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Abstract: The influence of the process conditions of pulping of a trihybrid clone Paulownia on pulp properties for the soda anthraquinone process has been investigated in a semi-pilot scale. A composite central experimental design and a multiple regression were used to find the relationship between independent process variables and pulp properties.

The ash content (0.89%) is lower and cellulose content (44.0%) is higher than those found for other species of Pauwlonia and other energetic crops. The elemental composition has a low content in S and N (0.21%) in comparation with poplar or willow. With a gross heating value of 20335 J/g, Paulownia is a suitable feedstock for use as solid biofuel. This is somewhat higher than those for hardwood, slightly higher than those for Pinus pinaster and softwood, and much higher than those for residues of food plants and agricultural crops. This supports the use of the genus Paulownia as an energy crop.

The soda-anthraquinone pulping could be and adequate process for Paulownia. Fibre length (0.97 mm) is similar to hardwoods and suitable physical characteristics of paper sheets (tensile index) and acceptable chemical characteristics and yield pulping could be obtained by operating at low-intermediate temperature (163-171ºC) and alkali concentration (20%) and high or medium values for operation time (120-150 min). The pulp obtained at these conditions has suitable chemical (pulp) and physical (paper sheets) characteristics: yield (47.0%), ethanol-benzene extractives (2.22%), holocellulose contents (96.0%), α-cellulose contents (75.8%), lignin contents (8.28%), Shopper Riegler degree (23.2 ºSR), and tensile index (36.0) kN m/kg.

## Highlights

The influences of the process conditions of pulping of a trihybrid clone Paulownia on pulp properties for the soda anthraquinone process have been investigated in a semi-pilot scale.

The soda-anthraquinone pulping could be and adequate process for trihybrid clone *Paulownia* is similar to hardwoods and suitable physical characteristics of paper sheets.

Trihybrid clone *Paulownia* is a suitable feedstock for use as solid biofuel.

# *Paulownia* as solid biofuel and cellulose pulp source

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## 1. Introduction

The alkaline processes (as kraft) are dominant for the conversion of lignocellulosic material into pulp fibers in world. The key to the kraft process is the kraft recovery furnace that is quite efficient at recovering the pulping chemicals, sodium hydroxide and sodium sulphur. However, energy efficiency is becoming more important each passing year and there is a sense of inevitability that gasification of the black liquor will replace the furnace for chemical and energy recovery [1]. A gasifier yield around 7.9 GJ per ton of pulp versus 3.2 GJ in a furnace from a kraft process [2]. Then the soda/anthraquinone pulping can be proposed as a suitable process for pulp and paper production with a more efficient recovery of energy that kraft process. Moreover, do not has, the problems associated to presence of sodium sulphur in gasification of black liquors [1].

Soda anthraquinone pulping is an alternative process for producing Paulownia pulp. In comparison to kraft pulping, soda-anthraquinone process has higher yield and the same kappa level and better delignification, on diverse raw materials, without environmental damage due to the absence of sulphur emissions [3]. Effectively, this process is sulphur free, which simplifies the recovery of chemicals and eliminates emissions of odorous sulphur compounds to the air [4]. Also the soda-anthraquinone pulping is equally considered suitable for small scale mills.

Soda and soda-anthraquinone pulping has traditionally been used for non-wood fibers, such as straw, sugarcane bagasse, flax, and other alternatives sources of fibers [5-7]. Such raw materials played a dominant role as sources of pulp up to about a century ago and they still remain an important fiber source for many types of papers in certain developing countries and for specialty grades in developed countries. Soda anthraquinone pulping is also used to produce high yield hardwood pulps that are employed to make packaging papers and boards. This process has recently been investigated in laboratory scale for a trihybrid clone of *Paulownia fortunei x tomentosa x elongate*, [8, 9] where the conditions of pulping were fixed (temperature: 160ºC, operation time: 90 min, anthraquinone concentration: 0.1% o.d.b. and soda concentration: 23% o.d.b.).

Paulownia genus has attracted considerable attention as dedicated energy crop. Because it has modest water requirements, Paulownia has aroused interest as an industrial raw material despite its difficult growth in marginal areas [10]. 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 [11]. 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). Also, they exhibit fast growth and can produce as much biomass in one year as other species in several [10, 12]. 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. 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 tomentosa clone used by Ayan *et al* [13] 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 [11, 13].

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 [14]. 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 [9, 11]. 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 uniform and regular growth. 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 [8, 16].

In this study, the influence of the process conditions (soda concentration, temperature and cooking time) of pulping of a trihybrid clone *Paulownia fortunei x tomentose x elongate* on pulp properties for the soda anthraquinone process have been investigated in a semi-pilot scale. A composite central experimental design and a multiple regression were used to find the relationship between independent process variables and pulp properties.

## 2. Materials and methods

### 2.1. Raw material. Provision and characterization

A trihybrid variety of *Paulownia Elongata x Fortunei x Tomentosa* 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 a 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.

Characterization experiment involved the following parameters: 1% NaOH solubles (Tappi 212 om-98), hot water solubles (Tappi 207 cm-93), ethanol-benzene extractives (Tappi 204 cm-07), $\alpha$-cellulose (Tappi 203 cm-09), lignin (Tappi 222 om-06), ash (Tappi 211 om-07), and holocellulose (Wise method) contents. All treatments in this study were in a completely randomized design with five replications (variation coefficient less than 5%). Also, the metal contents (Ca, Cu, Fe, K, Mn and Na) was determined according standard Tappi 266 om-06 and the elemental composition (N, C, H, S, O) in Fisons EA 1108 Elemental Analyzer.

The gross calorific values (constant volume) were determinate according “CEN/TS 14918:2005 (E) Solid biofuels—Method for the determination of calorific value” and UNE 164001 EX standards by using a Parr 6300 Automatic Isoperibol Calorimeter.

Fiber length was determined according Tappi standard (T233 cm-06).

## 2.2. Experimental design for pulping conditions. Characterization of cellulosic pulp and paper sheets

*Paulownia fortunei x tomentosa x elongate* wood trimming were used for pulp and papermaking, but only wood was considered as it contained the bark, which was very thin and difficult to strip off also, it accounted for only < 1% of the overall mass.

The raw material and water were mixed in the desired proportions and reacted in a 10 L stainless steel MK-systems Inc. reactor fitted with recirculation for obtaining cellulose pulps. The reactor was then closed and simultaneously heated and actuated to assure good mixing and uniform swelling of Paulownia hidrolizated-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 fibres, during 10 min to 2000 rpm. Characterization experiments of pulp involved the following parameters: yield (Tappi 257 cm-02), ethanol-benzene extractives (Tappi 204 cm-07), $\alpha$-cellulose (Tappi 203 cm-09), holocellulose (Wise method), lignin (Tappi 222 om-98) and soluble lignin (Tappi 250 Wd-96) contents.

Paper sheets were prepared with an ENJO-F-39.71 sheet machine according to the Tappi 205 sp-06 standard. From paper sheets, grammage (Tappi 220 sp-06), Shopper-Riegler degree (Tappi 227 om-09) tensile index (Tappi 494 om-06), and Gurley porosity (Tappi 536 om-07) were determined.

To be able to relate the dependent variables (Yield, ethanol-benzene extractives, holocellulose, α-cellulose, Klason lignin, Soluble lignin, Shopper-Riegler degree, Tensile index, and Gurley porosity) with independent variables (NaOH concentration, temperature and time of process) in pulping process with the minimum testing, it was used 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. Independent variables were normalized by using the following equation:

$$X_n = \frac{X - \overline{X}}{(X_{max} - X_{min})/2} \qquad (1)$$

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. A liquid/solid ratio of 12/1 and anthraquinone concentration of 0.1 (dry wt. basis) were used in all experiments.

The number of tests required was calculated as $N = 2^n + 2 \cdot n + n_c$; $2^n$ 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 = 16.

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

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

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 T-test and having a 95% confidence interval excluding zero).

## 3. Results and discussion

### 3.1. Properties of the materials

Table 1 summarizes of chemical composition of *Paulownia fortunei, P. tomentosa, P. elongata* (bibliographic references) and the trihybrid used in this work: *Paulownia fortunei* + *tomentosa* + *elongata*. The trihybrid of Paulownia was harvested after 3 years of growth and produced more than 50 tonnes of biomass per hectare per year [17]. The mean fiber length was 0.97 (0.52) mm with a minimum of 0.5 mm and a maximum of 2.9 mm. This is a fiber length similar to hardwoods like *Eucalyptus globulus*.

The soluble content is lower than other raw materials and similar to wood species. Specifically, the content of extractives compounds in *Paulownia fortunei x tomentosa 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 [10, 16]. 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 [18] and could cause problems by adhering to machinery and reducing the quality of pulp.

The ash content is lower than those found for other species of Paulownia and other energetic crops as willow or poplar (1.99% and 1.87% respectively) [19]. A low ash and silicon content is preferable because silicon entering alkaline pulping processes will cause problems in the chemical recovery line at the pulp mill. The ash content is also related with chlorine content. Chlorine is very corrosive and can affect the yield in boiler [20]. In *Paulownia fortunei x tomentose x elongata*, ash percent are low. Then the amount of solid waste after combustion will be lower and yield of boiler not will be affected.

The cellulose content in *Paulownia trihybrid*, is 6.0%-17.6% (expressed as glucan), lower than those found for *Eucalyptus globulus*, 46.8% -53.4%, but higher (22.2%-14.9%) that those found for *Paulownia Fortunei*, whereas lignin content is 6.7%-17.6%, higher than Eucalyptus's one, 22.9%. The holocellulose content was higher to that of *Paulownia fortunei or* eucalyptus (66.9%) and lower than that of *Paulownia tomentosa* and Paulownia elongate [21]. These contents were similar to result from other authors for hardwoods [22].

Respect to gross heating value, table 2 shows selected calorific values reported by several authors. In short, softwood and related materials typically have values in the region of 20000 J/g and hardwood such as that from *Eucalyptus globulus* yields about 18000 J/g, whereas other deciduous plants (and their residues) give lower values. The gross calorific value for *Paulownia fortunei x tomentosa x elongata* are somewhat higher than those for hardwood, slightly higher than those for *Pinus pinaster* and softwood (see Table 2), and much higher than those for residues of food plants and agricultural crops. This supports the use of the genus Paulownia as an energy crop.

Also, gross heating value for Paulownia was higher than other solid biofuels as willow or poplar (between 18841 J/g and 19678 J/g) [23]. Overall, the inferior heating value (between 15910 J/g and 16748 J/g for *Paulownia fortunei x elongata, P. tomentosa* and *P. elongata* with a 30% of humidity) for genus Paulownia is higher than other species as *Pinus Pinaster, Pinus radiata* or *Eucalyptus globulus* (between 12979 J/g and 15492 J/g) [20].

The elemental composition of *Paulownia fortunei x tormentosa x elongata* has a low content in S and N in comparison with poplar (1.5% of S [20], 0.41% of N [19] or willow (0.38% of N [19]). S and N are responsible of $SO_x$ and $NO_x$.

The Ca contents are similar to willow (59.7 ppm) and poplar (53.2 ppm), but alkaline metals contents are higher than these species (K: 15.6 ppm and 20.4 ppm; Na: 1.3 ppm and 1.3 ppm for willow and poplar respectively) [19]. The use of ash from poplar and willow as fertilizer has been proposed by Tharakan *et al.*, [19] because his content in Ca and K.

### 3.2. Experimental design, modelization and optimisation

The normalized values of independent variables and properties of the pulp and paper sheets obtained in the pulping process, using the proposed experimental designs are shown in table 3. Each value in experimental results is an average of five (chemical pulp properties), three (Shopper Riegler degree and Gurley porosity) or twelve (tensile index) samples. The deviations for these parameters from their respective means were all less than 5%. Substituting the values of the independent variables for each dependent variable in table 3 into the polynomial expression used yielded the equations showed in table 4.

Chemical pulping process includes complex chemical reactions and the effect of different process variables are most effectively investigated in design experiments. The advantage of designed experiments under controlled conditions in laboratory or semi pilot scale is the possibility to find out the effect of all independent variables, including interaction and quadratic effects. In fact, identifying the independent variables with the strongest and weakest influence on the dependent variables in equations 1 to 8 is not so easy since the former contain quadratic terms and other factors involving interactions between two independent variables, although here we would like to highlight the following:

Observing the linear terms of the equations in Table 4, we can reach the most obvious conclusions on how independent variables would affect dependant variables. For example, to achieve the best holocellulose and α-cellulose contents and tensile index we advise, as a general rule, operation at high values for independent variables (temperature and operation time), but in order to achieve the best yield, as a general rule, we recommend operation of independent variables at low values. The influence of the linear term $X_A$, was negligible in the operation range selected. For the holocellulose and α-cellulose contents the “linear” trend at which work is most convenient, at high temperature and operation time, could be corrected by terms $-X_T^2$ and $+X_t^2$ respectively, that would show the possibility of working at intermediate temperature and operation time levels. As far as interaction terms are concerned, we can see the recurrence of term $X_AX_T$ in almost all equations in Table 4 with a similar effect to the general effect of the quadratic terms. The linear operation trend is corrected or modulated under the most thorough operating conditions. Although the $+X_T$ and $+X_A$ interaction effect brings an increase in α-cellulose content, this may be due to a hemicellulose degradation process. In fact, term $+X_AX_T$ of equation 5 (lignin) and $–X_AX_T$ of equation 3 (holocellulose) suggest the combination of low operation temperatures with high alkali concentrations or vice versa, and this shows that cellulose chain degradation is higher under more thorough operating conditions.

In order to determine the values of the independent variables giving the optimum values of dependent variables, the response surfaces for each dependent variable were plotted at two extreme levels of the independent variable most strongly influencing each and a fixed value of the two least influential variables.

An example of the above is Figure 1 that shows that low lignin content may be obtained at low active alkali concentration, intermediate pulping temperature and intermediate or high pulping time. This partly confirms the prior analysis of the quadratic terms and the effect of excessive hemicellulose degradation (increase the holocellulose and lignin content) in combination with high temperature and operation time.

The response surface for the yield (not show) shows that greater yield is achieved at low independent variable levels with the influence of temperature being less pronounced at low alkali concentration levels.

Holocellulose contents are greater at high operation temperature, with this being the most influential independent variable (the coefficient $X_A^2$ is important but active alkali concentration in equation 3 did not have a statistically significant linear coefficient, over the ranges considered), although the temperature effect is less pronounced at higher alkali concentration levels. Then an intermediate operation time and temperature at high alkali concentration could be selected (figure 2). In a similar scenario, optimum levels of α-cellulose content may be reached. The greatest α-cellulose content may be obtained at high operation time and alkali concentration when operation temperature is relatively low, 171ºC.

The response surface for the tensile index (figure 3) also shows it is convenient to operate at high time with the effect being much less relevant than in the case of operation temperature, although here we must highlight the positive effect over the tensile index of a greater alkali concentration with high operation time. The response surface for Shopper Riegler degree (not show) shows that a suitable refining degree is achieved at high operation time and intermediate alkali concentration.

## 4. Conclusions

The ash content (0.89%) is lower and cellulose content (44.0%) is higher than those found for other species of Paulownia and other energetic crops. The elemental composition has a low content in S (0.00%) and N (0.21%) in comparison with poplar or willow. With a gross heating value of 20335 J/g, *Paulownia fortunei x tomentosa x elongata* is a suitable feedstock for use as solid biofuel. This is somewhat higher than those for hardwood, slightly higher than those for *Pinus pinaster* and softwood, and much higher than those for residues of food plants and agricultural crops. This supports the use of the genus *Paulownia* as an energy crop.

The soda-anthraquinone pulping could be and adequate process for *Paulownia fortunei x tomentosa x elongata.* Fiber length (0.97 mm) is similar to hardwoods and suitable physical characteristics of paper sheets (tensile index) and acceptable chemical characteristics and yield pulping could be obtained by operating at low-intermediate temperature (163-171ºC) and alkali concentration (20%) and high or medium values for operation time (120-150 min). The pulp obtained at these conditions has suitable chemical (pulp) and physical (paper sheets) characteristics: yield (47.0%), ethanol-benzene extractives (2.22%), holocellulose contents (96.0%), α-cellulose contents (75.8%), lignin contents (8.28%), Shopper Riegler degree (23.2 ºSR), and tensile index (36.0) kN m/kg.

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**Table**

| | *Paulownia fortunei x tormentosa x elongata* | *Paulownia fortunei* | | | *Paulownia Tormentosa* | *Paulownia elongata* |
|---|---|---|---|---|---|---|
| **Hot Water solubles %** | 13.32 | [16] | [10] | [15] | [11] | [24] |
| **1% NaOH solubles %** | 28.63 | | | | | |
| **etanol benzene extractives %** | 4.66 | | | | | |
| **Klason lignin %** | 24.6<br>27.8 | 27.2 | 22.4 | 28.0 | 22.1 | 20.5 |
| **Holocellulose %** | 71.42<br>65.2 (glucan + hemicelluloses) [9] | 56.9 | 70.9 | 69.6 | 78.8 | 75.7 |
| **α-cellulose,%** | 37.70<br>44.0(as glucan) [9] | 34.2 as glucan | 37.4 as glucan | n.d. | 48.3, as glucan | 43.6 |
| **Ash (%)** | 0.89 | 2.09 (*elongata x fortunei*)[20] | | | 1.98 [20] | 1.88 [20] |
| **Gross Heating value, J/g-over dry basis** | 20,335 | 19,843 [25]<br>17,648 (*elongata x fortunei*) [20] | | | 18,339 [20] | 17,690 [20] |
| **N, C, H, S, O,%** | 0.21, 46.0, 5.86, 0.00, 48.0 | | | | | |
| **Ca, Cu, Fe, K, Mn, Na (ppm)** | 57.0, 0.39, 1.56, 93.0, 0.16, 7.1 | | | | | |

Table 1: Chemical composition of *Paulownia fortunei, P. tormentosa, P. Elongata* and the trihybrid used in this work: *Paulownia fortunei + tormentosa + elongata*.

| **Raw material** | **Gross calorific value ( J/g)** |
|---|---|
| Pine Cone | 27,350 [26] |
| Wood bark, Gossweilerodendron balsamiferum, Chlorophora excels, Cedrus atlantica, Wheat straw | 20,500–20,300 [27,28] |
| Spruce wood, Pinus pinaster, Softwood, Pseudotsuga menziesii, Pinewood | 20,100-19,600 [27-29] |
| Hazelnut shell, Hazelnut seedcoat, Beech wood, Fraxinus angustifolia, Hymenaea courbaril, Fagus sylvatica, Olive husk, Entandrophragma , cylindricum, Ailanthus wood | 19,300 -19,000 [27,28] |
| Populus euro-americana, Hardwood, Quercus robur, Castanea sativa, Acer pseudoplatanus, Prunus avium, Salix babilónica | 18,800-18,200 [27,28] |
| Corn stover, Tobacco stalk, Eucalyptus globulus, Tobacco leaf, Tea waste, Waste material, Corncob, Flax straw, Soybean stalk | 17,800-17,000 [27-30] |
| Timothy grass, Barley straw | 16,700-15,700 [29] |

Table 2 Gross heating values for different raw materials (bibliographic references)

| Normalized values of active alkali concentration, time and temperature | Yield (%) | Ethanol-benzene extractives (%) | Holocellulose (%) | α-Cellulose (%) | Klason Lignin (%) | Soluble Lignin (%) | ºSR | Gurley porosity (%) | Tensile index (kN m/kg) |
|---|---|---|---|---|---|---|---|---|---|
| 0 0 0 | 49.8 | 2.45 | 93.10 | 76.93 | 9.19 | 0.017 | 20.1 | 27.9 | 23.91 |
| 0 0 0 | 48.0 | 2.41 | 94.60 | 76.38 | 9.14 | 0.013 | 20.5 | 30.8 | 23.80 |
| +1 +1 +1 | 45.0 | 2.30 | 96.85 | 78.88 | 11.00 | 0.014 | 23.0 | 42.7 | 38.42 |
| +1 +1 -1 | 47.2 | 2.13 | 96.70 | 75.21 | 9.97 | 0.009 | 22.0 | 14.7 | 18.90 |
| +1 -1 +1 | 45.9 | 2.55 | 95.48 | 72.24 | 11.00 | 0.008 | 20,0 | 21.6 | 24.25 |
| +1 -1 -1 | 51.0 | 2.55 | 93.55 | 71.00 | 9.60 | 0.008 | 19.0 | 20.6 | 24.76 |
| -1 +1 +1 | 34.5 | 1.92 | 99.70 | 73.73 | 9.00 | 0.009 | 24.0 | 55.5 | 50.07 |
| -1 +1 -1 | 51.5 | 1.30 | 94.09 | 74.43 | 9.60 | 0.010 | 19.0 | 14.3 | 21.91 |
| -1 -1 +1 | 47.0 | 1.81 | 96.45 | 71.00 | 9.20 | 0.009 | 19.4 | 20.0 | 13.30 |
| -1 -1 -1 | 55.2 | 1.70 | 91.22 | 71.07 | 10.00 | 0.010 | 15.0 | 10.2 | 18.90 |
| +1 0 0 | 52.1 | 2.29 | 95.71 | 76.38 | 9.04 | 0.118 | 19.0 | 9.5 | 17.00 |
| -1 0 0 | 52.1 | 1.78 | 95.05 | 76.00 | 8.80 | 0.009 | 19.0 | 5.9 | 14.82 |
| 0 +1 0 | 48.4 | 2.06 | 95.16 | 76.60 | 8.00 | 0.011 | 23.0 | 36.5 | 33.87 |
| 0 -1 0 | 50.3 | 2.59 | 93.95 | 71.31 | 8.15 | 0.011 | 20.0 | 17.1 | 21.16 |
| 0 0 +1 | 40.1 | 2.58 | 94.70 | 78.04 | 10.26 | 0.009 | 22.2 | 60.3 | 29.81 |
| 0 0 -1 | 49.7 | 2.13 | 89.41 | 75.61 | 10.39 | 0.009 | 18.0 | 37.3 | 13.75 |

Table 3 Values of the independent variables and the physico-chemical properties of the pulp and paper sheets obtained in the pulping process by using the proposed experimental design.

**Table**

| Eq. | Equation | **$r^2$** | **F** |
|---|---|---|---|
| **1** | $Y_{YI} = 49.24 – 2.26\ X_t – 4.21X_T + 2.56\ X_A^2\ 4.58\ X_T^2 + 1.44\ X_A\ X_t – 2.22\ X_A X_T$ | 0.87 | 18 |
| **2** | $Y_{EEB} = 2.37 + 0.33\ X_A – 0.15\ Xt + 0.13\ X_T – 0.34\ X_A^2$ | 0.87 | 25.1 |
| **3** | $Y_{HO} = 93.51 + 1.19\ X_t +1.82\ X_T +2.03\ X_A^2 + 1.21\ X_t^2 – 1.29\ X_T^2 – 1.10\ X_A\ X_T$ | 0.89 | 20.9 |
| **4** | $\alpha\text{-C} = 76.55 + 0.75\ X_A + 2.22\ X_t + 0.66\ X_T – 3.01\ X_t^2 + 0.59\ X_A\ X_t + 0.71\ X_A\ X_T$ | 0.93 | 32.9 |
| **5** | $Y_{LI} = 8.94 + 0.84\ X_A – 0.66\ X_t^2 + 1.59\ X_T^2 + 0.48\ X_A\ X_T$ | 0.91 | 36.8 |
| **6** | $Y_{SR} = 20.19 – 0.66\ X_A + 1.76\ X_t + 1.56\ X_T – 1.26\ X_A^2 + 1.24\ X_t^2 – 0.92\ X_A \cdot X_T$ | 0.93 | 34.8 |
| **7** | $Y_{GP} = 28.68 + 7.43\ X_t + 10.31\ X_T – 22.37\ X_A^2 + 18.85\ X_T^2 – 3.03\ X_A\ X_t - 2.76\ X_A\ X_T + 7.29\ X_t\ X_T$ | 0.98 | 97.9 |
| **8** | $Y_{TI} = 21.78 + 6.08\ X_t + 5.76\ X_T – 3.79\ X_A^2 + 7.81\ X_t^2 – 3.93\ X_A\ X_t + 6.72\ X_t\ X_T$ | 0.92 | 30 |

Where YI denotes yield (%), EBE the Ethanol-benzene extractives (%), HO the holocellulose (%),α-C the α-cellulose (%), LI the Klason lignin (%), SR the Shopper Riegler degree, GP, the Gurley porosity, TI the tensile index (kN m/kg), and $X_T$, $X_t$, and $X_A$the value of the temperature, time and active alkali respectively.
The differences between the experimental values and those estimated by using the previous equations never exceeded 10% of the former.

Table 4 Equations yielded for each dependent variable.

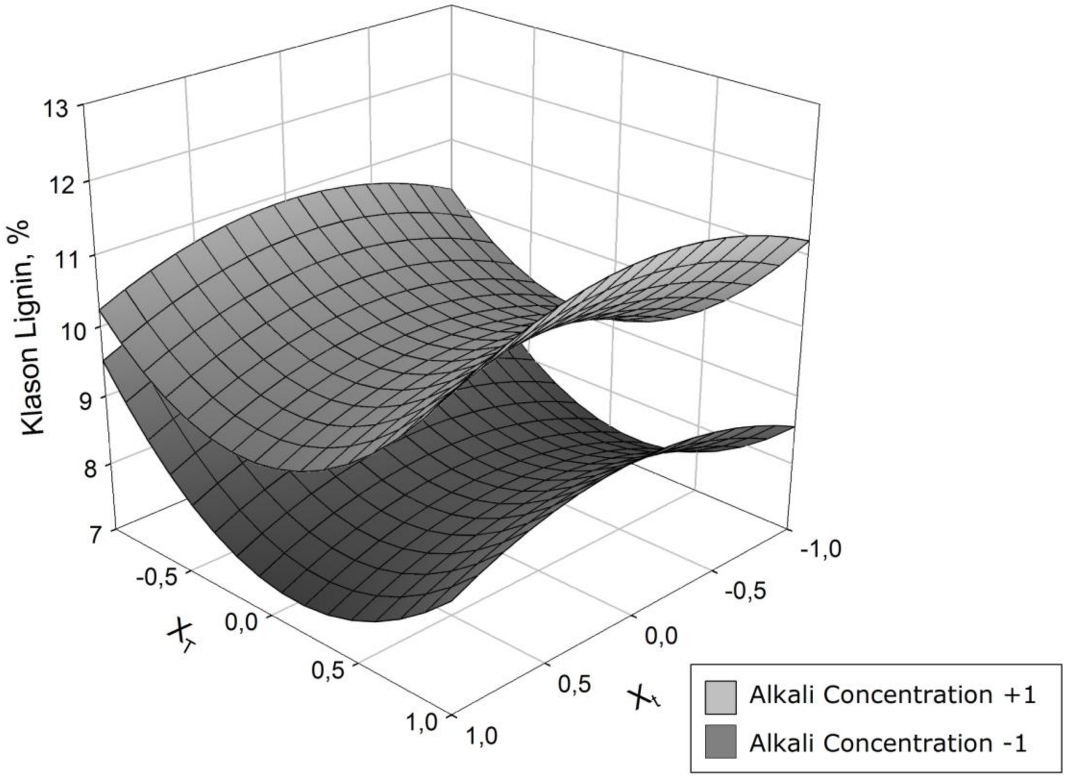

Figure 1. Variation of Klason lignin with time and temperature at extreme alkali concentrations

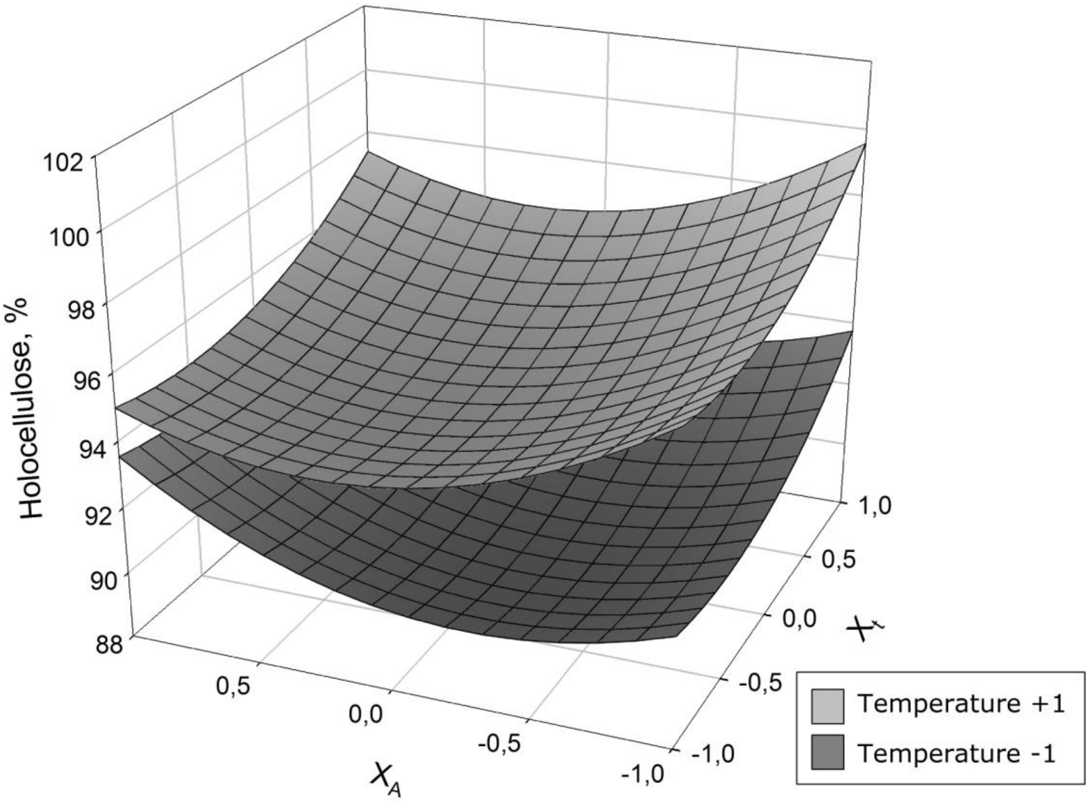

Figure 2. Variation of Holocellulose with time and alkali concentration at extreme temperatures

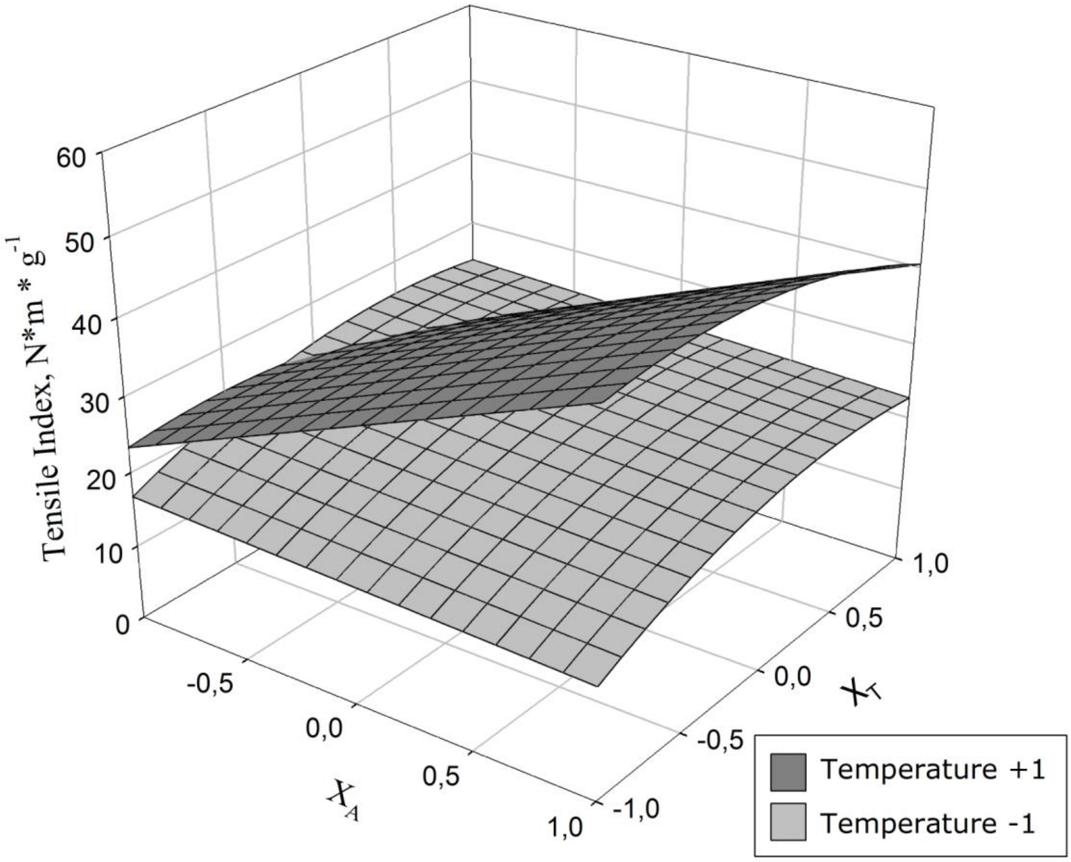

Figure 3. Variation of Tensile index with time and alkali concentration at extreme temperatures