Paulownia as a raw material for the production of pulp by soda–anthraquinone cooking with or without previous autohydrolysis

Short Title: Pulp and paper from Paulownia with or withont previous autohydrolysis

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ABSTRACT

BACKGROUND: A central composite experimental design and multiple regresion were used to model the production of pulp and paper sheets from a *Paulownia fortunei* × *tormentosa* × *elongata* trihybrid clone. Also, the same experiments were carried out with the solid phase after autohydrolysis of the raw material.

RESULTS: The results were compared with those obtained from the solid phase remaining after autohydrolysis of the raw material with a view to optimizing the use of hemicelluloses and cellulose by fractionation. Pulp and paper sheets from trihybrid clone surpass those from other Paulownia species such as *P. fortunei* and *P. elongata* in physical properties such as tensile index (11.6–49.1Nm/g), burst index (0.23–2.41MPa·m^2/kg), tear index (0.71–3.03mN·m^2/g) and intrinsic viscosity (427–958 cm^3/g).

CONCLUSIONS: The solid phase obtained by autohydrolysis of the Paulownia trihybrid provides pulp and paper sheets comparable in strength-related properties to those of pulp directly produced from the raw material, but using substantially milder operating conditions and with the added advantage that the autohydrolysis treatment provides highly valorizable liquor.

Keywords: Paulownia; autohydrolysis; soda-AQ pulp; paper.

INTRODUCTION

The genus Paulownia originated in China, where it has been a traditional tree for more than two thousand years. Because it has modest water requirements, Paulownia has aroused interest as an industrial raw material despite its difficult growth in marginal areas [1,2]. The genus encompasses nine different species most of which exhibit very fast growth and can be harvested only 15 years after planting to obtain products with a substantial added value [3]. Paulownia plants possess a high biomass production and resprouting potential: up to 50 tons/ha/yr, which is among the highest reported figures (especially in relation to annual crops) [4]. Also, they exhibit fast growth and can produce as much biomass in one year as other species in several [5]. Under favourable conditions, an intensive plantation of 2000 trees/ha can yield up to 150–300 tons of wood per year only 5–7 years after planting [6]. As shown in this work, such a high biomass production can be further increased by using hybrids of some varieties. Thus, the *Paulownia fortunei–Paulownia tormentosa* clone used by Ayan *et al.* (2006)[7] grew up to 76.2 cm within one year after direct seeding.

The genus Paulownia has lately been introduced and naturalized in a number of countries including the USA, and been a subject of study as regards adaptation to soil and survival of diverse varieties in some areas [7-9].

This work is novel because there are few references to industrial uses of Paulownia. There are, however, a number of agronomic, genetic and health-related studies about this genus. Worth special note among them are those suggesting the potential advantages of its use in energy crops by virtue of its favourable energy input/output ratio and biomass production [10,11]. Others suggested uses for Paulownia included veneer or plywood, furniture, handicrafts, tools, musical instruments, particleboard, charcoal and there also various attempts to generate energy from paulownia chips [3, 12-14]. One of them is its use as source for pulp. The most suitable variety of Paulownia for this purpose is *Paulownia Fortunei* [15], characterized by a fast development and an uniform and regular growth [12]. Also, this plant could be susceptible to produce xylooligomers under a hydrolytic process, as well as provide a low degraded lignocellulosic residue to the pulping process, which is justified by some Paulownia characteristics, such as its growth and physicochemical properties [16,17].

Ethanol for delignification of *Paulownia fortunei* was used by these last authors after extraction of sugars and oligosaccharides by autohydrolysis. Kraft pulp with kappa number of 25, tensile index of 106 Nm/g, burst index of 6.47 kPa m^2/g and tear index of 2.67 mN m^2/g was obtained by Rai et al (2000). Kraft-AQ, soda-AQ and ethanol process were studied on *Paulownia fortunei* by Ates et al. (2008) [18]. Kappa numbers between 27.8 and 42.1 and breaking length between 1.53 and 2.59 km (15.3 – 25.9 Nm/g approximately) were obtained.

Paulownia fortunei has an average fiber length of 1.42 mm [15]. Other authors [1,2,18] offer values of 0.94 mm for *Paulownia fortunei*, 0.82 mm for *Paulownia elongate*, 0.84 for *Paulownia tormentosa* and average values for 1.12 mm for other 7 species of Paulownia, similar to those found for Eucalyptus wood, whose typical values are 0.7-1.3 mm.

In this work, we used a central composite experimental design in combination with multiple linear regression to model the production of cellulose pulp and paper sheets from a *Paulownia fortunei* × *tormentosa* × *elongata* trihybrid clone. Also, the same experiments were carried out with the solid phase after autohydrolysis of the raw material. Both results were compared. In this way the optimization of use of hemicelluloses and cellulose by fractionation can be discussed.

MATERIAL AND METHODS

Raw material. Provision and characterization

A trihybrid variety of Paulownia (*Elongata x Fortunei x Tormentosa*) clone obtained by in vitro replication was used for field experiences. The material was harvested after three years of growth in plantations used to exploit biomass crops in Extremadura (southwestern Spain). The material was supplied by the firm Vicedex Europa. Paulownia wood trimming samples were milled to pass an 5-mm screen, because no diffusional limitations were observed for this particle size in the preliminary studies. Samples were air-dried, homogenized in a single lot (to avoid differences in composition among aliquots) and stored in a dry site.

Aliquots from the homogenized wood were subjected to moisture determination (drying at 105 ºC to constant weight) and quantitative acid hydrolysis with 5 mL of 72% sulfuric acid for an hour (Tappi T248 em-85). Before HPLC analysis, the solid residue from posthydrolysis was recovered by filtration and considered as Klason lignin. The monosaccharides and acetic acid contained in hydrolysates were determined by HPLC in order to estimate (after corrections for stoichiometry and sugar decomposition) the contents in cellulose (as glucan), hemicelluloses (xylan + arabinan), and acetyl groups. Chromatographic determination was performed using an Agilent 1100 HPLC equipped with an ion-exchange resin BioRad Aminex HPX-87H column under the following conditions: mobile phase, 0.005 mol·L-1 of sulphuric acid; flow rate, 0.6 mL·min^{-1}; and column temperature, 50 °C. The volume injected was 20 μL. The holocellulose and extractives compounds were determined according the method of Wise et al.(1946)[20] and TAPPI 204 cm-07 standard respectively.

Autohydrolysis process, Pulping procedure and formation of paper sheets

Wood chips and water were mixed in the desired proportions and reacted in a 10 L stainless steel MK-systems Inc. reactor fitted with recirculation. The operating conditions were temperature of 187° C and operating time of 15 minutes.

The reactor was then closed and simultaneously heated and actuated to assure good mixing and uniform swelling of Paulownia chips. When the autohydrolysis time elapsed, the reactor was chilled to a temperature of 25ºC.

Pulping and paper sheets formation

Cellulose pulps (from solid phase after autohydrolysis) were obtained in the same reactor used in the hydrothermal pretreatment. Also, cellulose pulps were obtained for raw materials without hydrothermal pre-treatment.

The reactor was then closed and simultaneously heated and actuated to assure good mixing and uniform swelling of Paulownia chips. When the pulping time elapsed, the reactor was chilled to a temperature of 25ºC.

Following cooking, the pulp was separated from the liquor and disintegrated, without breaking the fibers, during 3 min. Pulping yield, Kappa number and intrinsic viscosity were determinated according Tappi standard procedures 257, 236 and 230, respectively.

Paper sheets were prepared with an ENJO-F-39.71 sheet machine according to the Tappi 205 sp-95 standard. From paper sheets, grammage (T 220 sp-96), burst index (Tappi T 403 om-97), tear index (Tappi 414 om-98), tensile index (Tappi 494 om-96) and brightness (Tappi 525 om-92) were determined.

Experimental Design for Pulping Conditions. Multiple Regresion Model

To be able to relate the dependent variables (yield, kappa number; intrinsic viscosity, brightness, tensile index, burst index, and tear index) with independent variables (NaOH concentration, temperature and time of process) in pulping process with the minimum testing, a 2n central composite factor experimental design, that enabled construction of second-order polynomial in the independent variables and the identification of statistical significance in the dependent 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.

Three levels of alkali concentration (17, 20 and 23 % dry wt. basis), temperature (163, 171 and 179 ºC) and operation time (90, 120 and 150 min) were used for the raw material without pretreatment. For the solid phase after autohydrolysis process the next operation conditions were used: alkali concentration: 14, 17 and 20 % dry wt. basis, temperature: 155, 163 and 171 ºC and operation time: 60, 90 and 120 min. A liquid/solid ratio of 10/1 and anthraquinone concentration of 0.1 (dry wt. basis) were used in all experiments.

The number of tests required was calculated as $N = 2^n + 2*n + n_c$; 2^n being the number of points constituting the factor design, 2*n that of axial points and n_c that of central points. Under our conditions, N = 16.

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 95% confidence interval excluding zero).

RESULTS AND DISCUSSION

Raw material characteristics

Table 1 summarizes the chemical composition of *Paulownia fortunei*, *P. tormentosa*, the trihybrid used in this work (*Paulownia fortunei* × *tormentosa* × *elongata*) and various other materials including *Eucalyptus globulus* as reference for woody species.

The content of extractives compounds in *Paulownia fortunei x tormentosa x elongata* is 4.66%, higher than those found for Eucalyptus wood, with a value of 2.09% but lower that the results (5.46%) reported by other authors[1, 16, 17]. These compounds could cause problems related to pitch. Pitch deposits in the manufacturing of pulp, represent a complex phenomenon which has increased in recent years [25] and could cause problems by adhering to machinery and reducing the quality of pulp [26]. The cellulose content in Paulownia trihibrid [19], is 6.0%-17.6% (expressed as glucan), lower than those found for *Eucalyptus globulus*, but higher (22.2%-14.9%) that those found for *Paulownia Fortunei*, whereas lignin content is 17.6%-28.2%, higher than Eucalyptus's one. The *Paulownia hybrid fortunei x tormentose x elongate*, used in this work, was harvested after 3 years of growth and produced more than 50 tons of biomass per hectare per year [27]. Its content in acetyl groups was the highest among the raw materials in Table 1, which is suggestive of an increased amenability to autohydrolysis –in fact; the acetyl/xylan ratio was 25% higher than in eucalyptus wood-.

The holocellulose content was similar to that of eucalyptus and lower than that of *Paulownia fortunei* and *Paulownia elongata*. These previously reported values, however, were determined with the method of Wise et al. (1946) [20] and for Paulownia hibrid as a combination of monomers and oligomers. Lignin values are slight higher and xylan, arabinan and acetyl groups values are similar than that found for other materials.

NaOH-antraquinone pulping process of *Paulownia fortunei x tormentose x elongate* and solid phase after autohydrolysis

Paulownia can be considered as promising plantation grown pulp wood [15,18]. In this work, the raw material (a trihybrid variety: *Paulownia fortunei x tormentosa x elongate* -RM-) and the solid fraction from autohydrolysis (SF) were delignified by using a NaOH-antraquinone pulping process and the process was modelized and optimized using a linear multiple regression polynomial model and a factorial experimental design. Tables 2 and 3 show the results for the experimental points proposed in the experimental design for RM and SF.

Variations with the soda concentration, temperature and time process of the different properties of pulp and paper sheets (yield, kappa number, intrinsic viscosity, brightness, tensile index, burst index and tear index), are shown. The results were modelled by using the above-described multiple regression methodology. The ensuing models are shown in Table 4.

As can be seen from Tables 2 and 3, the yields in cellulose pulp obtained without and with autohydrolysis of the raw material (RM), respectively, were very similar. However, the kappa number for the pulp samples obtained directly from RM was smaller than those for SF; thus, the former ranged from 9.0 to 30.2 and the latter from 12.2 to 69.1, which suggests easier bleaching of RM pulp. Also, brightness in RM pulp was higher than in SF pulp (35.0% in the low end and 25.8% in the high end of the range). This was not a result of less efficient delignification of SF, but rather of a higher relative lignin content by effect of the removal of a substantial fraction of raw material (sugars and oligomers). The brightness values predicted by eq. 4 ranged from 27.1 to 38.0 % ISO and are suggestive of effective delignification of the raw material with the proposed soda–anthraquinone process. Intrinsic viscosity was also higher as a whole in RM pulp than in SF pulp.

Table 2 show the tensile index, burst index and tear index for the paper sheets obtained by soda–anthraquinone cooking of RM; all were high for unbeated pulp. Substituting the data of Table 2 into eqs 5–7 provided values spanning the range 11.62–49.14 mN m^2/g for tensile index, 0.23–2.41 MPa m^2/kg for burst index and

0.71–3.05 mN m^2/g for tear index. These values are quite similar to those for reference woody species such as eucalyptus [28].

Temperature was the most influential variable on yield, kappa number and intrinsic viscosity of RM pulp (eqs 1, 2 and 3 in Table 4), as so was the active alkali concentration on brightness (eq. 4 in Table 5), which was also the most influential variable for brightness in SF pulp (eq. 11 in Table 4). On the other hand, processing time and, to a lesser extent, temperature, were the two variables most markedly affecting the physical properties of the paper sheets. This is apparent from models 5–7 in Table 4 and contradicts the results for SF paper (eqs 12–14 in Table 4). For SF, the active alkali concentration was the individual variable most strongly influencing strength-related properties in pulp and paper sheets. Overall, these results can be ascribed to substantial destructuring of fibres in the raw material by effect of the autohydrolysis treatment, which essentially affects the operation time or temperature to be used in the subsequent, delignification step to facilitate access of the reagents to the cellulose matrix [29,30].

For comparison, in Table 5 show our results with and without autohydrolysis (Tables 2 and 3), and those of other authors for cellulose pulp and paper obtained from diverse Paulownia varieties. The four right-most columns correspond to the pulping of RM and SF for the trihybrid clone studied here. In previous work (results not shown), we modelled and optimized the extraction of sugars and oligomers from the liquor of an autohydrolysis process providing a residual solid phase with a good glucan content. The process was conducted under an isothermal regime, using a temperature of 187 °C for 15 min. This provided a solid phase retaining most of the initial glucan and a highly valorizable liquor containing 11.5% of the initial raw material as xylose and oligomers.

A comparison of the data in Tables 2 and 3 with those of Table 5 reveals that applying the soda–anthraquinone process to the *Paulownia fortunei* × *tormentosa* × *elongata* trihybrid clone results in substantially improved physical properties for the paper sheets in relation to *P. fortunei* and other Paulownia species. As can be seen from Table 5, the best results were those reported by Rai *et al.* (2000) [15], which, however, were obtained with strong beating of the cellulose pulp.

In order to better envisage the influence of operational variables on pulp and paper properties, and to compare the pulp and paper sheets directly obtained from RM and SF, we constructed the hyperspaces of variation of the dependent variables via the response surfaces of Figs 1–3. The space between two response surfaces represents the whole range of values for each dependent variable –we used two extreme values, +1 and –1, for the most influential variable in each case. Plotting the results for RM and SF pulp together in the same figure marked overlap between the spaces and allowed us to identify the particular operating conditions affording the production of cellulose pulp or paper sheets with a given value of some property from RM or SF. Obviously, such conditions would differ. As a rule, the soda–anthraquinone delignification of the *Paulownia fortunei* × *tormentosa* × *elongata* trihybrid clone (RM) resulted in better 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 obtainment of a highly valorizable liquor containing abundant sugars and oligomers [19].

For example, (figure 1) the space between the two responses surfaces shaded in the lighter tone corresponds to cellulose pulp obtained from SF. The upper surface was obtained at an active alkali concentration of 20% (+1) (o.d.b.) and the lower one at 14% (–1). The space between the two response surfaces shaded in the darker tone corresponds to pulp obtained from RM; the upper surface at an operating time of 150 m (+1) and the lower one at 90 min (–1). Roughly, the whole space between the surface for RM pulp at time –1 and that for SF pulp at an active alkali concentration of +1 represents the tensile index values for the paper sheets one can obtain under much milder operating conditions by using SF instead of RM. For example, based on eqs 5 and 12 in Table 4, the highest tensile index for paper sheets from SF would be 34.34 N m/g and obtained by using an active alkali concentration of 20% (+1), a temperature of 164.6 °C (+0.2) and a processing time of 120 min (+1). Obtaining a comparable tensile index (37.30 N m/g) for paper sheets from RM entails using an active alkali concentration of 21.8% (+0.6), a temperature of 177.4 °C (+0.8) and a time of 144 min (+0.8). Therefore, RM requires an 8.3% higher alkali concentration, a 7.2% higher temperature and a 16.7% longer time.

A similar effect can be derived from the response surfaces for burst index (Fig. 2), which in addition, spans a similar range for RM and SF. The burst index for SF paper sheets peaks at 2.2 mN m^2/g (eq. 14 in Table 4), which is obtained at an active alkali concentration of 20% (+1), a temperature of 169.4 °C (+0.8) and a time of 60 min (–1). Obtaining a comparable tear index for RM sheets entails using an active alkali concentration of 23.0% (+1), a temperature of 174.8 °C (+0.6) and a time of

144 min (+0.8), which are 13.0%, 3.1% and 58.3% higher than the values for SF. As can be seen from Fig. 3, the effect of the operating conditions on tear index, yield, intrinsic viscosity and kappa number is less marked; again, however, RM and SF share a wide range of values that allow the use of milder conditions with SF. In fact, only brightness (models 4 and 11 in Table 4) failed to overlap between the two materials.

CONCLUSIONS

The *Paulownia fortunei*×*tormentosa*×*elongata* trihybrid clone has a glucan content slightly higher than that of eucalyptus but exceeding those of other Paulownia species. This, together with its high content in acetyl groups, makes it a suitable material for the extraction of hemicellulose derivatives by autohydrolysis.

Pulp and paper sheets from this trihybrid surpass those from other Paulownia species Solid phase obtained by autohydrolysis of the Paulownia trihybrid provides paper sheets comparable in strength-related properties to those of pulp and paper directly produced from the raw material, but using substantially milder operating conditions and with the added advantage that the autohydrolysis treatment provides a highly valorizable liquor.

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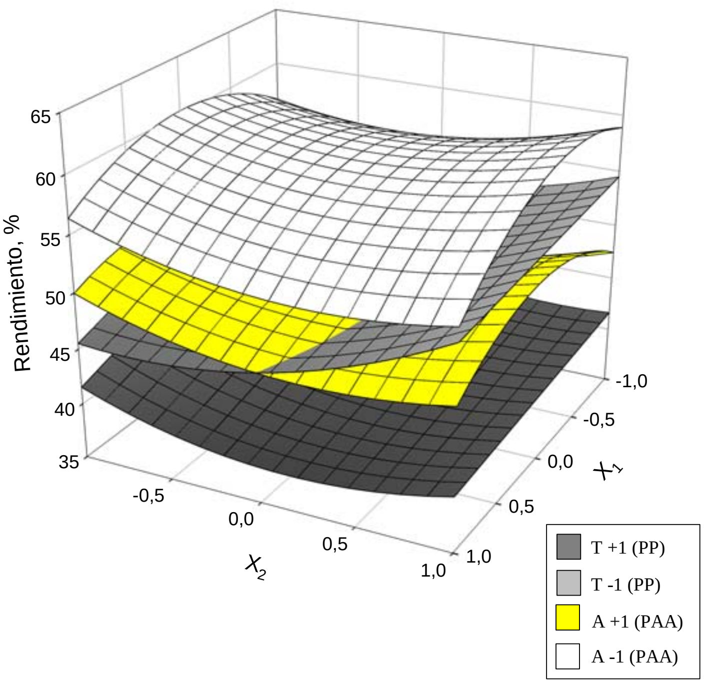
GRÁFICAS DE PASTA DE PAULOWNIA CON NaOH – AQ
(SIN Y CON AUTOHIDRÓLISIS)

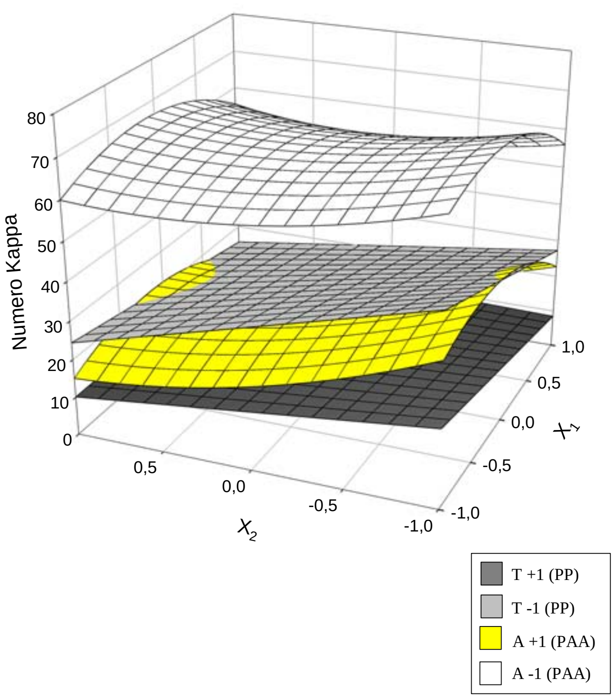
Leyenda

Gráfica	X1	X2	Constante
Proceso de Pasteado (PP)			
Rendimiento	Xt	XA	T
Numero Kappa	Xt	XA	T
Viscosidad	Xt	XA	T
Blancura	Xt	XT	A
índice de Tracción	XT	XA	t
Índice de Desgarro	XT	XA	t
Índice de Reventamiento	XT	XA	t
Proceso de Pasteado Despues Aurohidrolisis (PAA)			
Rendimiento	Xt	XT	A
Numero Kappa	Xt	XT	A
Viscosidad	Xt	XT	A
Blancura	Xt	XT	A
ïndice de Tracción	XT	Xt	A
Índice de Desgarro	XT	Xt	A
Índice de Reventamiento	XT	Xt	A

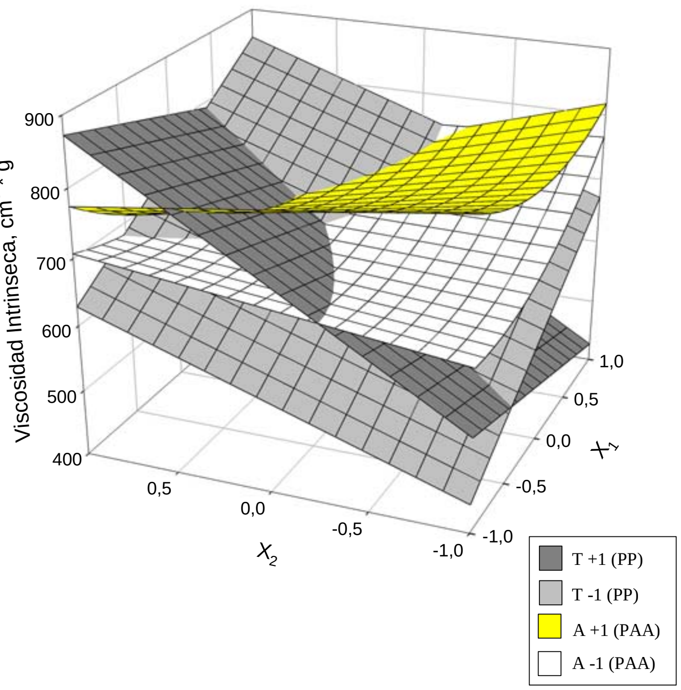
Temperatura = T; tiempo = t; Concentración de NaOH = A

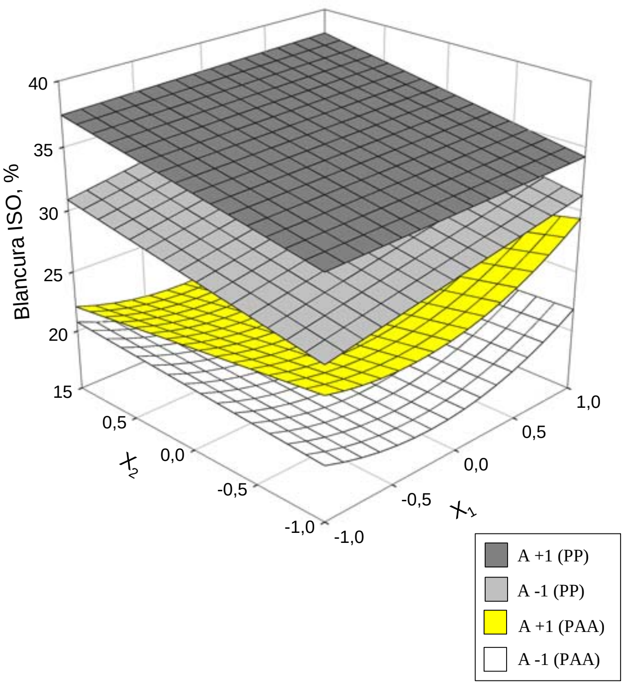


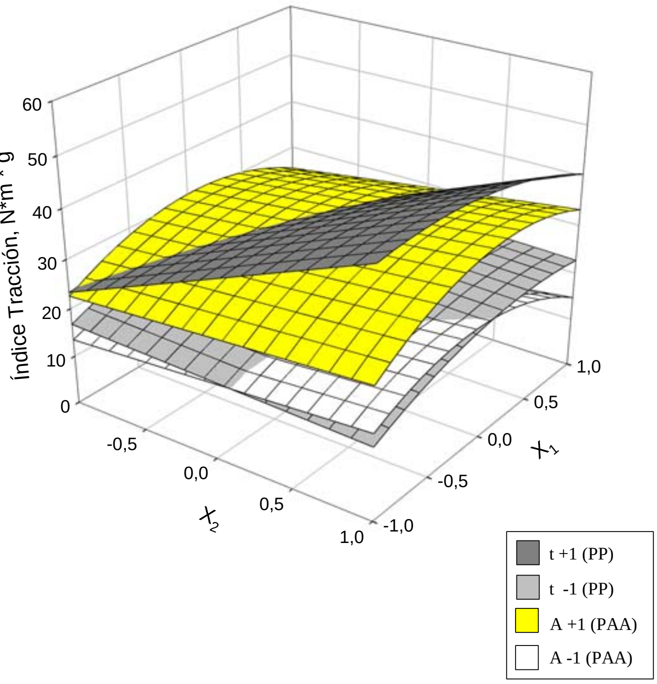
65
60
55
50
45
40
35
Rendimiento, %
-0,5
0,0
0,5
1,0
X2
-1,0
-0,5
0,0
0,5
1,0
X1
T +1 (PP)
T -1 (PP)
A +1 (PAA)
A -1 (PAA)



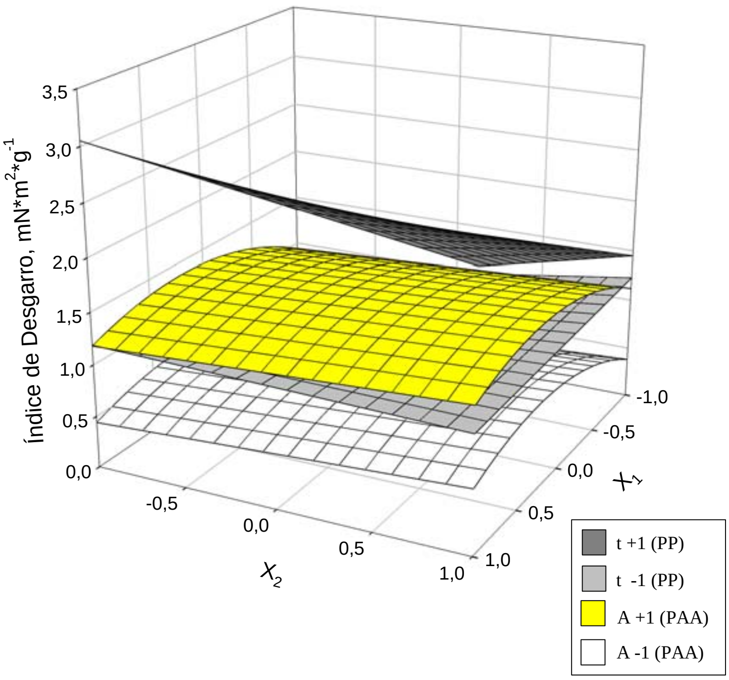
80
70
60
50
40
30
20
10
0
Numero Kappa
0,5
0,0
-0,5
-1,0
X2
1,0
0,5
0,0
-0,5
-1,0
X1
T +1 (PP)
T -1 (PP)
A +1 (PAA)
A -1 (PAA)


900
800
700
600
500
400
Viscosidad Intrinseca, cm3 * g-1
0,5
0,0
-0,5
-1,0
X2
1,0
0,5
0,0
-0,5
-1,0
X1
T +1 (PP)
T -1 (PP)
A +1 (PAA)
A -1 (PAA)

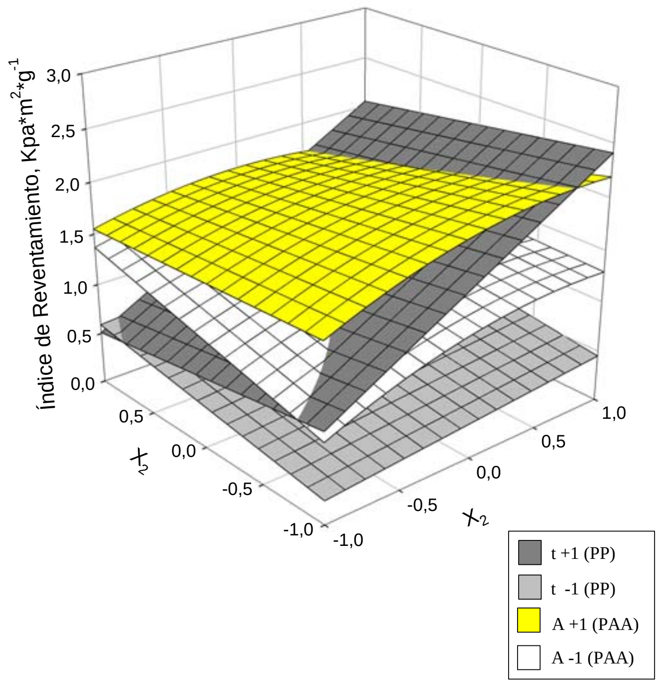

40
35
30
25
20
15
Blancura ISO, %
0,5
0,0
-0,5
-1,0
X2
1,0
0,5
0,0
-0,5
-1,0
X1
A +1 (PP)
A -1 (PP)
A +1 (PAA)
A -1 (PAA)



Índice Tracción, N*m * g-1
60
50
40
30
20
10
0
-0,5
0,0
0,5
1,0
X2
1,0
0,5
0,0
-0,5
-1,0
X1
t +1 (PP)
t -1 (PP)
A +1 (PAA)
A -1 (PAA)



Índice de Desgarro, mN*m2*g-1
3,5
3,0
2,5
2,0
1,5
1,0
0,5
0,0
-0,5
0,0
0,5
1,0
X2
-1,0
-0,5
0,0
0,5
1,0
X1
t +1 (PP)
t -1 (PP)
A +1 (PAA)
A -1 (PAA)



Índice de Reventamiento, Kpa*m2*g-1
3,0
2,5
2,0
1,5
1,0
0,5
0,0
0,5
0,0
-0,5
-1,0
-1,0
-0,5
0,0
0,5
1,0
X2
t +1 (PP)
t -1 (PP)
A +1 (PAA)
A -1 (PAA)