185	90	10	38.22	47.0	6.46	829	13.0	0.50	1.07	26.7
5	155	90	10	48.90	89.5	14.37	393	16.0	0.61	1.24	19.8
6	185	40	16	34.86	37.1	4.87	587	24.0	0.95	1.32	28.2
7	155	40	16	44.23	85.5	13.20	571	14.0	0.54	0.97	18.8
8	185	40	10	45.02	88.6	13.20	685	11.0	0.37	0.95	18.2
9	155	40	10	51.81	91.6	15.51	145	15.0	0.58	1.25	13.5
10	170	90	13	40.75	44.6	6.77	889	16.0	0.61	1.09	25.0
11	170	40	13	43.55	50.7	7.91	781	19.0	0.71	1.32	22.6
12	170	65	16	36.94	39.5	6.19	666	21.0	0.83	1.15	27.3
13	170	65	10	44.86	80.5	11.13	577	13.0	0.55	1.09	20.0
14	185	65	13	36.83	38.1	5.98	691	20.0	0.74	1.16	27.2
15	155	65	13	43.32	59.7	10.15	575	14.0	0.62	1.18	22.5
16	162.5	52.5	11.5	45.85	70.0	12.87	570	15.7	0.69	1.23	20.1
17	177.5	77.5	14.5	36.40	37.5	5.42	810	18.9	0.70	1.10	25.9

T, *t* and *S* = Temperature, time, and soda concentration, respectively; YI = Yield; KN = Kappa Number; Li = Lignin; VI = Viscosity; TE = Tensile index; BI = Burst Index; TI = Tear Index; BR = Brightness. Standard deviation for all variables is less than 3% in all cases.

$$\mathrm{TI} = 1.14 + 0.11X_TX_S \quad (R^2 = 0.55; p < 0.0015; t > 4.00) \tag{12}$$

$$\mathrm{BR} = 23.29 + 2.88X_T + 2.69X_t + 3.28X_S \quad (R^2 = 0.90; p < 0.0003; t > 5.20) \tag{13}$$

where YI is the yield, KN Kappa number KN, Li lignin content, VI viscosity, TE tensile index, BI burst index, TI tear index, BR brightness, and X_T, X_t, X_S are normalized values of temperature, time and soda concentration, respectively.

The values estimated using the above equations reproduce the experimental results of the dependent variables with errors less than 6%, 20%, 20%, 11%, 13%, 20%, 13% and 12%, respectively for the pulp yield, Kappa number, lignin content, viscosity, tensile index, burst index, tear index and brightness.

The polynomial models were validated by conducting two pulping experiments (rows 16 and 17 of Table 2). The errors in the predictions of experimental results are similar or lower to than those found for the 15 experiments of the experimental design; this fact confirms the validity of the proposed polynomial models.

From the Eqs. (6)–(13) can be deduced that if a high value of brightness, tensile index and burst index is desired, it is necessary to operate with high values of temperature and soda concentration; for brightness also a high value of processing time is necessary. Similarly, high values of temperature and time have a positive influence to obtain pulps with a lower Kappa number and lignin content; in order to obtain these pulps, the appropriate value of soda concentration should be moderately high. For high viscosity, the temperature and soda concentration should be medium and the time long. Finally it must be noted that the pulp yield and the tear index were favored by low values of operational variables.

Simulating the pulping process by the Eqs. (6)–(13) can propose several alternatives for the values of operating variables, so as to obtain pulps with features not far from their optimum values, while saving energy, reagents and immobilized capital for industrial facilities, operating with values of temperature, soda concentration and time processing less than the maximum considered, also achieving a better utilization of raw material when operating under not severe conditions. Thus, one possible compromise could be to operate with medium–high standardized values of the three operating variables (+0.8), since the properties of the sheets of paper decreased slightly with respect to their maximum values, which are not very high (7.60% in the worst case for the tensile index). Under these conditions the losses of pulp yield and viscosity are still high (34.89% and 25.94%, respectively), but lower than those that occur when operating under the harshest operating conditions (36.12% and 30.15%, respectively) (which also involve major capital expenditure and fixed manufacturing costs).

3.2.2. Neural fuzzy models

The experimental data of Table 2 were fitted to Eq. (2) to estimate the constants c_l in the equation for the neural fuzzy model established with linear membership functions at two different levels (high and low) for two operational variables and a Gaussian membership function at three levels (high, medium and low) for the other one (Table 3).

The predictions by neural fuzzy model, for yield, Kappa number, lignin, viscosity, tensile index, burst index, tear index and brightness departed by less than 3%, 15%, 11%, 20%, 8%, 12%, 5% and 5% from their respective experimental counterparts. Usually, these errors are fewer than those obtained for the case of using polynomial models.

The neural fuzzy models were validated by conducting two pulping experiments (rows 16 and 17 in Table 2).The errors from the predictions are similar to those found for the experimental design experiments, which confirmed the validity of the proposed neural fuzzy models.

The neural fuzzy models allow the influence of each operational variable on the target properties to be assessed. This can be easily illustrated with the results for yield. Applying rules 1 and 2 in Table 3 reveals that, with low levels of the operational variables (rule 1), increasing the soda concentration (rule 2) decreases the c_l for yield (from 51.82% to 44.26%). Likewise, a comparison of rules 1 and 3 reveals that, at low temperature and soda concentration levels, increasing the pulping time decreases the c_l for yield from 51.82% to 49.02% on average. Finally, increasing the temperature (rules 1 and 4) decreases the c_l for yield from 51.82% to 45.01%. Based on the foregoing, soda concentration is the most influential independent variable and time the least.

One can virtually freely combine two rules with identical levels for two variables and a different one for the third to assess the influence of the last on each target property.

Fuzzy neural and polynomial models have also been tested successfully for other non-wood materials: vine shoots, *Leucaena leu-*

Table 3
Values of the constants c_i in the neutral fuzzy model for the pulp properties and R^2 value.

Rule	T (°C)	t (min)	S (%)	YI (%)	KN	Li (%)	VI (mg/L)	TE (N m/g)	BI (kN/g)	TI (mN m^2/g)	BR (%)
1	155	40	10	51.82	92.7	15.70	119	15.0	0.61	1.24	13.0
2	155	40	16	44.26	84.8	13.44	574	14.5	0.55	0.95	18.8
3	155	90	10	49.02	90.6	14.54	377	16.3	0.61	1.25	19.5
4	185	40	10	45.01	90.2	13.50	692	10.3	0.38	0.92	17.7
5	185	90	10	36.08	47.5	6.41	840	12.7	0.49	1.07	26.6
6	185	40	16	34.68	35.4	4.86	581	24.7	0.97	1.33	28.5
7	155	90	16	37.97	37.6	6.14	792	17.9	0.68	1.11	27.1
8	185	90	16	34.19	23.6	2.74	532	21.8	0.71	1.14	30.2
9	155	65	10	47.39	–	–	–	–	0.49	–	–
10	155	65	16	39.35	–	–	–	–	0.75	–	–
11	185	65	10	40.92	–	–	–	–	0.59	–	–
12	185	65	16	33.17	–	–	–	–	0.91	–	–
9	155	40	13	–	56.8	9.82	671	15.7	–	1.32	20.7
10	155	90	13	–	53.6	8.22	789	12.3	–	1.07	22.7
11	185	40	13	–	36.0	5.96	796	22.4	–	1.31	25.3
12	185	90	13	–	32.7	5.21	887	18.7	–	1.04	27.1
R^2				0.99	0.99	0.99	0.88	0.97	0.97	0.97	0.98

T, *t* y *S* = Temperature, time, and soda concentration, respectively; YI = Yield; KN = Kappa Number; Li = Lignin; VI = Viscosity; TE = Tensile index; BI = Burst Index; TI = Tear Index; BR = Brightness.

cacephala, *Chamaecytisus proliferus* and cotton stalks (Rodríguez et al., 2008; Jiménez et al., 2009b). This confirms the universal validity of these models.

3.3. Energy valuation of orange tree pruning

3.3.1. Heating values

Table 4 presents the experimental results of the heating values of the two factions of the orange tree pruning. These values are of the same magnitude as those found in the literature (Jiménez et al., 1991; Jiménez and González, 1991) for different lignocellulosic materials (wheat straw, sunflower stalks, vine shoots, cotton stalks, pruning olive, olive stones, olive marc, holm oak residues and eucalyptus residues).

In the literature (Jiménez et al., 1991; Jiménez and González, 1991) are found empirical equations that predict the heating values (HV, kJ/kg) of lignocellulosic materials:

$$\text{HV} = 393.81C + 230.22 \quad (14)$$

$$\text{HV} = 436.66C - 305.51 \quad (15)$$

$$\text{HV} = 173.89\text{Ce} + 266.29L + 321.87E \quad (16)$$

$$\text{HV} = 173.89\text{Ce} + 266.29(100 - \text{Ce}') \quad (17)$$

$$\text{HV} = (1 - A/(\text{Ce} + L + E))(173.89\text{Ce} + 266.29L + 321.87E) \quad (18)$$

$$\text{HV} = 339.82T - 14308.93 \quad (19)$$

$$\text{HV} = 313.30T - 10814.08 \quad (20)$$

where C is the total carbon content (%), Ce, L, E and Z content of holocellulose, lignin, extractives and ash (all in %), Ce′ the holocellulose content on free base of extractives (%), and T is the sum of the contents of volatile and fixed carbon.

The heating values and errors of the estimates are provided in Table 4. The lowest errors were found with Eqs. (14), (16) and Eq. (20) reproduces the heating values of the fractions of pruning orange tree with errors less than 6%, improving the values predicted by the Eq. (15).

3.3.2. Flame temperature and dew point temperature

Using the elemental analysis of the orange tree pruning (Table 1) and following the estimation techniques described in the literature (Jiménez et al., 1991) the flame and dew point temperatures for different values of excess air used in combustion were determined (Table 5). The flame temperature and dew points are of the same magnitude as those of other lignocellulosic materials (wheat straw, sunflower stalks, vine shoots, cotton stalks, olive wood and olive tree pruning) (Jiménez et al., 1991). The high flame temperature of both fractions suggests the possibility of using these materials in the production of steam. The dew point is low for combustion gases, thus condensation in chimneys and flue pipes that could cause corrosion would be minimal, and would likely not be an issue given the low sulfur content (Jiménez et al., 1991).

Table 4
Heating values of the fractions of the orange tree pruning experimentally and determined by Eqs. (14)–(20), and errors compared to recent experimental.

Heating values (kJ/g)	Main fraction	Residual fraction
Experimental	18626	16870
Calculated Eq. (14)	18133(2.65%)	16853(0.10%)
Calculated Eq. (15)	19545(4.93%)	18126(7.45%)
Calculated Eq. (16)	19190(3.03%)	17048(1.06%)
Calculated Eq. (17)	19144(2.78%)	19600(16.81%)
Calculated Eq. (18)	18522(0.56%)	14059(16.66%)
Calculated Eq. (19)	18528(0.53%)	14630(13.28%)
Calculated Eq. (20)	19460(4.48%)	15867(5.95%)

Standard deviation for all variables is less than 2% in all cases.

3.3.3. Comparison of cost of the heat units obtained by combustion

Table 6 compares the heating values, unit cost of the fuel and cost of the heat units obtained by combustion of the different types of fuel. The residual fraction from tree prunings (paper industry wastes) and empty fruit bunches from the oil–palm industry (an agrofood waste) are the cheapest, since only the costs of upgrading the energy plant have to be considered, and other costs, such as for collection in the fields and transport to the combustion plant do not have to be considered.

As can be seen in Table 6, the MkJ of energy obtained by combustion of industrial waste is cheaper than that obtained from the agricultural residues (olive tree and orange tree and banana tree pruning), which in turn is cheaper than the one obtained from mineral coal and even much cheaper than the one obtained from fossil fuel fluids. Moreover, the lignocellulosic residues are renewable, release only small amounts of sulfur dioxide in combustion gases and produce smaller amounts of ash than the solid fossil fuels.

Table 5
Flame temperature and dew point temperature in combustion of orange tree pruning.

Temperature (%)	Material	Heat loss (%)	Excess air in the combustion				
			10	20	30	40	50
Flame temperature	Main fraction	10	2189	2079	1981	1893	1815
		20	1999	1899	1811	1732	1661
		30	1806	1717	1638	1568	1505
		40	1609	1531	1463	1401	1346
		50	1409	1342	1283	1231	1184
Flame temperature	Residual fraction	10	2174	2067	1972	1886	1809
		20	1986	1889	1803	1726	1656
		30	1794	1708	1631	1562	1501
		40	1599	1524	1456	1396	1342
		50	1400	1336	1278	1227	1181
Dew point temperature	Main fraction	–	51.8	50.3	48.8	47.5	46.3
	Residual fraction	–	53.0	51.4	50.0	48.7	47.5

Table 6
Comparison of heating values and energy costs obtained by combustion of various fuel types.

Fuel	Heating values (MkJ/t)	Cost of fuel (€/t)	Cost of the unit of heat (€/MkJ)
Main fraction of orange tree pruning	18.63	60	3.22
Residual fraction of orange tree pruning	16.87	30	1.78
Main fraction of olive tree pruning	19.11	60	3.14
Residual fraction of olive tree pruning	18.70	30	1.60
Hesperaloe funifera	17.76	60	3.38
Empty fruit bunches of oil-palm industry	19.05	30	1.57
Banana tree	17.75	60	3.38
Coal	25.94	100	3.86
Heating oil	37.67	800	21.24
Commercial propane	43.89	1650	37.59

Data supplied by the company Ecopapel of Écija, Seville, Spain, http://www.ecopapel.es.

4. Conclusions

The main (wood) fraction of orange tree pruning can be used to obtain soda pulp of acceptable quality. The residual fraction (young stems and leaves) of orange tree pruning is suitable for cheap energy production by combustion. The utilization of orange tree prunings in this manner can decrease costs to farms and reduce potential damage to ecological systems (fire, pests, etc.), while avoiding deforested by using a non-wood raw material.

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