

Influence of Input Variables over the Wood Digestion in a Sulfite Pulp Mill for Biorefinery Purposes

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Abstract

Eucalyptus globulus is the most profitable specie in Europe for pulping. However, in recent years some diseases and pests have caused its defoliation, affecting the available quantity of wood. In this sense, the mills are studying how to avoid the loss of pulp yield as well as the optimization of byproducts in order to take advantage of every component present in the wood using biorefinery processes. One of the possible options is to complete the consumption of *E. globulus* with other species such as *Eucalyptus nitens*.

The objective of this paper is to study the behavior of both species related to the dissolving pulp manufacturing process as well as the characteristics of the resulting products. Parameters that can be very useful for the evaluation of the raw material, such as the growing data or density of both species, have been compared. Major attention has been focused on the composition of both species and how it affects the characteristics of the possible final products.

E. nitens presents good results of forestry characteristics, being better than *E. globulus* in terms of growing and resistance to frosts. The cellulose content of both species is similar, however the digestion process in the case of *E. nitens* needs to be improved in order to optimize the involved energy and the obtained products.

Keywords: Biorefinery, Eucalyptus, Globulus, Nitens, Pulp, Spent liquor, Sulfite process

1. Introduction

Forest fiber resources are the main materials to produce cellulose products and paper pulp. In recent years, half of the world's round wood harvest is used for industrial purposes (Willför et al., 2011). In addition, valorization options in pulping have been studied during the last decades because of the sugar and lignin content of wood, converting the pulp mills into the biorefinery concept (Rueda et al., 2015).

Eucalyptus is one of the genus most commonly used in Europe and specifically in the north of Spain to produce pulp because of its fiber size, quality and lighter color as well as its productivity and plasticity (Pérez Cruzado et al., 2011). It is the most profitable in Europe for this type of use, beating birch and other hardwoods.

Pulp manufacturers in northern Spain had traditionally used *Eucalyptus globulus* as raw material in their pulping processes to obtain dissolving pulp. However, in recent years some diseases, such as *Mycosphaerella*, and pests, such as *Gonipterus scutellatus*, have affected the quantity available of this wood (Otero et al., 2006; Sánchez et al., 2011). These pests and diseases cause massive defoliation in young leaves annually, producing important losses of growth and weakness of the masses, with generalized mortality in frost exposed zones (Tejedor, 2004a).

In this way, some factories are studying how to palliate this situation. One option is the selection of the most resistant families and the cloning of the best trees of *Eucalyptus globulus* in order to get varieties tolerant to the diseases. Another option is to improve *Eucalyptus nitens* characteristics, such as wood density, growth, diseases resistance and coppice ability.

In this sense, the basic knowledge of the chemical composition and structure of the wood as raw material (Sixta, 2006) is fundamental to understand the pulping process and the possible valorization of the waste materials obtained in this process. Wood is formed mainly by cellulose, hemicellulose, lignin and small percentages of extractives and ash. In an acid sulfite process, the process in which this work is focused, the fraction of cellulose is oriented to obtain dissolving pulp; however, the rest of the components, lignin and hemicellulose, are byproducts that can be valorized into new high value-added products such as ethanol or xylitol from fermentable sugar (from the hemicellulose) and lignosulfonates (from the lignin) (Rueda et al., 2015).

The objective of this work is to study how far *E. nitens* affects the parameters of the existing process as well as the properties of the obtained pulp but also of the spent liquor for its valorization opportunities.

It is important to mention that the timber arriving to any factory is a quite heterogeneous material because many factors are involved in the wood quality: the genetic of the tree (not just the species, but also the provenance, the family, clones...) the soil of the forest, the age of the trunk, the position inside the trunk, etc. (Tejedor, 2004a). So, this *Eucalyptus* species can not be seen as a unique value applicable for *E. nitens* or *E. globulus* but a wide variety of values that can be found inside each species.

2. Materials and Methods

2.1 Raw Materials

The wood used has been *Eucalyptus globulus* and one selected tree of *Eucalyptus nitens* from Cantabrian forests supplied by Sniace SA group (Torrelavega, Spain). Logs have been chipped and classified. The chips used in the cooking experiments have 15-30 mm length and 2-4 mm thickness.

The parameters of the cooking liquor (made by dolomite, water and SO₂) have to be as homogeneous as possible. In this work, the parameters have been the following: total SO₂ (7.5-8.5g/100 mL liquor), combined SO₂ (1.5-1.8g/100 mL liquor).

2.2 Cooking Experiments

The cooking experiments were carried out in one-liter stainless steel reactors with temperature and pressure control and an external electric heating system. For this, wood and fresh liquor were loaded into the digesters and after some operation conditions (pressure, SO₂ content and residence time) unbleached pulp and spent liquor were obtained.

The process for each experiment has been the following: wood and cooking liquor are loaded

into the digesters using a fixed liquid/solid ratio. Then, the reactors are pressurized using argon gas until 9 bar of pressure and the heating ramp until desired constant temperature is set. The digesters have an automatic system of pressure control which maintains the pressure constant throughout the whole process. After the desired time at maximum temperature, the heating is stopped and the pressure is relieved. Once atmospheric pressure is achieved, digesters are discharged. Finally, the composition of resulting spent liquor and pulp, if applicable, is analyzed.

2.3 Analytical Methods

The following methods were used for wood characterization. Tappi T257 (Tappi, 1993) was used to carry out the sample preparation. Extractives were determined according to the standard UNE EN ISO 14453 (ISO, 1999) for pulp, modified for its use with wood by means of an extraction with acetone as solvent using a soxhlet apparatus. Ash was measured using the standard Tappi T211 (Tappi, 1993) and a furnace at 525 °C. Lignin was determined by Tappi T222 (Tappi, 1993) as follows: carbohydrates in wood are hydrolyzed and solubilized by sulfuric acid; the acid-insoluble lignin is filtered off, dried, and weighed; the acid-soluble lignin can be determined in a solution after filtering off the insoluble lignin using a spectrophotometer based on absorption of ultraviolet radiation at 205 nm. Hemicellulose was determined with the Wise method (Wahab et al., 2013). The sample is treated with water and sodium chlorite in an acid medium achieved by the addition of some drops of acetic acid and it is heated using a water-bath at 75-80 °C. This procedure is repeated until the sample is bleached. In this case, it was carried out three times. After that, the sample is cooled, filtered and washed with water and acetone. Finally, the sample is dried and weighed. Cellulose in this work was determined as alpha-cellulose. Its content was measured using the standard Tappi T203 (Tappi, 1993) by means of which, the obtained material after the hemicellulose determination is extracted consecutively with 17.5% and 9.45% sodium hydroxide solutions at 25 °C. The soluble fraction, consisting of beta- and gamma-celluloses, is determined volumetrically by oxidation with potassium dichromate, and the alpha-cellulose, as an insoluble fraction, is derived by difference.

The quality grade of cellulose pulp was evaluated by means of kappa index according to Tappi T236 standard (Tappi, 1993) and intrinsic viscosity following the guidelines of ISO/FDIS 5351 (ISO, 2009).

The spent liquor was characterized as follows. Free, total and combined SO₂ were determined according to the titration method Tappi T604 (Tappi, 1993) with iodine and sodium hydroxide. Sugar content (glucose, xylose, arabinose, rhamnose and galactose), acids (acetic, levulinic and formic) and other decomposition products (furfural and hydroxymethyl furfural) were measured by a Shimadzu Prominence HPLC with SHODEX SH1011 column and Refraction Index detector according to Llano et al. (2013) method using 0.5mL/min of 0.005M H₂SO₄ as a mobile phase, 60 °C in the oven and 198 psi of column pressure.

3. Results and Discussion

Different characteristics of both species have been analyzed in order to study the possibilities

of consuming both species in a pulp mill; starting with the forestry characteristics and followed by their density and chemical composition. Finally, the behavior in the chemical pulping has been assessed to improve the separation of the main components of the wood, giving the best valorization options of the spent liquor.

3.1 Plantation

Eucalyptus globulus is very sensitive to frosts, especially during the first two years, putting up with 5 and 30 frosts by year although in coastal areas this phenomenon is less frequent, but in higher altitudes these can present more than 70 times per year (Brinkmann, 1992). The most favorable temperature for the specie is situated as an annual average between 10 and 15 °C, with limits of -4.5 ° and 40.5 °C for short periods of time (Brinkmann, 1992). This specie must not exceed seven dry months.

However, *Eucalyptus nitens* is able to bear the same temperatures and to grow enduring between 50 and 150 frosts per year. In comparison with *E. globulus*, previous studies (Tejedor, 2004a) have demonstrated that *E. nitens*, in general, is less affected by pests and diseases previously mentioned, especially in altitudes higher than 300 meters above sea level. There are also important differences between provenances of *E. nitens* for diseases resistance.

Additionally, the growing data of both species for the region of Cantabria, 24-29 m³/ha/year for *E. nitens* and 13-17 m³/ha/year for *E. globulus* (Forest Patrimony of Sniace group), gives an idea of how the productivity in the whole process from the forest to the factory can increase. Therefore, *E. nitens* presents better forestry characteristics according to the purpose of this work.

3.2 Density

Density is one of the most important properties of wood and pulping processes because it is related to mechanical resistance and to cellulosic and energetic flair. Optimal density is a function of end-use of wood.

Table 1 shows the density values obtained by other authors and those of this work. First, what attracts attention is that the density of *Eucalyptus nitens* is slightly lower than *Eucalyptus globulus*. In Cantabrian forest, the results are 475-650 Kg/m³ for adult trees of *E. globulus* and 460-520 Kg/m³ for *E. nitens*. *E. globulus* presents more density than *E. nitens* in all cases; however, the studied interval of variation is wider in the case of *E. globulus*. Density is a parameter which is very dependent on other factors such as the genetic of the tree (provenance, family, clone...), the age of trunk, the position inside the trunk and the forest location.

Table 1. *E. globulus* and *E. nitens* density values (Kg/m³)

	<i>E. globulus</i>	<i>E. nitens</i>
Cotterill (1997)	533	463
Evans (2000)	-	471.1
Monteoliva (2002)	549	-
Paz (2003)	554.5	499
This work	532	485

3.3 Composition

Composition knowledge is very important to evaluate the wood for pulping and valorization options. Between hardwoods and softwoods, generally the first kind has a higher proportion of holocellulose and less lignin although a greater percentage of extractives. However, among different species of hardwoods there are differences that make them suitable or non-suitable for a fixed pulping process. For dissolving pulp, higher contents of cellulose and lower of lignin are more interesting. Thus, the results obtained by different authors and those of this work are shown in tables 2 and 3.

 Table 2. *E. globulus* composition values (%)

	Alonso (1976)	Pereira (1988)	Cotterill (1997)	Paz (2003)	This work
Extractives*	0.42/1.4	4.9	-	1.18	1.5
Ash	0.48	0.40	-	-	0.35
Lignin	21.24	23.1	27	20.85	23.28
Alpha-cellulose	-	54**	45	50.65	51.3
Holocellulose	79.47	-	-	84.1	80.5

* Different solvents employed by each author

** Total content of cellulose

 Table 3. *E. nitens* composition values (%)

	Alonso (1976)	Cotterill (1997)	Paz (2003)	This work
Extractives*	0.31/0.76	-	1.57	1.98
Ash	0.24	-	-	0.31
Lignin	21.33	27	21.41	25.43
Alpha-Cellulose	-	43	47.33	51.2
Holocellulose	83.14	-	83.27	78.4

* Different solvents employed by each author

The first parameter in both tables is related to the percentage of extractives, which has very different values due to the use of different solvents such as acetone (this work), ethanol (Paz, 2003), ether (Alonso, 1976), ethanol-benzene (Alonso, 1976) or a mixture of dichloromethane, ethanol and water (Esteves, 2008; Pereira, 1988).

The second parameter is ash. Very similar results have been obtained for both species.

According to other parameters such as alpha-cellulose, it can be said that *E. nitens*, which presents similar values to *E. globulus*, theoretically looks suitable at the same level as *E. globulus* in order to obtain pulp. Regarding lignin, its slightly higher content in the case of *E. nitens* can make the process a little more complicated; however, this content can increase the valorization options of the byproducts oriented to a biorefinery.

3.4 Pulping

In order to evaluate the suitability of both species to produce pulp and the development of valorization options, an analysis of the cooking stage has been done in two steps: the study of the impregnation step and the study of the behavior of both species in pulping.

3.4.1. Impregnation Step

The non-uniformity of pulp has unfavorable effects on the cooking and bleaching steps. To obtain a uniform pulp, the cooking liquor must be equally distributed into wood chips, that is, to achieve a uniformly impregnated wood in the minor possible time (Gullichsen and Sundqvist, 1995; Gustafsson, 1988). This is why the impregnation step has an important role in the whole process because it is the first and can have a bearing on the improvement of the delignification and penetration times of liquor into wood. The effect of the quality of the impregnation has been studied by other authors (Gullischen et al., 1995; Hart et al., 2011; Kazi et al., 1998; Malkov et al., 2004).

Some impregnation trials were carried out at laboratory scale. The impregnation value in each trial is obtained taking into account the difference of liquor volume among the input and the output, and using an average value of liquor density of 1054 kg/m³ obtained in a previous work (Rueda et al., 2015). Therefore, the final impregnation value is given in mg/g dried matter (DM).

Once the impregnation is obtained in mg/g, the percentage of impregnation can be calculated in every period of time taking into account the impregnation at that time and the maximum value that the wood can be impregnated for each specie (showed in table 4). Based on the experience of the mill, an 80% of impregnation is considered to be enough in this type of process.

In order to obtain a representative curve of the impregnation, data were represented by first order exponential equations according to equation 1; where y represents impregnation in mg/gDM and a , b are constants. The obtained parameters are shown in table 4 and the curves in figure 1.

$$y = a(1 - e^{(-bx)}) \quad (1)$$

Table 4. Obtained parameters for both types of wood

	<i>E. globulus</i>	<i>E. nitens</i>
a (mg/gDM)	558.56	764.68
b (min ⁻¹)	0.01292	0.009350

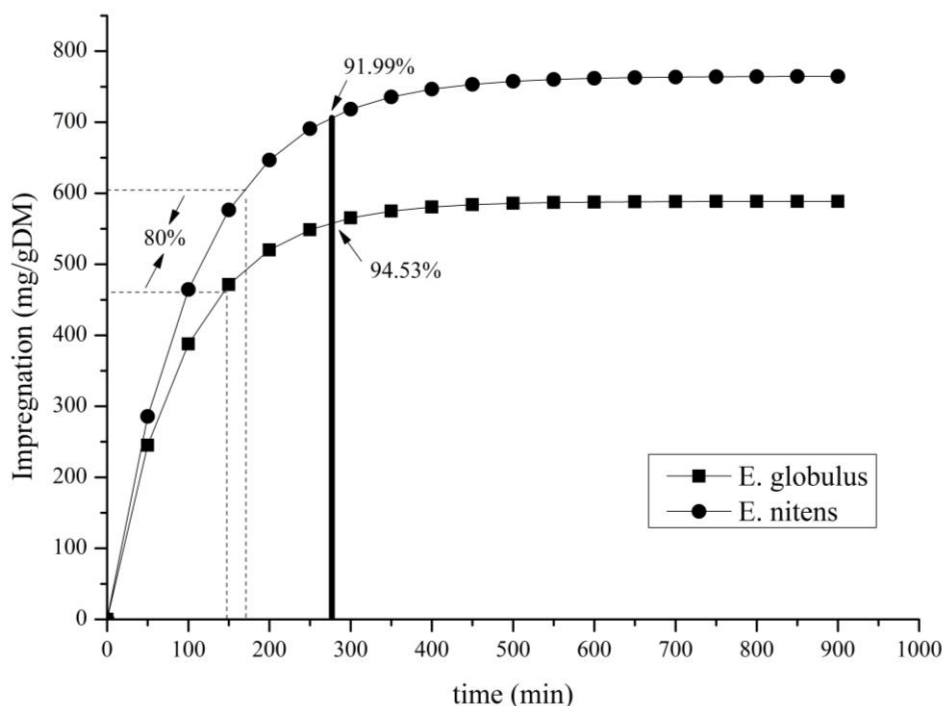


Figure 1. Tendency of impregnation stage of *E. globulus* and *E. nitens*, in mg/gDM and in impregnation percentage.

As can be seen in table 4 and figure 1, *E. nitens* has a higher maximum impregnation value, which means that for the same conditions at certain time, it will impregnate more milligrams per grams of available dry wood; however, it will be less impregnated than *E. globulus* because it needs more time to reach the same percentage of impregnation. Furthermore, both types of wood fulfill the requirement of an impregnation of 80%. Focusing on time employed in the existing mill, the impregnation obtained for *Eucalyptus globulus* represents a 94.53% of the available wood versus 91.99% for *Eucalyptus nitens*. Both values exceed the 80% declared as enough, therefore *E. nitens* could be used without big modifications in the existing process.

3.4.2. Cooking Step

A literature search was carried out focusing on the pulping behavior of these two types of wood in different processes. The aim is to evaluate the yields and the properties of both in pulp and byproducts, to get better products and higher valorization opportunities.

All the literature consulted refers to alkaline processes and not to sulfite processes. On one hand, the study of Paz (Paz, 2003) shows that *Eucalyptus globulus* (~54%) has a little more yield than *Eucalyptus nitens* (~52.7%) for a final kappa closest to 14 ± 1 . Escalona et al. (Escalona et al., 2000) affirms that *E. globulus* has the best pulping yield and furthermore, *E. nitens* consumes more energy in cooking to reach a similar grade of delignification (kappa 16 ± 1). On the other hand, Clarke (Clarke, 2000) affirms that the yields of the samples that are used in his work are compared favorably to those reported for *Eucalyptus globulus*.

In order to check some of the statements of the literature, some laboratory tests have been carried out and the properties of the resultant products after the cooking process have been analyzed.

The experiments were carried out as was mentioned in the epigraph 2.2., fixing the liquid/solid ratio at 20:1 L/Kg in order to avoid the diffusion and to study only the effect of the cooking stage, as the impregnation stage was previously studied.

Figure 2 shows the evolution of lignosulfonates and total monomer content of spent sulfite liquor of *E. nitens* and *E. globulus* digestion. The *E. nitens* showed a higher delignification grade since a higher content of lignosulfonates was found in the spent sulfite liquor. Furthermore, the total sugar content is slightly lower in the spent liquor obtained using *E. nitens* than in the one obtained by *E. globulus* digestion. This indicates that *E. nitens* spent liquor would reach a greater degree, better for lignosulfonate valorization purposes than sugars.

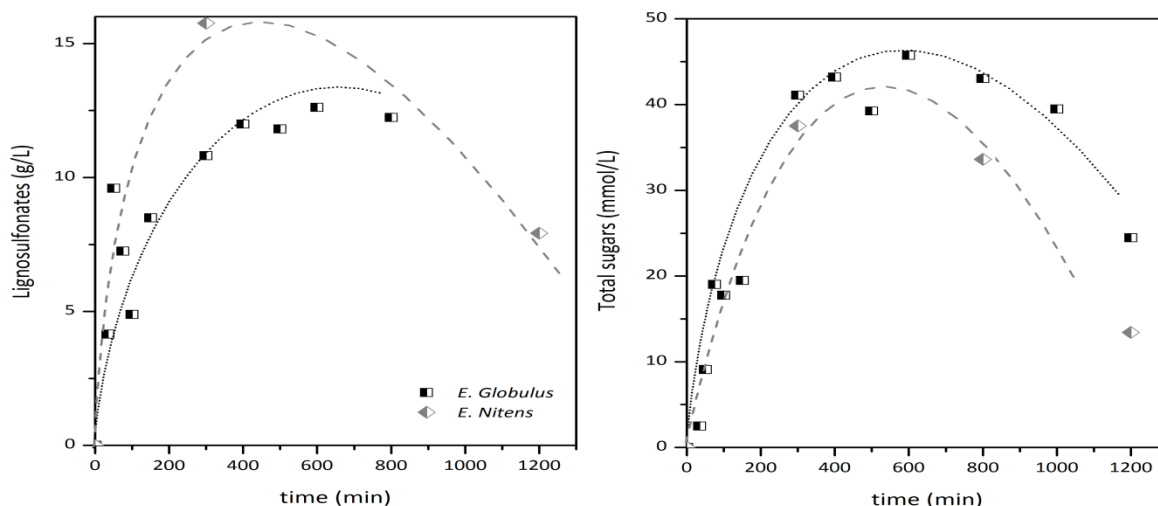


Figure 2. Evolution of lignosulfonates and total sugar content in spent sulfite liquor with time for *E. globulus* and *E. nitens* digestion.

Taking a cooking time of 300 minutes, the properties of the obtained pulp have been determined and shown in table 5.

Table 5. Kappa number and viscosity for different samples

TIME (min)	Kappa		Viscosity (mL/g)	
	<i>E. globulus</i>	<i>E. nitens</i>	<i>E. globulus</i>	<i>E. nitens</i>
300	7.57	10.87	578.19	374.40

Regarding to the pulp properties, it seems that the digestion process in the case of *E. nitens* is more aggressive and it is able to isolate the lignin and hemicellulose from cellulose; however, the degradation of sugar could be too accused, turning their transformation into other decomposition products such as furfural or hydroxymethyl furfural (HMF) in the spent liquor. This means that the process could be carried out at a lower temperature which involves an

important decrease of the energy costs.

Based on the obtained results and the biorefinery purposes, *E. nitens* shows better results in terms of lignin valorization; however, the optimization of the pulping process using *nitens* is needed in order to increase the hemicellulosic content in the hydrolyzate (spent liquor).

4. Conclusions

Eucalyptus globulus commonly used in northern Spain and the Iberian peninsula to produce pulp, has been afflicted in recent years by some pests and diseases that affect the available quantity of this wood. The evaluation as a raw material in the sulfite pulping process of other species such as *Eucalyptus nitens* has been analyzed in this work.

In terms of forestry characteristics, *E. nitens* is less affected by pests and diseases and also less sensitive to frosts. Furthermore, it has higher growing data. Another important characteristic is the wood density which in the studied case is lower for *E. nitens*. However, *E. nitens* can make a difference with higher growth and occupying areas where *E. globulus* does not withstand; which means that it could be planted successfully in these areas.

Regarding the chemical composition of both species, *E. nitens* presented in the studied case a higher content of lignin and similar content of alpha-cellulose than *E. globulus*, which theoretically would mean that *E. nitens* is a good candidate for producing dissolving pulp and furthermore, it would produce more byproducts, valid for biorefinery purposes. To verify this, some laboratory tests were carried out showing that in the first part of the process, the impregnation step, *E. nitens* needs a bit more time to reach the desired impregnation of the wood. However, in the second step of the process, the cooking stage, *E. nitens* gives a higher concentration of lignin in the spent liquor with only a little decrease in the sugar content and worse results of the quality parameters in the dissolving pulp. A decrease of the temperature of the digestion is recommended in this case in order to improve the properties of pulp and decrease other decomposition products in the hemicellulose fraction of the spent liquor, giving more productivity in the process.

To conclude, it can be said that *E. nitens* can be used to produce dissolving pulp and other byproducts which can be valorized by means of different biorefinery processes. However, an optimization of the pulping process should be carried out.

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