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Abstract: Lignocellulosic material from Leucaena leucocephala was subjected to a two-stage fractionation process to obtain a valorized effluent containing hemicellulose derivatives and a solid phase for producing cellulose pulp by conventional soda-anthraquinone delignifi¬cation.
The first autohydrolysis stage allows up to 46.6% of the initial hemicellulose in the raw material to be extracted as xylooligomers, xylose and furfural into the liquid phase.
Alkaline cooking of the solid residue from this stage with NaOH-AQ allows the production of cellulose pulp and paper sheets (161-177 ºC, 17-25 %, 60-120 min) with properties on a par with those obtained without a prior autohydrolysis stage (169-185 ºC, 21-29 %, 90-150 min) and by using milder delignification conditions; also, it provides a hemicellulose-containing liquid phase with added value.

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L. leucocephala was subjected to a two-stage fractionation process.

L. leucocephala was obtained a valorized liquor, containing hemicellulose derivatives

The properties of the pulp sheets were better obtained with prior autohydrolysis.

Biorefinery process for production of paper and oligomers from *L. leucocephala* K360 with or without prior autohydrolysis

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Abstract

Lignocellulosic material from *Leucaena leucocephala* was subjected to a two-stage fractionation process to obtain a valorized effluent containing hemicellulose derivatives and a solid phase for producing cellulose pulp by conventional soda–anthraquinone delignification.

The first autohydrolysis stage allows up to 46.6% of the initial hemicellulose in the raw material to be extracted as xylooligomers, xylose and furfural into the liquid phase.

Alkaline cooking of the solid residue from this stage with NaOH–AQ allows the production of cellulose pulp and paper sheets (161–177 ºC, 17-25 %, 60–120 min) with properties on a par with those obtained without a prior autohydrolysis stage (169-185 ºC, 21-29 %, 90-150 min) and by using milder delignification conditions; also, it provides a hemicellulose-containing liquid phase with added value.

Keywords

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1. INTRODUCTION

Lignocellulosic biomass (LCB) is considered to be a major source for 'green' chemicals, biofuels, and biobased products. Among the advantages of using LCB can be mentioned that it is abundantly available around the world, non competitive with food production, and it is a renewable and sustainable resource. Achieving more advantageous use of these natural renewable resources is a political goal in which societies are more and more immersed. This is especially the case when biomass derived products can become competitive with fossil oil derivatives in the short term. To meet this challenge, the biorefinery concept is receiving a renewed interest, with emphasis on using all the fractions present in plant biomass. (Ligero et al., 2011). Biorefining is the sustainable processing of biomass into spectrum of marketable products (food, feed, materials and chemicals) and energy (fuels, power and heat) (Huijgen et al., 2012).

The biorefinery or integral fractionation from lignocellulosic biomass can be achieved using various stages of hydrolysis and delignification. The development of wood autohydrolysis and acid-catalysed prehydrolysis dates back to the 1940s (Overbeck and Muller, 1942). In the field of cellulosic pulp and paper, by means of these processes, short-chain polysaccharides are removed from wood before the production of dissolving pulp (Rydholm, 1965).Today, autohydrolysis is seen as a potential stage to precede alkaline pulping for the production of both paper grade and dissolving-grade pulps. In conventional alkaline pulping, the dissolved hemicelluloses are mainly used as low calorific value fuel. Autohydrolysis applied before pulping yields low molecular weight xylooligosaccharides and monomeric xylose as well as substantial amounts of acetic acid, and these can be commercialized as value added products (Testova et al, 2010). The expanding range of xylooligosaccharides applications includes products for the food industry as novel sweeteners and prebiotics (Playne and Crittenden, 2004; Vazquez et al., 2006) and for the pharmaceutical industry (Abad-Romero et al., 2009).

Monomeric xylose and its dehydration product furfural (Zeitsch, 2000) can be converted to a variety of chemicals through chemical and biochemical pathways.

A raw material of high biomass production that can be used in these process of biorefinery is the genus Leucaena. Specifically *Leucaena leucocephala* is a leguminous tree, arises from the easy adaptability to Mediterranean ecological conditions (Rout et al., 1999, Ma et al., 2003), high biomass productivity and beneficial effects in the recovery of degrade soils (Vanlauwe et al., 1998, Goel and Behl, 2002). It has been described for various uses (production of bioethanol, conversion from the biomass crops to ethanol including sugar fermentation (Keffer et al., 2009). Pulping and papermaking of variety *Leucaena diversifolia* by soda-anthraquinone-ethanol was studied, in order to investigate the effects of cooking variables on properties of pulp and paper (López et al., 2010a, Feria et al., 2012a), etc. Leucaena genus is one of these crops with very high production of biomass and re-sprout capacity (more than 50 tons/ha/year, specially in annual crops, -Feria et al., 2011-).

For delignification of the material after autohydrolysis stages, alkaline or organic solvents stages could be used (Feria et al., 2012a, Romani et al., 2011). In this work an alkaline process with soda and antrhaquinone was used. It’s a complex heterogeneous reaction process. In such a multivariable system, the effect of a single parameter is not comparable unless all the other conditions are kept constant. Experimental designs provide effective tools with which to study the influences of different cooking parameters on pulping processes. Unlike the approach of one factor at a time, factorial designed experiments allow one to estimate the effects of several factors simultaneously (Tjeerdsma et al., 1994; Dong and Fricke, 1996). In this way, the hemicellulose fractions doesn´t have to be exposed to the relativity severe process conditions required for traditional delignification (Huijgen et al., 2012).

The aim of this work was the integral exploitation of *L. leucocephala*, a high-yield lignocellulosic material, by fractionation in two stages leading to the obtainment of a valorized liquid effluent containing abundant hemicellulose derivatives and a solid phase affording the production of cellulose pulp by conventional soda–anthraquinone delignification.

2. MATERIALS AND METHODS

2.1. Raw material.

A variety of *L. leucocephala K360* was obtained by in vitro replication that was harvested after seven years of growth in plantations used to experimental energy crops in Huelva (southwestern Spain). The productivity of *L. leucocephala K360* is 49.6 ± 10.67 ton/ha/yr of total dry weigh, harvested timber weighed 86 kg total mass (Feria et al., 2011), which was collected, reduced and homogenized in chips.

L. leucocephala K360 samples were milled to pass an 8 cm screen. The chips were reduced again to pieces between 2 and 10 mm long in order to prevent alterations of their components and removed the fines by sieving through 0.6 mm mesh. Samples were air-dried and stored in a dark and dry place until use.

2.2. Autohydrolysis of *L. leucocephala* wood. Pulping and sheets production

Wood chips, water, soda and anthraquinone were mixed in the desired proportions and reacted in a 10 L stainless steel, MK-systems Inc., reactor fitted with recirculation at a proportion of 8 g of water/g of oven-dry wood. The suspension was heated following the standard temperature profile (Feria et al., 2011) until reaching temperatures to 178 °C and 22.5 min operating time. The reactor was then closed and simultaneously heated and actuated to assure goodmixing and uniform swelling of Leucaena chips. When the autohydrolysis time had elapsed, the reactor was chilled to a temperature of 25 ºC.

The operating conditions were 161 ºC, 169 ºC and 177 ºC temperature; 60, 90 and 120 min operating time and 17%, 21% and 25% NaOH content using 0.1% anthraquinone content and a liquid/solid ratio of 8 kg water by kg raw material, on a dry basis. The reactor was then closed and simultaneously heated and actuated to assure good mixing and uniform pulping of Leucaena chips. Once the pulping time had elapsed, the reactor was chilled until 25 ºC. Following cooking, the pulp was separated from the liquor and disintegrated, without breaking the fibers, for 20 min in a high concentration pulper machine.

Pulp sheets were prepared with an ENJO-F-39.71 sheet machine according to the TAPPI T 205 sp-06 “Forming Handsheets for Physical Tests of Pulp”.

2.3.. Characterization and physical properties wood pulp fibres

The following properties: pulping yield (TAPPI T 257 cm-02 “Sampling and Preparing Wood for Analysis”), Kappa number (TAPPI T 236 om-06 “Kappa Number of Pulp”) and viscosity (TAPPI T 230 om-04 “Viscosity of Pulp”), were determined in *L. leucocephala* pulp.

From pulp sheets, grammage, and burst, tear and tensile indexes, Schopper-Riegler degree and ISO brightness were determined according to TAPPI T 220 sp-06, TAPPI T 403 om-10, TAPPI T 414 om-04, TAPPI T 494 om-06, ISO 5267/1, TAPPI T 525 om-06, respectively.

2.4. Experimental design for pulping process.

In order to relate the dependent (yield, kappa number, viscosity, Schooper Riegler degree, brightness, tensile, burst and tear index) and independent variables (temperature, time and soda concentration of process) in pulping process with the minimum possible number of experiment, a 2^n central composite factor experimental design was used, making it possible to construct a second-order polynomial in the independent variables and the identification of statistical significance in the variables was used. Independent variables were normalized by using the following equation.

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

Where X is the absolute value of the independent variable concern $\overline{X}$ is the average value of the variable, and X_{max} and X_{min} are its maximum and minimum values respectively. Temperature and operation time have the highest influence. The range of variation of independent variables was determinated according previous experiments (data not show) and other works (Alfaro et al., 2009, López et al., 2010a).

The number of tests required was calculated as $N = 2^n + 2\cdot n + n_c$, 2^n being the number of points constituting the factor design, $2\cdot n$ that of axial points, and n_c that of central points. Under our conditions, N = 10.

The experimental results were fitted to the following second-order polynomial:

$$Y = a_o + \sum_{i=1}^{n} b_i X_{ni} + \sum_{i=1}^{n} c_i X_{ni}^2 + \sum_{i=1;\, j=1}^{n} d_i X_{ni} X_{nj} \quad (i < j)$$

The independent variables used in the equations relating to both types of variables were those having a statistical significant coefficient (viz. those not exceeding a significance level of 0.05 in the student´s-test and having a 90% confidence interval excluding zero).

3. RESULTS AND DISCUSSION

3.1. Raw material characteristics and paper properties

Leucaena genus has been previously evaluated for cellulosic pulp production. Also has been chemically characterized (Bhola and Sharma, 1982; Majumer and Gosh, 1985, Jímenez et al., 2007; Malik et al., 2004). Also various varieties of leucaena genus have been characterized previously by authors of the present work (Feria et al., 2012b; Díaz et al., 2007; López et al., 2008, López et al., 2010b, López et al., 2011). Briefly, the majority fraction is cellulose (analyzed as glucan), at 37.2% (or 41.0 at TAPPI T 203-om-93), followed by the Klason lignin, at 24.1%, were higher than those for *Eucalyptus globulus* (Garrote et al., 2003) and hemicelluloses fraction (calculated as the sum of xylan, araban and acetyl groups) at 19.9%. This composition is comparable to other raw material such as *Eucalyptus globulus*. With regard to hemicelluloses, the predominant monomer was xylose, with a similar degree of substitution with acetyl groups as that of other lignocellulosic materials and a lesser degree of substitutes with arabinose. This composition is typical of O-acetyl glucuronoxylans, present in hardwoods (Tunc and Van Heiningen, 2008). The content of acetone extractive compounds in *L. leucocephala* was lower than that of eucalyptus wood (Feria et al., 2012b).

3.2. Autohydrolysis process

The operational conditions in an autohydrolysis process previous to delignification were selected in other work (Feria et al., 2011). In these conditions are ensured extraction levels hemicellulosic derivatives near the optimum values, while being controlled very significantly the degradation of cellulosic polymer. The selected conditions ensured the extraction of hemicelluloses derivatives in near-optimal yields while avoiding undue degradation of cellulose polymers. The solid phase yield of the autohydrolysis process was 77.7%, so 22.3% of the material was extracted into the liquid phase. This value differs by less than 5% from (21.3 %) that obtained by using the models Feria et al., 2011. Based on which, the liquid phase of the autohydrolysis presents a concentrations of glucose 2.42 %, xylose 6.10 %, arabinose 23.48 %, glucooligomers 14.51 %, xylooligomers 38.28 % and arabanoligomers 38.74 % respect initial glucan, xylan and araban in raw material (dry basis) respectively. Oligomer contents are given as monomer equivalents. Also, a concentration of furfural and 5-OH methyl furfural of 2.2 % and 0.47 % respect initial xylan and glucan in raw material (dry basis) respectively. The results differed by less than 10% from the experimental values for the liquid phase after autohydrolysis in this work: glucose, 2.27 %; xylose, 6.38 %, arabinose, 24.78 %, glucooligomers, 14.61 %, xylooligomers, 36.62 %, arabanoligomers 36.99 %, furfural 1.96 % and 5-OH methyl furfural 0.43 %.

3.3.Pulping process after autohydrolysis

In Table 1 can be observed results for total Pulp yield, Kappa number, Schooper Riegler degree, Viscosity, Brightness and physical properties of paper sheets (Tensile, Burst and Tear index) obtained by using a NaOH-AQ pulping process with prior autohydrolysis.

The operating conditions used were those of the proposed experimental design. Except for pulp yield, each result was the mean of at least 4 replications - or 12 for the physical properties of the paper sheets- Substituting the values of the independent variables for each dependent variable into the polynomial expression, the equations showed in Table 2 can be obtained. The differences between experimental and calculated results were less than 6% (Pulp yield, kappa number, Schooper Riegler degree, tensile index and brightness) or 10 % (Viscosity, burst and tear index).

Identifying the independent variables with the strongest and weakest influence on the dependent variables in equations in Table 2, is not easy since the former contain quadratic terms and other factors involving interactions between two independent variables. Temperature was the most influential linear variable in the development of strength properties of pulp sheets, kappa number, brightness, Shopper Riegler degree and viscosity. Pulp yield was affected by the linear variation of the all independent variables. Overall, temperature was the individual variable most strongly affecting kappa number and brightness, with a significant influence of the quadratic terms X_TX_T and X_tX_t, respectively, both with negative sign. Both terms limited the positive influence of the linear terms X_T and X_t on development of these two dependent variables. The significant value of the coefficient of X_CX_C makes it advisable to use medium active alkali concentrations for optimal kappa number and brightness values.

At low temperatures, kappa number and brightness differed markedly between the pulp samples obtained with and without autohydrolysis. Higher levels, however, led to better pulp properties with autohydrolysis than without it (Kappa number of 12.1 at 177ºC) than for cellulose pulp without prior autohydrolysis (Kappa number of 12.9 at 185 ºC) (Feria et al., 2012b). This values are similar than those reported by López et al. (2011) for *Leucaena diversifolia* ranging between 9.8 - 57.8 and better than the results of kappa number of cellulose pulp obtained from a kraft process (20.5 to 40.3) from *L. leucocephala* (Khristova et al., 1988) or results of Gillah and Ishengoma (1995) whose kappa number was of 28.

Temperature was also the individual variable most strongly influencing pulp viscosity, Shopper Riegler degree, and paper strength. The increased value of the coefficient for the term X_TX_T, with negative sign, makes it advisable to use medium values in the operating range for each variable as Temperature. At high delignification temperatures, the absence of a prior autohydrolysis stage gave pulp of poorer viscosity than its presence (292 cm^3/g) (Feria et al., 2012b) that use prior autohydrolysis (347 cm^3/g) this is suggestive of a strong influence of temperature on cellulose degradation –one exceeding that of the degrading effect of a two-stage process: autohydrolysis and alkaline delignification. The viscosities values are slightly lower than those obtained by López et al. (2011) for *L. diversifolia* ranging between 595-1504 cm^3/g.

The values of tensile, burst, and tear index, and brightness are similar than those obtained by López et al. (2010a) for *L. diversifolia* ranging between 6.2 - 18.7 Nm/g, 0.24 - 0.74 kPam^2/g, 0.64 - 1.06 mNm^2/g and 21.3 – 46.4 %ISO respectively and higher than those obtained by Jiménez et al. (2007) for *L. leucocephala* in a ethylenglycol process (Tensile index: 3.07 Nm/g, Tear index:, 0.24 mNm^2/g and Brightness: 37.56% ISO respectively).

Strength properties of paper sheets can be improved by applying a refined to cellulose pulp as made Gillah and Ishengoma (1993) between 18 ºSR (not beated) and 23.5 ºSR (2000 revolutions PFI). The results obtained by Jimenez et al. (2007) and Bhola and Sharma (1982) from pulps beated of *L. leucocephala* to maximum revolutions were similar in tensile index (between 53 N m/g and 84 N m/g). Also, the results obtained by Gillah and Ishengoma (1993) from pulps kraft of *L. leucocephala* grown in Morogoro (Tanzania) beaten in a PFI mill between 0 and 4000 revolutions show tensile index (42.7 to 106.7 N m/g) and tear index (6.7 to 8.4 mN m^2/g) values, these results are comparable to those of a reference material such as eucalyptus wood. In the work of Mutje et al. (2005) concerning refining a commercial eucalyptus kraft pulp to 26.5ºSR, values of tensile index: 76.7 N m/g and burst index: 7.8 kPa m^2/g were obtained. In the work of Gillah and Ishengoma (1993), values for tensile index of 84.1 N m/g and tear index of 11.8 N m^2/g for eucalyptus Kraft pulp (1000 revolutions PFI) were reported.

In order to better envisage the influence of operational variables on pulp and paper properties, and to compare the pulp sheets directly obtained from raw material and from solid phase with prior autohydrolysis, the surface responses in Fig. 2 were plotted. The space between two response surfaces represents the whole range of values for each dependent variable that was used at two extreme values, +1 and −1, of operation temperature (the most influential variable). Plotting the results for both pulps (with and without prior autohydrolysis) together in the same figure displays the overlap between the spaces and allows one to identify the particular operating conditions affording the production of cellulose pulp sheets with a given value of some property from raw material and for solid phase after an autohydrolysis process. As a rule, and for variation ranges applied for both process, the soda–anthraquinone delignification of the *L. leucocephala* with and without prior autohydrolysis resulted in similar paper properties. However, there were wide ranges of overlap for the dependent variables where the pulping conditions required for the solid phase from autohydrolysis were much milder than those for the original material. The economy inherent in using a lower temperature, time or alkali concentration is thus supplemented by the production of valorized liquor containing abundant sugars and oligomers.

For example (Fig.2), the response surface for pulp yield, kappa number and brightness (not shown) contained a wide range of identical values –and even some that were better in pulp subjected to no autohydrolysis– between the two planes corresponding to the highest temperatures; also, pulp yields were even better at medium operating times.

The response surface for viscosity, and that for SRº (not shown), exhibited a wide range of identical values for the processes with and without autohydrolysis. The physical properties of the paper sheets obtained from pulp subjected to autohydrolysis at a high temperature fell in between the maximum and minimum values for pulp without autohydrolysis over wide ranges of the operating conditions. Some properties such as tear index were even better in the pulp samples obtained by NaOH–AQ delignification after autohydrolysis.

For example, the experimental conditions represented by point Temperature: +1, Operation time: +1 and Alkali active concentration: 0. The properties of the cellulose pulp and paper sheets obtained with conventional delignification and prior autohydrolysis in the experimental design were as follows: Pulp Yield: 34.0 % vs 27.6 %, Kappa number: 15.2 vs 14.5, Viscosity: 317.1 cm^3/g vs 469.4 cm^3/g, Brightness: 31.6 % ISO vs 39.3 % ISO, Tensile index: 10.5 N m/g vs 7.8 N m/g, Burst index: 0.29 MPa m^2/kg vs 0.43 MPa m^2/kg and Tear index: 1.1 mN m^2/g vs 1.74 mN m^2/g. Since the operating conditions for the two processes were Temperature: 185 ºC and 177 ºC, Operation time: 150 minutes and 120 minutes and Alkali active concentration: 25 % and 21 %, the two-stage process affords the obtainment of acceptable paper with the added advantages of giving a valorized liquid phase containing hemicellulose derivatives and allowing mild operating conditions in relation to conventional delignification without autohydrolysis to be used.

4. CONCLUSIONS

L. leucocephala has proved a suitable raw material for biorefining in two stages: autohydrolysis and alkaline delignification. The first autohydrolysis stage allows up to 46.6% of the initial hemicellulose in the raw material to be extracted as xylooligomers, xylose and furfural into the liquid phase.

Alkaline cooking of the solid residue from this stage with NaOH–AQ allows the production of cellulose pulp and paper sheets with properties on a par with those obtained without a prior autohydrolysis stage and by using milder delignification conditions; also, it provides a hemicellulose-containing liquid phase with added value.

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Table 1. *Normalized values of independent variables and physical characterization of pulps obtained in the pulping process using the experimental design with autohydrolysis.*

Normalized values of independent variables: X_T	**X_t**	**X_C**	**Yield, %**	**Schooper Riegler degree, ºSR**	**Kappa number**	**Viscosity cm^3/g**	**Brightness, %**	**Tensile index, N m/g**	**Burst index, MPa·m^2/kg**	**Tear index, mN m^2/g**
0	0	0	38.4	15.0	39.5	679	22.7	5,89	0.19	1,13
0	0	0	38.0	15.0	40.1	680	22.8	5,93	0.18	1,14
-1	-1	-1	44.8	6.5	57.2	299	9.0	4,30	0.03	0.99
-1	-1	+1	40.6	12.5	47.9	730	20.0	3,22	0.06	0,73
-1	0	0	36.8	13.0	43.5	563	19.3	2,12	0.05	0,41
-1	+1	-1	40.5	9.5	55.7	518	12.4	2,77	0.05	0,55
-1	+1	+1	37.1	16.0	41.7	711	18.8	3,02	0.08	0,61
0	-1	0	43.8	14.5	44.1	693	20.4	6,63	0.16	1,45
0	0	-1	42.5	12.5	51.0	616	15.8	5,92	0.12	1,28
0	0	+1	38.6	15.0	37.8	763	26.5	5,86	0.21	1,04
0	+1	0	35.2	16.5	40.1	686	23.7	6,50	0.25	1,37
+1	-1	-1	44.0	16.0	28.1	645	21.3	7,20	0.16	1,78
+1	-1	+1	40.6	15.5	20.1	593	32.2	6,20	0.21	1,19
+1	0	0	31.9	17.5	12.1	512	36.8	6,30	0.27	1,23
+1	+1	-1	31.5	17.0	22.9	573	33.8	6,88	0.26	1,60
+1	+1	+1	29.1	17.5	15.2	347	39.9	8,93	0.53	1,94

Table 2. Equation obtained for each dependent variable of pulping process with and without autohydrolysis.

		Equation	**R^2**	**F-Snedecor**
With autohydrolysis	1	$\mathbf{Y_{YI}} = 38.06 – 2.27\ X_T – 4.04\ X_t – 1.93\ X_C – 3.63\ X_TX_T + 1.52\ X_tX_t + 2.57\ X_CX_C – 2.03\ X_TX_t$	0.989	98
	2	$\mathbf{Y_{Ka}} = 39.14 – 14.76\ X_T – 2.18\ X_t – 5.42\ X_C – 11.01\ X_TX_T + 3.29\ X_tX_t + 4.59\ X_CX_C$	0.985	74
	3	$\mathbf{Y_{vis}} = 686.2 - 15.2\ X_T + 49.4\ X_C – 137\ X_TX_T – 64.6\ X_TX_t – 112.7\ X_TX_C – 51.7\ X_tX_C$	0.983	85
	4	$\mathbf{Y_{SR}} = 15.25 + 2.6X_T + 1.15\ X_t + 1.5\ X_C – 1.45\ X_CX_C - 0.44\ X_TX_t – 1.56\ X_TX_C$	0.986	182
	5	$\mathbf{Y_{BR}} = 23.39 + 8.45\ X_T + 2.57\ X_t + 4.51\ X_C + 4.34\ X_TX_T – 1.66\ X_tX_t - 2.56\ X_CX_C + 2.25\ X_TX_t – 1.18\ X_tX_C$	0.995	170
	6	$\mathbf{Y_{TI}} = 5.82 + 2.01\ X_T – 1.45\ X_TX_T + 0.91\ X_tX_t + 0.52\ X_TX_t + 0.24\ X_TX_C + 0.55\ X_tX_C$	0.988	128.6
	7	$\mathbf{Y_{BI}} = 0.18 + 0.12\ X_T + 0.05\ X_t + 0.05\ X_C + 0.05\ X_TX_t + 0.03\ X_TX_C + 0.03\ X_tX_C$	0.956	36
	8	$\mathbf{Y_{TeI}} = 1.13 + 0.44\ X_T – 0.07\ X_C – 0.28\ X_TX_T + 0.31\ X_tX_t + 0.14\ X_TX_t + 0.16\ X_tX_C$	0.988	62
Without autohydrolysis [23]	9	$\mathbf{Y_{YI}} = 40.98 – 2.53\ X_T – 1.99\ X_t – 4.46\ X_C + 2.94\ X_CX_C – 2.49\ X_TX_t + 0.89\ X_tX_C$	0.981	76
	10	$\mathbf{Y_{Ka}} = 17.33 - 2.21\ X_T – 1.85\ X_C + 1.20\ X_TX_T – 1.12\ X_tX_t + 1.26\ X_CX_C – 1.06\ X_TX_C – 0.79\ X_tX_C$	0.985	74
	11	$\mathbf{Y_{vis}} = 607.2 - 123.2\ X_T – 45.9\ X_C – 103.1\ X_tX_t + 86.7\ X_CX_C – 63.8\ X_TX_t – 59.5\ X_TX_C – 28.3\ X_tX_C$	0.982	62
	12	$\mathbf{Y_{SR}} = 20 + 1.85\ X_T + 1\ X_t + 1.05\ X_C – 2.85\ X_TX_T$	0.980	133
	13	$\mathbf{Y_{BR}} = 33.49 + 0.97\ X_T + 2.72\ X_C – 1.23\ X_TX_T – 0.68\ X_tX_t – 1.38\ X_CX_C – 0.45\ X_tX_C – 0.98\ X_TX_t$	0.996	291
	14	$\mathbf{Y_{TI}} = 13.42 + 3.51\ X_T + 1.38\ X_C – 2.71\ X_TX_T – 2.35\ X_tX_t + 0.72\ X_TX_C – 1.37\ X_TX_t$	0.992	182
	15	$\mathbf{Y_{BI}} = 0.41 + 0.14\ X_T + 0.08\ X_C – 0.19\ X_TX_T – 0.04\ X_tX_t + 0.1\ X_CX_C – 0.03\ X_TX_t$	0.986	81
	16	$\mathbf{Y_{TeI}} = 1.32 + 0.27\ X_T + 0.1\ X_t + 0.09\ X_C – 0.22\ X_TX_T – 0.37\ X_tX_t + 0.09\ X_CX_C + 0.08\ X_TX_C – 0.06\ X_tX_C$	0.997	141

Where: Y_{YI} denotes solid yield (%), Y_{ka} Kappa number; Y_{Acet}, acetone extractives in pulp; Y_{vis}, viscosity; $Y_{SR,}$ Schooper Riegler degree; Y_{BR}, Brightness; $Y_{TI,}$ Tensile index; Y_{BI}, Burst index and Y_{TeI}, Tear index. X_T, X_t and X_C denote normalized pulping temperature, time and soda concentration, respectively.

Figure

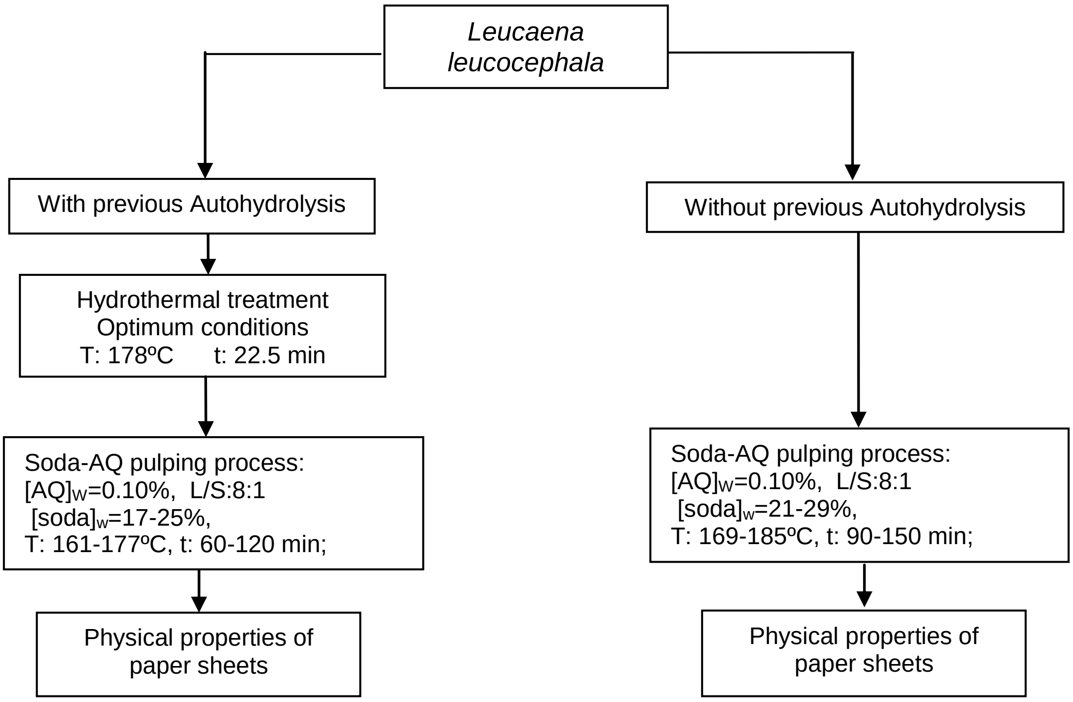

Fig. 1. Scheme of experimental work. Abbreviations: [soda]$_w$: soda concentration by weight; [AQl]$_w$: concentration of anthraquinone by weight; T: process temperature (ºC); t: processing time (min). L/S: liquid/solid ratio.

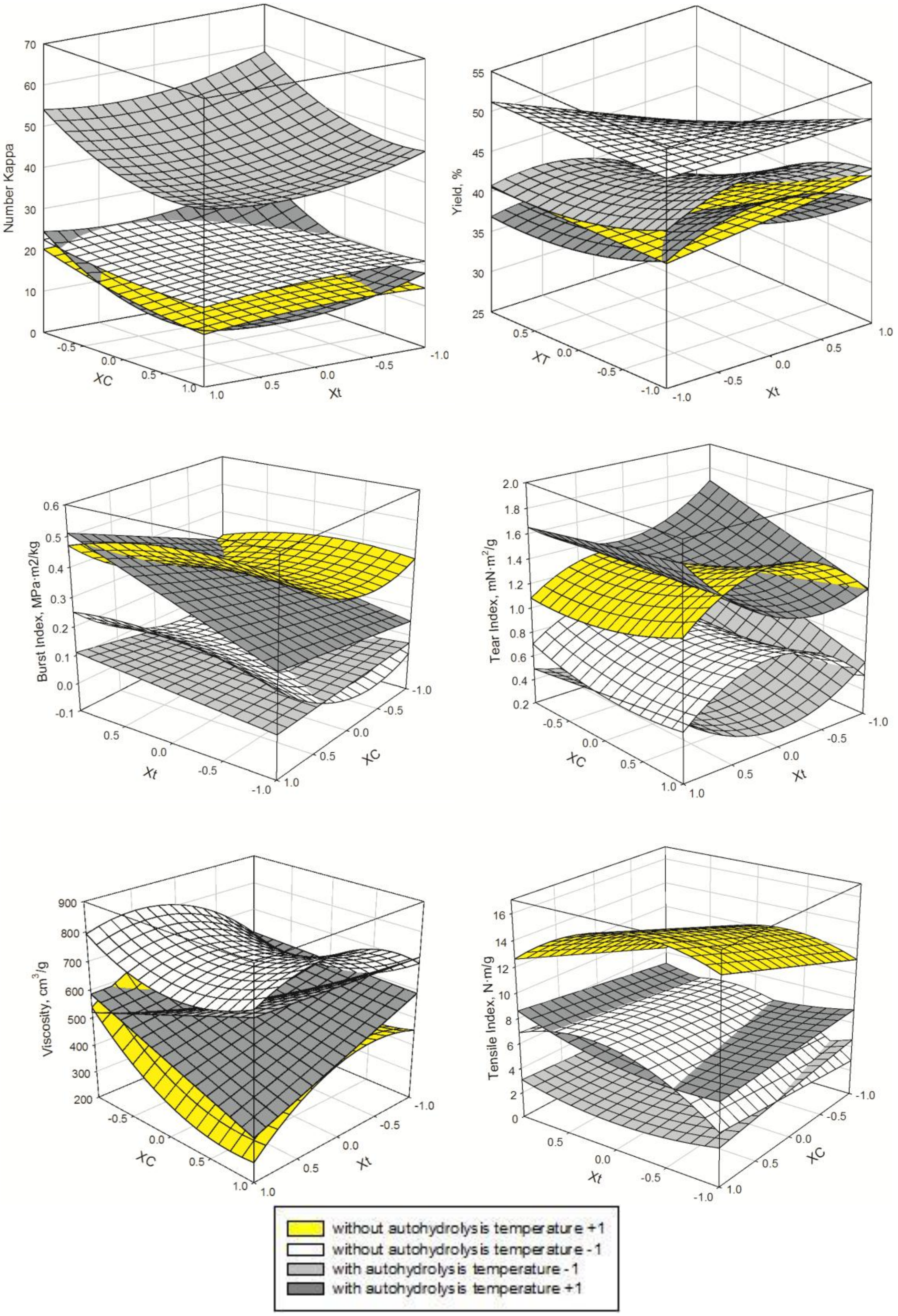

Fig. 2. Variations of properties as a function of independent variables of pulping process.