Refining of Soda-AQ, Kraft-AQ, and Ethanol Pulps from Orange Tree Wood

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The pulp yield of orange tree wood was tested under various conditions including processing with soda-anthraquinone (soda-AQ), kraft-anthraquinone (kraft-AQ), or ethanol under different temperature, time, reagent concentration, and PFI laboratory beater beating regimes. Beating grade and stretch properties were studied, with a view to identifying the optimum operating conditions. Polynomial equations were derived that generally reproduced the dependent variables, with errors in most cases much less than 20%. Kraft-AQ pulping was the most efficient. The values of the tensile, burst, and tear indices obtained with kraft-AQ (78.04 Nm/g, 4.84 kN/g, and 2.97 mNm^2/g, respectively), were in most cases higher than those found for soda-AQ and ethanol pulps. Using lower values of operational conditions than those required to maximize the studied paper properties (170 °C, 65 min, 13% active alkali, and 2700 number of PFI beating revolutions), it was possible to provide a more energy- and chemically-efficient process for industrial facilities.

Keywords: Orange tree wood; Soda-anthraquinone; Kraft-anthraquinone; Ethanol; Pulp refining

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INTRODUCTION

Since the 1970s, pulp production from non-woody plants using non-conventional raw materials has increased from approximately 7% to almost 12% of the total pulp produced, growing at a rate of 2 to 3 times greater than wood pulps (Atchinson 1996; Simula 2002; Alaejos *et al.* 2004; González *et al.* 2011).

The use of agricultural and agro-industry residues and alternatives to food crops seems to be a good alternative to raw wood material, which can lead to excellent paper products with special properties and can serve as the sole source of raw materials in some geographical areas (Jiménez 2005).

Orange tree prunings could provide a new source of non-wood raw material. Spanish production of orange tree prunings from felling operations is more than 5 million tons per year (Rodríguez *et al.* 2010). Orange tree prunings and crop residues in general must be removed to control pollution, fire, pests, interference with soil cultivation, and occupation of large areas. It is advantageous to try to exploit the different fractions of waste as a method to reduce disposal costs.

Many non-wood raw materials such as orange tree prunings contain fractions unsuitable for the production of pulp, including the leaves, bark, pith, and young stems, which contain relatively low cellulose content. However, these fractions, which can be called residue, could be used as fuel for producing heat for heating (Arvelakis and Koukios 2002; Ozturk and Bascetinlik 2006; Overend and Wright 2005; González *et al.* 2012).

The main fraction of orange tree prunings, such as branches and stems with diameters larger than 1 cm, could be used to produce cellulosic pulps for paper. A single study related to pulping of orange tree prunings was found in the literature (González *et al*. 2011). In this work, a chemical characterization of orange tree prunings (α-cellulose, holocellulose, and lignin contents) was conducted. The group also studied the variables associated with soda-AQ pulping on pulp and papersheet characteristics. The conclusion of this work was that this raw material has potential for cellulosic pulp production.

The aim of the present work was to study and compare three pulping processes, soda-AQ, kraft-AQ, and ethanol, applied to the main fraction of orange tree prunings, as well as the refining of the pulps obtained. The influence of the operational variables, temperature, time, reagent concentrations, and number of PFI beating revolutions, on the characteristics of the papersheets obtained was studied to determine the optimal operating conditions.

EXPERIMENTAL

Raw Material

This work used the main fraction of orange tree (*Citrus sinensis*) prunings, which consisted of the wood from branches and stems with diameters larger than 1 cm. The orange wood was characterized according to TAPPI standards, namely, T9m 54, T203 os-61, T222, T211, and T204 for holocellulose, α-cellulose, lignin, ash, and ethanol-benzene extractives, respectively.

Pulping and Pulp and Paper Characterization

Pulps were obtained using a 15-L batch cylindrical reactor. The raw material was cooked in the reactor using soda-AQ, Kraft-AQ, or ethanol. Next, the cooked materials were fiberized in a wet disintegrator at 1200 rpm for 30 min, and the screenings were separated by sieving through a screen of 0.14-mm mesh size. The pulps were beaten on a PFI refiner from Metrotec with precise control of the number of beating revolutions used. The drainability or beating grade (Shopper-Riegler index) of the pulps was determined according to TAPPI T220 sp-96. Pulp yield was determined by weighing, after removing the uncooked materials.

Paper sheets were prepared with an ENJO-F-39.71 sheet machine by Metrotec according to the TAPPI T205 ps-95 standard. The tensile index, burst index, and tear index of paper sheets were determined according to TAPPI standards T494 om-96, T403 om-97, and T414 om-98, respectively.

Experimental Design

The following procedure was carried out to quantify the effects of the operational variables (3 variables of pulping process and 1 of the refining process): a 2^n factorial design was used for the three pulping process variables, consisting of a central experiment (in the centre of a cube) and 14 additional points (additional experiments lying at the cube vertices and side centers) (Montgomery, 1991); then, the pulps of each of those 15 experiments were subjected to 3 refining experiments, resulting in a total of 60 experiments (with 15 experiments without refining).

RESULTS AND DISCUSSION

The chemical characterization of orange tree wood, as well as other similar non-wood materials and wood of pine and eucalyptus is presented in Table 1.

Table 1. Chemical Characterization of Wood an Non-wood Materials

Parameter	Orange tree wood	Olive tree wood (Jiménez, 2005)	Vine shoots (Jiménez, 2005)	Cotton stalks (Jiménez, 2005)	*Eucalyptus globulus* (Jiménez, 2005)	*Pine pinaster* (Jiménez, 2005)
Holocellulose, %	73.2 ± 0.8	61.5	67.1	72.9	80.5	69.6
α-cellulose, %	48.0 ± 0.5	35.7	41.1	58.5	52.8	55.9
Lignin, %	20.0 ± 0.6	19.7	20.3	21.5	20.0	26.2
Extractives, %	3.6 ± 0.3	10.4	4.9	1.4	1.2	2.6
Ashes, %	3.4 ± 0.3	1.4	3.5	2.2	0.6	0.5

The orange tree wood holocellulose content is higher than the other materials considered with the exception of eucalyptus. The α-cellulose content is higher than the content of olive tree and vine shoots, but less than other materials. The lignin content is similar to the materials considered with the exception of the pine. The extractives are high compared with the cotton stalks, pine and eucalyptus, but lower than the other materials. The ashes are high, as in the case of vine shoots, in comparison with other materials (Table 1).

Operating conditions in the three experimental designs applied to three pulping processes of orange tree wood (soda-AQ, kraft-AQ and ethanol) are presented in Table 2.

Table 2. Experimental Conditions used in the Soda-AQ, Kraft-AQ, and Ethanol Pulping Applied to Orange Tree Wood

Experiment	Soda-AQ (liquid/solid ratio = 8; AQ conc. = 1% (o.d.w.)			Kraft-AQ (liquid/solid ratio = 8; sulfidity = 20% (o.d.w.); AQ conc. = 1% (o.d.w.)			Ethanol (liquid/solid ratio = 8)		
	T, ºC	t, min	S, % (odw.)	T, ºC	t, min	A, %	T, ºC	t, min	E, %
1	170	65	13	170	65	13	185	90	70
2	185	90	16	185	90	16	200	120	80
3	155	90	16	155	90	16	170	120	80
4	185	90	10	185	90	10	200	120	60
5	155	90	10	155	90	10	170	120	60
6	185	40	16	185	40	16	200	60	80
7	155	40	16	155	40	16	170	60	80
8	185	40	10	185	40	10	200	60	60
9	155	40	10	155	40	10	170	60	60
10	170	90	13	170	90	13	185	120	70
11	170	40	13	170	40	13	185	60	70
12	170	65	16	170	65	16	185	90	80
13	170	65	10	170	65	10	185	90	60
14	185	65	13	185	65	13	200	90	70
15	155	65	13	155	65	13	170	90	70

T = temperature; t = time; S = soda conc.; A = active alkali conc.; E = Ethanol concentration

Table 3 shows the normalized values of the operational variables for the 15 experiments of each experimental design, as well as the average values of three experimental values of the pulp yield.

Table 3. Normalized Values of the Pulping Operational Variables and Experimental Average Values (of 3 experiments) of the Pulp Yield for Different Pulps from Orange Tree Wood

Experiment	X_T	X_t	X_C	Pulp yield,% (soda-AQ)	Pulp yield,% (Kraft-AQ)	Pulp yield,% (ethanol)
1	0	0	0	40.67	42.20	44.85
2	1	1	1	34.10	37.40	39.74
3	-1	1	1	38.04	39.45	57.99
4	1	1	-1	38.22	43.70	34.74
5	-1	1	-1	48.90	50.16	48.73
6	1	-1	1	34.86	39.79	44.86
7	-1	-1	1	44.23	46.20	61.50
8	1	-1	-1	45.02	44.15	36.56
9	-1	-1	-1	51.81	54.90	55.55
10	0	1	0	40.75	44.58	47.05
11	0	-1	0	43.55	49.36	47.35
12	0	0	1	36.94	43.63	47.00
13	0	0	-1	44.86	49.88	41.48
14	1	0	0	36.83	42.82	36.90
15	-1	0	0	43.32	48.62	57.63

X_T, X_t and X_c = normalized values of temperature, time and reagent concentration (soda, alkali active or ethanol). These values were calculated by

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

where X is the actual experimental value of the variable concerned (T, t or C); $\bar{X}$ is the mean of X_{max} and X_{min}; and X_{max} and X_{min} are the maximum and minimum value, respectively, of each operational variable.

In all the experiments, deviations from the experimental values with respect to the mean values are always less than 5%

Fifteen pulps were obtained from each of the experimental designs and were refined in three different tests with a number of PFI beating revolutions of 1000, 2000, and 3000. Table 4 shows the values of the operational variables to obtain different refined pulps, as well as the average values of six trials of the beating grade of the pulps and of the tensile, burst, and tear indices of the paper sheets.

Table 5 shows the equations obtained to fit the experimental data of the Table 4 with a polynomial model.

For pulp yield, beating grade and tear index (in the three pulping processes) and for tensile index (in the soda-AQ and kraft-AQ pulping process), the values estimated using the equations in Table 3 reproduced the experimental results with errors smaller than 20% in all cases, being in most cases much less than 20%. Figure 1, for the beating grade in the soda-AQ pulping, confirms what is stated above. For the other dependent variables similar graphics were obtained.

For the burst index (in the three pulping processes) and for the tensile index in the ethanol pulping, polynomial models were not suitable because the values estimated from these variables deviated greatly from the experimental values

Using non-linear programming as implemented in the More and Toraldo method (1989), it was possible to identify the values of the operational variables providing the greatest values of the dependent variables for the pulp and paper sheets (Table 6). It can be seen that for each pulping process, the operating conditions to get the maximum value from a given dependent variable are different. Thus, for example, for soda-AQ pulping, if the paper sheets require a high tensile index (a high breaking length), the pulp should be obtained with medium temperature and a medium-high soda concentration, in addition to a high number of PFI beating revolutions in the pulp refining.

Table 7 shows the highest values of the variations of the dependent variables (obtained from the equations in Table 5, by varying each of the operational variables and keeping the values of the remaining operational variables with their optimal values). Table 7 also shows the maximum deviation of the dependent variables (in %) with respect to their optimal values due to the maximum variations calculated previously.

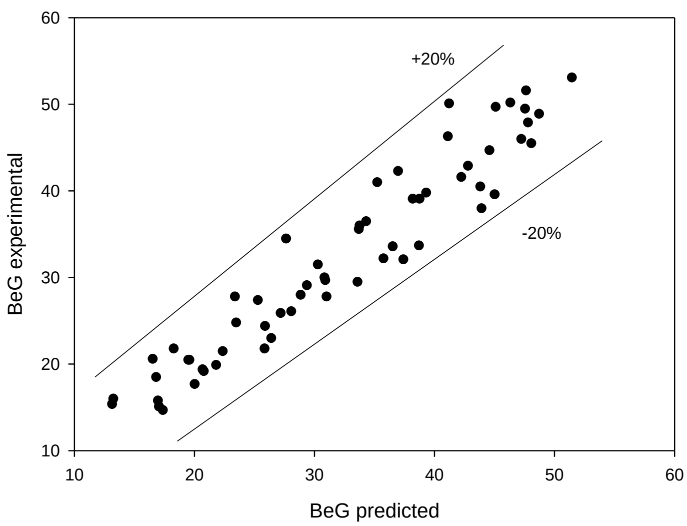

Fig. 1. Values predicted versus experimental for the beating grade (°SR)

From Table 7, it can be concluded that, in the soda-AQ and kraft-AQ pulping, the most influential operational variable on the strength properties of paper was the number of PFI beating revolutions. By contrast, the pulping time affected stretch the least; in the case of ethanol pulping, the most influential variable was also the number of PFI beating revolutions, but the least influential factor was the ethanol concentration.

On the other hand, comparing the results in Table 4, for soda-AQ and kraft-AQ pulping, the burst index values corresponding to the different pairs of experiments that are differentiated only by the value of one of the operational variables (keeping the other three operational variables equal in each of the two compared experiments) verify that the most influential variable on the value of the burst index was the number of PFI beating revolutions and the least influential was the pulping time; for ethanol pulping, the least influential variable on the burst index was ethanol concentration. For ethanol pulping (see Table 4), the most influential variable for the tensile index value was PFI beating revolutions, while the ethanol concentration and the pulping time were less important.

Table 4. Normalized Values of the Pulping and Refining Variables and Experimental Average Values (of 6 experiments) of the Paper Properties for Different Pulps from Orange Tree Wood

Experiment	X_T	X_t	X_C	X_R	Soda-AQ pulps				Kraft-AQ pulps				Ethanol pulps			
					BeG (ºSR)	TsI (Nm/g)	BuI KN/g)	TeI (mNm^2/g)	BeG (ºSR)	TsI (Nm/g)	BuI KN/g)	TeI (mNm^2/g)	BeG (ºSR)	TsI (Nm/g)	BuI KN/g)	TeI (mNm^2/g)
1	0	0	0	-1	21.7	20.00	1.10	1.36	17.7	18.86	0.92	1.12	25.7	15.14	0.66	1.45
2	0	0	0	-0.33	34.4	42.22	2.15	2.29	34.7	43.23	2.33	2.64	26.0	24.22	1.21	1.80
3	0	0	0	+0.33	42.2	60.14	2.99	2.55	45.4	58.38	3.78	2.87	33.9	54.11	2.88	2.46
4	0	0	0	1	50.1	68.00	3.28	2.69	52.1	65.14	3.86	2.93	44.8	63.29	3.46	2.58
5	1	1	1	-1	19.8	19.00	0.70	1.13	21.5	21.75	0.88	1.21	25.4	11.45	0.54	1.25
6	1	1	1	-0.33	31.4	40.51	1.74	2.21	36.0	42.18	2.44	2.67	25.6	27.41	1.28	1.98
7	1	1	1	+0.33	39.0	49.58	2.86	2.54	56.7	61.79	3.95	2.83	35.1	47.95	2.74	2.37
8	1	1	1	1	45.9	55.69	3.08	2.49	62.8	79.74	4.87	3.02	49.1	55.00	3.05	2.42
9	-1	1	1	-1	18.4	17.00	0.70	1.10	22.0	22.20	0.78	1.16	14.7	6.32	0.24	1.08
10	-1	1	1	-0.33	27.3	33.26	1.24	2.10	32.9	49.66	2.72	2.61	14.8	17.93	0.73	2.15
11	-1	1	1	+0.33	35.9	44.50	2.62	2.61	48.8	81.87	4.39	2.82	17.0	24.82	0.88	1.86
12	1	1	-1	1	41.5	55.99	3.87	2.78	62.6	87.31	5.61	3.07	25.1	20.56	1.52	1.77
13	1	1	-1	-1	19.3	10.00	0.30	1.06	20.6	11.76	0.40	0.96	22.2	12.88	0.51	1.18
14	1	1	-1	-0.33	29.6	29.85	1.61	2.28	28.7	31.96	1.59	2.30	22.7	25.21	1.16	1.57
15	1	1	-1	+0.33	46.2	32.63	1.75	2.37	48.3	50.05	2.90	2.75	27.4	37.10	2.00	2.09
16	-1	1	-1	1	48.8	53.21	2.47	2.49	57.1	58.85	3.39	2.86	36.1	44.89	2.62	2.06
17	-1	1	-1	-1	19.1	15.00	0.60	1.24	22.6	21.14	0.71	1.46	15.6	12.87	0.61	1.43
18	-1	1	-1	-0.33	27.7	24.35	1.05	2.08	26.6	45.80	2.15	2.59	15.9	27.06	1.13	1.85
19	-1	1	-1	+0.33	50.0	47.64	3.55	2.75	46.7	63.31	3.38	2.74	20.1	44.31	2.23	2.21
20	-1	1	-1	1	53.0	60.60	3.21	2.51	44.6	59.87	2.88	2.71	27.5	45.42	2.49	2.33
21	1	-1	1	-1	21.4	21.00	0.90	1.32	23.5	14.59	0.61	1.15	18.4	15.49	0.60	1.34
22	1	-1	1	-0.33	29.9	37.00	1.64	1.85	24.6	36.76	1.90	2.53	18.1	30.34	1.34	2.10
23	1	-1	1	+0.33	39.7	43.99	2.25	2.33	36.0	53.83	3.40	2.87	22.9	40.67	2.26	2.24
24	1	-1	1	1	47.6	48.24	2.66	2.53	55.0	70.58	4.31	2.89	29.2	52.48	3.03	2.42
25	-1	-1	1	-1	14.6	13.50	0.40	0.97	20.2	20.28	0.69	1.24	7.0	4.51	0.13	0.95
26	-1	-1	1	-0.33	21.7	26.87	1.27	1.70	24.0	43.78	2.34	2.77	7.7	11.92	0.39	2.00
27	-1	-1	1	+0.33	36.4	37.36	1.74	2.01	30.5	59.06	3.62	2.92	12.3	16.80	0.68	2.47
28	-1	-1	1	1	42.8	60.19	3.17	2.60	45.6	76.89	4.57	3.03	17.6	23.82	0.87	2.62
29	1	-1	-1	-1	15.3	11.00	0.40	1.15	18.3	13.55	0.38	1.17	19.7	10.35	0.22	1.25
30	1	-1	-1	-0.33	27.7	29.66	1.45	2.66	25.1	32.79	1.55	2.37	19.3	26.47	1.37	1.66
31	1	-1	-1	+0.33	29.4	31.66	1.63	2.40	37.2	47.40	2.66	2.68	28.7	35.91	2.13	2.15
32	1	-1	-1	1	40.4	45.11	2.49	2.67	51.8	55.47	3.35	2.80	38.0	44.17	2.47	2.21
33	-1	-1	-1	-1	15.9	15.00	0.30	1.25	51.9	18.33	0.66	1.34	10.0	6.98	0.14	1.10
34	-1	-1	-1	-0.33	24.7	26.06	1.08	2.12	28.3	36.65	1.64	2.34	11.9	12.79	0.41	2.03
35	-1	-1	-1	+0.33	35.5	34.82	1.66	2.35	38.5	55.28	2.58	2.61	13.5	17.93	0.69	2.37
36	-1	-1	-1	1	37.9	44.51	2.04	2.46	59.4	68.88	3.63	2.80	18.0	24.82	0.89	2.52

37	0	1	0	-1	17.6	19.00	0.80	1.19	21.2	20.84	0.98	1.21	28.3	18.85	0.83	1.52
38	0	1	0	-0.33	29.0	32.58	1.75	2.33	30.5	50.56	2.69	2.77	27.6	31.15	2.12	2.32
39	0	1	0	+0.33	33.6	44.09	2.38	2.52	38.7	71.84	4.33	2.93	36.5	43.99	2.52	2.44
40	0	1	0	1	45.4	55.30	2.90	2.78	54.4	80.63	5.43	2.98	43.5	58.50	3.38	2.62
41	0	-1	0	-1	20.5	19.00	0.90	1.32	20.8	19.58	0.74	1.22	19.5	16.53	0.73	1.52
42	0	-1	0	-0.33	24.3	37.45	1.84	2.37	25.9	40.17	2.31	2.76	21.6	29.30	1.36	2.02
43	0	-1	0	+0.33	40.9	60.21	4.01	2.89	37.2	66.94	4.08	2.92	28.5	39.22	2.15	2.25
44	0	-1	0	1	44.6	70.28	4.19	2.88	44.5	76.54	5.04	2.93	35.0	48.93	2.71	2.39
45	0	0	1	-1	20.4	21.00	0.75	1.16	19.8	24.96	0.89	1.30	15.1	16.33	0.70	1.53
46	0	0	1	-0.33	26.0	32.71	1.77	2.37	29.4	49.88	2.93	2.82	16.9	35.76	1.74	2.27
47	0	0	1	+0.33	33.5	56.61	3.39	2.84	48.9	71.88	4.73	3.08	19.9	36.03	1.94	2.22
48	0	0	1	1	39.5	65.40	3.63	2.84	62.5	88.78	5.00	3.24	26.3	46.04	2.64	2.34
49	0	0	-1	-1	15.7	14.00	0.60	1.09	20.1	16.02	0.62	1.20	18.4	17.31	0.74	1.52
50	0	0	-1	-0.33	25.8	31.45	1.33	2.18	25.0	39.95	2.09	2.49	18.2	33.02	1.79	2.18
51	0	0	-1	+0.33	32.0	42.31	2.22	2.31	38.7	60.60	3.53	2.73	25.2	39.07	2.17	2.34
52	0	0	-1	1	51.5	55.51	3.25	2.44	58.3	79.37	4.68	2.79	31.9	53.60	2.85	2.46
53	1	0	0	-1	20.4	20.00	0.70	1.16	22.0	16.66	0.53	1.06	18.2	12.90	0.54	1.21
54	1	0	0	-0.33	27.9	35.66	1.96	2.34	30.6	44.18	2.58	2.52	22.2	20.67	0.89	1.55
55	1	0	0	+0.33	39.0	45.16	2.73	2.55	40.5	57.49	3.32	2.72	27.8	39.37	2.04	2.30
56	1	0	0	1	49.4	53.80	2.70	2.81	48.7	76.34	5.01	2.80	32.7	48.29	2.78	2.32
57	-1	0	0	-1	15.0	15.00	0.60	1.18	20.4	17.00	0.63	1.19	11.2	8.03	0.33	1.02
58	-1	0	0	-0.33	22.9	37.79	1.89	2.30	24.1	44.98	2.39	2.73	11.0	17.47	0.63	1.90
59	-1	0	0	+0.33	32.1	41.08	2.27	2.51	35.9	64.20	3.83	2.47	16.5	21.53	0.81	1.70
60	-1	0	0	1	49.6	51.72	3.31	2.78	44.3	87.69	5.05	2.77	22.7	27.07	1.20	1.87

X_T, X_t, X_C and X_R = normalized values of temperature, time and reagent concentration (soda, alkali active or ethanol) in the pulping, and number of PFI beating revolution, respectively. BeG = beating grade; TsI, BuI, and TeI = tensile, burst, and tear indices, respectively

Table 5. Polynomial Equations for the Dependent Variables in the Pulping and Refining from Orange Tree Wood Using Different Pulping Processes

Dependent Variable	a_0	a_1	a_2	a_3	a_4	a_{11}	a_{12}	a_{13}	a_{14}	a_{22}	a_{23}	a_{24}	a_{33}	a_{34}	a_{44}	R^2	F>	p<	t>
Soda-AQ pulping																			
YiP	40.56	-3.73	-1.95	-4.06	-	-	-	-	-	1.38	-	-	-	-	-	0.95	3.84	0.078	1.97
BeG	32.26	1.22	1.74	-	14.02	-	-	1.27	-	-	-2.02	-	-	-1.31	-	0.92	3.22	0.078	1.79
TsI	45.28	-	-	3.13	19.57	-5.66	-	1.60	-	-	-	-	-3.31	-	-2.86	0.92	3.35	0.073	1.83
TeI	2.55	-	-	-	0.70	-0.08	-0.07	-	-	-	0.06	-	-0.13	0.06	-0.50	0.93	2.41	0.127	1.55
Kraft-AQ pulping																			
YiP	45.13	-3.15	-1.92	-3.65	-	-	-	-	-	-	-	-	-	-	-	0.85	2.57	0.140	1.60
BeG	33.11	1.26	2.81	1.00	16.83	-	-	-	1.33	-	1.99	1.37	2.69	-	2.22	0.92	2.19	0.145	1.48
TsI	55.73	-3.87	2.59	4.98	27.89	-4.72	-	-	-1.52	-	2.02	-	-	2.64	-6.34	0.96	2.16	0.148	1.47
TeI	2.85	-	-	0.09	0.79	-0.11	-	-	0.06	-	-	-	-	0.06	-0.73	0.95	2.54	0.117	1.59
Ethanol pulping																			
YiP	45.55	-8.86	-1.76	3.40	-	1.87	-	-	-	-	-	-	-	-	-	0.97	3.91	0.076	1.98
BeG	25.28	5.78	3.26	-	7.03	-5.76	-	0.89	1.29	4.02	1.33	-	-4.56	-	3.74	0.97	2.91	0.094	1.71
TeI	2.25	-	-	-	0.50	-0.29	-	0.09	-	0.11	-	-0.06	-	-	-0.33	0.87	2.30	0.135	1.52

$$Y_e(\text{dependent variable}) = a_o + a_1X_T + a_2X_t + a_3X_c + a_4X_R + a_{11}{X_T}^2 + a_{12}X_TX_t + a_{13}X_TX_c + a_{14}X_TX_R + a_{22}{X_t}^2 + a_{23}X_tX_c + a_{24}X_tX_R + a_{33}{X_c}^2 + a_{34}X_cX_R + a_{44}{X_R}^2$$

X_T, X_t, X_C and X_R = normalized values of temperature, time and reagent concentration (soda, alkali active or ethanol) in the pulping, and number of PFI beating revolution, respectively. PiY = pulp yield; BeG = beating grade; TsI = tensile index; TeI = tear index

R^2, F, p and t = Statistical parameters of the equations

Table 6. Operational Variable Values in Orange Tree Wood Pulping and Refining to Obtain Optimal Values of Dependent Variables

Pulping Type	Dependent Variables	Maximum values of dependent variables	Normalized values of operational variables to obtain maximum values of the dependent variables			
			X_T	X_t	X_C	X_R
Soda-AQ	Pulp yield (%)	51.7	-1	-1	-1	-
	Beating grade (ºSR)	51.5	-1	1	-1	1
	Tensile I (Nm/g)	62.76	0.1	-	0.5	1
	Tear I (mNm^2/g)	2.83	0.43	0.1	0.4	0.72
Kraft-AQ	Pulp yield (%)	53.8	-1	-1	-1	-
	Beating grade (ºSR)	64.6	1	1	1	1
	Tensile I (Nm/g)	91.1	-0.57	1	1	1
	Tear I (mNm^2/g)	3.19	0.16	-	1	0.59
Ethanol	Pulp yield (%)	61.4	-1	-1	1	-
	Beating grade (ºSR)	45.7	0.63	1	0.21	1
	Tear I (mNm^2/g)	2.60	-0.15	-1	-1	0.85

X_T, X_t, X_C, and X_R =Normalized values of temperature, time, reagent concentration (soda, alkali active, and ethanol) and number of PFI beating revolutions, respectively

Table 7. Maximum Variation Values of the Dependent Variables Varying one of the Operational Variables in Orange Tree Wood Pulping and Refining

Pulping	Dependent	Variation in the dependent variables varying the following operational variables			
		T	*T*	*C*	*R*
Soda-AQ	Pulp yield (%)	7.46 (14.43%)	3.90 (7.55%)	8.12 (15.71%)	-
	Beating grade (ºSR)	0.10 (0.19%)	7.52 (14.60%)	8.80 (17.09%)	30.66 (59.53%)
	Tensile I (Nm/g)	6.49 (10.34%)	-	7.43 (11.84%)	39.15 (62.38%)
	Tear I (mNm^2/g)	0.16 (5.65%)	0.06 (2.12%)	0.31 (10.95%)	1.55 (54.77%)
Kraft-AQ	Pulp yield (%)	6.29 (11.69%)	3.83 (7.12%)	7.28 (13.53%)	-
	Beating grade (ºSR)	5.18 (8.02%)	12.44 (19.25%)	6.51 (10.08%)	39.06 (60.46%)
	Tensile I (Nm/g)	11.65 (12.80%)	9.22 (10.13%)	19.28 (21.18%)	62.79 (68.96%)
	Tear I (mNm^2/g)	0.15 (4.70%)	-	0.25 (7.84%)	1.84 (57.68%)
Ethanol	Pulp yield (%)	17.63 (28.69%)	3.51 (5.71%)	6.81 (11.08%)	-
	Beating grade (ºSR)	15.30 (33.49%)	8.33 (18.23%)	6.64 (14.53%)	15.68 (34.32%)
	Tear I (mNm^2/g)	0.38 (14.65%)	0.16 (6.15%)	0.02 (0.77%)	1.12 (43.08%)

T, *t*, *C* and *R* = temperature, time, reagent concentration (soda, active alkali, or ethanol), and number of PFI beating revolutions. Data in brackets are the percentages of these variations with respect to the optimum values of the dependent variables.

Table 8 shows the results of the simulation of the pulping and refining through the equations of Table 5. By use of these operating conditions acceptable values for the strength properties of the pulps were obtained, and at the same time they deviated very little from their maximum values (which are shown in Table 6). Moreover, there was only a small fall-off in the values of yield and beating grade. This mode of operation discards both mild and severe operating conditions. Mild operating conditions give rise to strength properties of the pulps that are too low, while severe operation conditions give rise to low pulp yield and excessive consumption of energy (operating at high temperature and number of PFI beating revolutions) and reagents (operating with high concentration), as well as a high capital assets for installation (operating for a high pulping time).

Table 8. Orange Tree Wood Pulping and Refining Simulation

Pulping	Operational conditions	Tensile index, Nm/g	Burst index, kN/g	Tear index, mNm^2/g	Pulp yield, %	Beating grade ºSR
Soda-AQ	170 ºC, 13% soda, 40 min, 2700 number of PFI beating revolutions	59.11 (5.82%)[1]	4.14[2] (1.19%)[3]	2.79 (1.41%)[1]	42.51 (17.78%)[1]	41.74 (18.95%)[1]
Kraft-AQ	170 ºC, 13% alkali, 65 min, 2700 number of PFI beating revolutions	73.98 (18.79%)[1]	3.84[4] (23.81%)[5]	2.97 (6.90%)[1]	45.13 (26.50%)[1]	47.99 (25.27%)[1]
Ethanol	185 ºC, 70% ethanol, 90 min, 2700 number of PFI beating revolutions	60.55[6] (4.33%)[7]	3.29[6] (4.91%)[7]	2.44 (6.15%)[1]	45.55 (25.82%)[1]	33.30 (27.13%)1

[1]: Percentage of deviation over the maximum value of table 6
[2]: Value extrapolated between rows 43 and 44 table 3
[3]: Percentage of deviation over the maximum value of table 3 (row 44)
[4]: Value extrapolated between rows 3 and 4 of table 3
[5]: Percentage of deviation over the maximum value of table 3 (row 12)
[6]: Value extrapolated between rows 3 and 4 of table 3
[7]: Percentage of deviation to the maximum value of table 3 (row 4)

The polynomial models obtained for the different dependent variables were similar to those previously reported for paper sheets from some types of agricultural and agro-industries residues: acetone, ethanol, and ethanol-acetone pulp from wheat straw (Jiménez *et al*. 2001, 2002 and 2004); kraft pulp from olive wood pruning (López *et al*. 2000, Díaz *et al*. 2005); and soda-anthraquinone pulp from empty fruit bunches of oil palm (Jiménez *et al*. 2009).

Table 9 shows the optimal results obtained in this work for orange tree wood pulps, as well as those of other studies for pulps beaten from wheat straw, olive wood, and empty fruit bunches (EFB) of oil palm.

Table 9. Comparison of Various Pulps Beaten from Non-Wood Raw Material

Type of Pulp	Yield (%)	Beating grade (ºSR)	Tensile I (Nm/g)	Burst I (kN/g)	Tear I (mNm^2/g)
Orange tree wood					
Soda-AQ	38.6	41.7	59.11	4.14	2.79
Kraft-AQ	45.1	48.0	73.98	3.84	2.97
Ethanol	45.6	33.3	60.55	3.29	2.44
Wheat straw acetone pulp (Jiménez *et al*. 2001)	46.5	29.5	49.94	2.63	3.35
Wheat straw ethanol pulp (Jiménez *et al*. 2004)	42.0	53.0	61.53	3.32	3.17
Wheat straw ethanol-acetone pulp (Jiménez *et al*. 2002)	53.2	13.3	45.70	1.81	3.34
Olive wood Kraft López *et al*. 2000	-	65.4	58.36	4.01	6.66
Olive wood Kraft Díaz *et al*. 2000	25.6	45.0	39.00	1.95	2.40
EFB of oil palm (Jiménez *et al*. 2009)	39.0	47.5	59.63	4.17	7.20

The data in Table 9 suggests that the values of the yield and beating grade of the pulps of orange tree pruning are intermediate to those of the non-wood materials considered. The values of the tensile and burst index notes are similar to those of the comparative materials, while the tear index values are lower. Finally, it can be concluded that orange tree wood produced beaten pulps with strength properties that can compete with and even surpass pulps of three singular raw materials pulped with organosolv, soda, and kraft processes: wheat straw, which is widely known and used in the world, olive wood, which contains a ligneous structure intermediate between hardwood and softwood, and EFB, which is a very abundant and very localized agrifood industry residue.

CONCLUSIONS

1 Orange tree wood has contents of holocellulose (73.2%), α-cellulose (48.0%) lignin (20.0%), extractives (3.6%), and ash (3.4%) of the same order as other non-wood and wood materials used for the production of pulp and paper, so it can be regarded as an alternative raw material.

2 For soda-AQ, kraft-AQ, and ethanol pulping of orange tree wood, polynomial equations were derived that reproduced yield, drainability, and strength properties with errors in most cases much less than 20%.

3 Kraft-AQ pulping can be regarded as the most favorable process because, when operated at 170 °C, 65 min, 13% alkali, and 2700 number PFI beating revolutions, values for the tensile index, burst index, and tear index of 73.98 Nm/g, 3.84 kN/g, and 2.97 mNm^2/g, respectively, were obtained, and these values are higher than those usually found for soda-AQ and ethanol pulping.

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