Floresta e Ambiente 2021; 28(2): e20200048
https://doi.org/10.1590/2179-8087-FLORAM-2020-0048
ISSN 2179-8087 (online)

ORIGINAL ARTICLE – Forest Products Science and Tecnology

Eucalyptus globulus bark valorization: Production of fibers by Neutral Sulphite Semi-Chemical Process for Liner Paper Manufacture

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Abstract

Eucalyptus globulus is the second most important economic forest species in Chile. Its main use is in the kraft pulp industry, where large amounts of bark waste are generated. Due to its fibrous characteristics, *E. globulus* bark is proposed as an alternative source of fibres for papermaking. This study focuses on obtaining fibres for liner paper manufacture. A neutral sulphite semi-chemical (NSSC) process was performed, varying the sodium sulphite (5% to 16%) and the sodium carbonate (2% and 4%) concentrations using two reaction temperatures (160°C and 170°C). The NSSC process at 170°C, 16% of sulphite, and 2% of sodium carbonate proved to be the best condition to obtain higher mechanical performance of papers. As the pulping conditions become more drastic, the yield drops, and the physicomechanical properties of paper increases. Results showed that pulps from *E. globulus* bark could turn into source of fibres for papermaking and other related products.

Keywords: Biomass valorisation, papermaking, paperboard, NSSC pulping.

1. INTRODUCTION AND OBJECTIVES

Eucalyptus is one of the most important tree species for the pulp and paper industry and the specie most widely cultivated in temperate regions (Neiva et al., 2018). It is a fast-growing tree, with a total plantation area between 16-19 million hectares and an annual production of 15-25 ton/ha (Romani et al., 2019). More than 55% of the current global *Eucalyptus* production is found in South America. The Chilean forestry industry consumes around 11.9 million m^3 of *Eucalyptus* wood per year, and 53% is destined for pulp production (ODEPA, 2016).

In the pulp and paper industry, the growing demand for its products, has add extra pressure on the increasingly scarce forest resources, forcing the search for new alternatives for fibre sources. Some alternatives that have been proposed as new raw materials for pulping and paper production are the new fast-growing tree species, annual plants, and residual biomass (Eugenio et al. 2019; Małachowska et al., 2019).

Many companies have considered the use of recycle fibres as an alternative in the pulp and paper industry. Although the use of recycled fibres has environmental and economic benefits, unfortunately, these processes imply the use of heterogeneous material and with several recycling cycles, which generates an intrinsic deterioration of the fibres due to shorter fibre length. Therefore, this could generate an increase in the amount of fines and a low drainability of papers (Rahmaninia and khosravani, 2015; Yang and Berglund, 2020).

In this context, residual biomass from pulp and paper processing has received increasing attention in recent years and may constitute an alternative resource for the extraction of cellulose fibres (Miranda et al., 2013). The advantages of being produced the fibres using residual biomass in the same process decrease the cost of transport and logistics of the supply.

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Therefore, *Eucalyptus* bark is presented as an interesting raw material for fibre making (Miranda et al., 2012).

Bark represents a substantial proportion of the above ground total biomass of trees, reaching about 10-15% of the total weight of the tree, and particularly in *Eucalyptus globulus* specie, it represents around 11% of the dry weight of the stem (Sartori et al., 2016). Bark has a higher content of extractives and ash than wood (Neiva et al., 2018). Physiologically, tree bark consists mainly of three types of cells—phloem fibres, sieve cells, and phloem parenchyma cells, which are responsible for conducting nutrients along the plant (Matsushita et al., 2010). Bark comprises all the tissues outside of the vascular cambium and represents the outermost layers of the stem and roots of woody plants. It is mainly composed of polysaccharides, lignin, polyphenolics extractives and inorganic materials (Neiva et al., 2018; Sillero et al., 2019). During industrial processing for pulping, bark is removed from the trunks and constitutes an important mill residual material that is usually burned for energy production, contributing to the sustainability of the pulp mill processes (Miranda et al., 2013). 20 tons of bark can be obtained in a pulp mill by 100 tons of pulp processed (Neiva et al., 2018).

Neutral sulphite semi-chemical (NSSC) pulping is a type of semi-chemical pulping process to produce unbleached pulp yields of around 80%, using sodium sulphite as the main cooking agent, buffered with sodium carbonate to maintain a neutral solution (Pereira et al., 2011; Oveissi et al., 2016). The presence of a buffered sodium sulphite cooking solution produces sulphonation and lignin hydrolysis when the cooking time is about 30 minutes at 170-180 ºC (Pereira et al., 2011; Bajpai, 2018). Sulphite pulping like a kraft process, also allows the recycling of chemical agents used, mainly to reduce costs (Neiva et al. 2018). If ultrafiltration membranes were used to recycle filtrate with a high content of sulphite, it would allow a saving of at least 25% in the cost of reagents (Monsalve et al., 2010).

This research aimed to evaluate the technical feasibility of using *E. globulus* bark as a source of fibres to produce liner-type paper, using an NSSC process. To assess the performance of our proposed method, the raw material and pulp obtained were chemically characterised, as well as the mechanical properties of paper sheets were determined.

2. MATERIALS AND METHODS

2.1. Raw material and preparation

E. globulus bark was obtained from a 15-year-old commercial tree plantation of a Chilean forest company located in Biobío region (southern Chile). Samples were thoroughly mixed to obtain a single uniform sample and dried at 40°C for 24 hours, cut into 2 x 2 cm chips and stored in a dry place until used.

2.2. Chemical characterization of E. globulus bark

E. globulus bark was milled in a knife mill and sieved to 45-60 mesh. Milled bark was extracted with a 90% acetone solution for 16 h to determine the quantity of extractives (TAPPI T-280 pm-99 method). Extractives-free bark was hydrolysed with 72% sulfuric acid at 30 °C for 1 h (300 mg of sample and 3 mL of sulfuric acid). The acid was diluted to 4% (adding 84 mL of water) and the mixture was heated at 121°C (1 atm) for 1 h. The residual material was cooled and filtered through porous glass filter number 4. The solids were dried to constant weight at 105 °C and determined as insoluble lignin according to the TAPPI standard (T222 om-02, 2006). The soluble lignin concentration in the filtrate was determined by the measurement of the absorbance at 205 nm and using the value of 110 L/g cm as the absorption coefficient. The concentration of monomeric sugars in the soluble fraction was determined by high-performance liquid chromatography (HPLC) (Merck Hitachi, Germany); column BIO-RAD HPX-87H at 45 °C, eluted at 0.6 mL/min with 5 mM H_2SO_4 using a refraction index detector at 30 ºC. Glucose, xylose and mannan were used as external calibration standards. The glucans content was calculated by multiplying the glucose content by 0.9; the xylan/mannan content obtained from the xylose/mannose content multiplied by 0.88 (Mendonça et al., 2008). All samples were analysed in triplicate.

2.3. Neutral sulphite semi-chemical pulping of E. globulus bark

Neutral sulphite semi-chemical pulping (NSSC) was carried out using a rotating digester equipped with four independent vessels of 1.2-liter capacity, 100 g of *E. globulus* bark (dry basis) and 500 g of liquor. For the screening-type experimental design, the charge of Na_2SO_3 varied between 5% to 16%, and the sodium carbonate (Na_2CO_3) charge, between 2% to 4%. The reactor heating rate was 2.1 ºC/min from room temperature and the cooking temperature was 160 ºC and 170 ºC. After each reaction, the black liquor was drained, and pulps were washed with water at 50 °C until neutralise the pH of the liquor and thereby ensure the remotion of carbonates and sulphites from the pulp. Pulp yield was measured using the weight of the washed material. Cooked material was defibrated using a 12" laboratory single disc refiner, the pulp was defibrated making pass 3 times the

material through the refiner, with disc opening of 0.02 mm. The consistency of defibration varied between 4% in the first past until 2% in the last. Defibration yield was measured after refining and screening of the cooked material. Samples were stored for the chemical characterization and for the elaboration of liner papers.

2.4. Bark pulps morphological properties

Fibre morphological properties such as, length and fines content of bark pulps were determined by a Fiber Tester apparatus (Lorentzen & Wettre, Sweden), where a 100 mg sample was previously disintegrated in 100 mL distilled water for 10 minutes. During the analysis, the equipment measured approximately 35000 fibres of each sample. The samples were evaluated in triplicate.

2.5. Paper sheets making

Sets of ten paper sheets were made with pulps obtained from NSSC pulping experiments. The paper sheets were prepared according to the TAPPI standard (T205 sp-02, 2002) to obtain grammages of 100 g/m^2. The formation of the paper sheets was carried out in a sheet former (Regmed, Brasil). All paper sheets were dried for 5 hours at room temperature and then were conditioned to 50% relative humidity and 23°C for at least 8 hours. After conditioning, the dry weight of all the elaborated paper sheets was determined.

2.6. Paper sheets mechanical properties

Ten paper sheets were made for each experiment of the NSSC pulping and their mechanical properties were measured using TAPPI standard methods. The tensile properties of papers were determined according to T494 om-01 (2001); the bursting properties of papers were determined according to T403 om-02 (2002); the internal tearing resistance of papers were evaluated according to T414 om-04 (2004); the flat crush of corrugating medium properties (CMT test) of papers were determined according to T809 om-99 (1999); and the ring crush of paperboard properties (RCT test) of papers were evaluated according to T818 om-97 (1997).

3. RESULTS AND DISCUSSION

3.1. Chemical characterization of E. globulus bark

The chemical composition of the *E. globulus* bark used in this research is detailed in Table 1. The main component found was glucans with 39.9%, followed by xylan/mannan with 23.0% and lignin with 21.0%. In relation to the content of glucans, differences in the total content are observed when compared with another research. Neiva et al. (2018) reported a glucan content of 37.5%, while that Miranda et al. (2012) reported 56%. This difference can be explained by the variations of the growth sites and the ages of the trees evaluated. Lignin content of 21% is slightly similar if it is compared with the research reported by Neiva et al. (2018) and by Fernandes et al. (2014), who reported values of 21.9% and 21.4%, respectively. Bark is usually rich in extractives -including organic solvent and water-soluble- and polyphenolics. The extractive content was 6.4%, which is consistent with results obtained by Neiva et al. (2018), who extracted with water, and Miranda et al. (2012), who reported an extractive content of 5.3% using a polar solvent. The bark may contain a significant percentage of ash and alkali-soluble extractives (Miranda et al., 2012) that were not determined, which is expressed in Table 1 as other components. This percentage may vary between 5% and 6% for *E. globulus* bark, as described by Neiva et al. (2018).

Table 1. *Eucalyptus globulus* bark chemical composition.

Component	Miranda, 2012	Fernández, 2014	Neiva, 2018	This work
Extractives, %	5.3	4.3	6.6	6.4
Lignin, %	16.9	21.4	21.9	21.0
Glucans, %	56.0	41.0	37.5	39.9
Xylan/mannan, %	23.7	13.5	23.6	23.0
Other, %	-	19.8	10.4	9.7

3.2. Pulp characterization

NSSC was performed on *E. globulus* bark at two different temperatures (160°C and 170°C) and in two concentrations of sodium carbonate (2% and 4%). Both variable conditions were studied with a variation of the sulphite concentration (5%, 8%, 12% and 16%). The results of these experiments are shown in Tables 2 and 3. The pulp yield (pulp basis)

obtained in the NSSC with 2% of carbonate is in the range of ~53%-63% (Table 2), while for pulps obtained with 4% of carbonate is between ~52%-61% (Table 3). The changes obtained in pulp yield are due to the sulphite concentration, which favours higher solubilization of lignin and hemicellulose fractions (Reyes et al., 2015). The yields obtained from bark pulps are lower than those obtained for semi-chemical pulps produced from wood, which yield 70%-90% (Pereira et al., 2011). However, the results showed are higher than those obtained by Neiva et al. (2016) and by Miranda et al. (2012), who used a kraft process in *E. globulus* bark and reported yields of 42% and 47%, respectively. The yields in the kraft process are lower than the sulphite processes, mainly because it is intended that the lignin is low enough to allow the separation of the fibre (Sixta, 2006).

The difference in the yield of different pulps studied could be explained by the anatomical difference and chemical characteristics of the two types of raw materials (Neiva et al., 2014). The pulping process could be affected by the loss of small parenchyma cells and phloem vessels during pulping. It is important to consider that the phloem cells in *E. globulus* bark are estimated to be 50% in volume. These cells could be easily degraded during pulping and thus could migrate with the effluent in the washing process (Neiva et al., 2016). The description of the anatomical characterization is supported by Quilhó et al. (2000), who determined that the type of parenchymal cells correspond to the main type of bark cell of *E. globulus,* which contributes to 50% by volume, followed by 28% of fibres. Moreover, chemically the bark has less hemicelluloses as reported by Neiva et al. (2014), which produces a reduction in fibrillation. As reported in the literature, the higher the hemicelluloses content, fibrillation capacity of cellulosic fibres it is easier (Iwamoto et al., 2008; Chaker et al., 2013).

Table 2. Pulp yield and chemical composition of pulp obtained from *E. globulus* bark with 2% of calcium carbonate.

Temperature, °C	Sulphite, %	Refined pulp yield, %	Cellulose (glucans), %	Hemicellulose (xylan/mannan), %	Acid soluble Lignin, %	Acid insoluble Lignin, %	Total lignin, %
		Calcium carbonate concentration, 2 %w/v					
160	5	61.7	43.6	11.2	1.3	15.8	17.1
	8	58.7	42.9	10.5	1.0	14.2	15.2
	12	56.5	42.1	9.1	1.3	11.8	13.1
	16	53.9	41.7	8.8	1.0	10.4	11.4
170	5	63.1	43.8	9.2	1.3	14.4	15.7
	8	59.5	41.7	9.0	1.4	11.9	13.3
	12	53.5	39.7	8.1	1.5	10.9	12.4
	16	51.2	38.6	7.7	0.9	10.6	11.5

Table 3. Pulp yield and chemical composition of pulp obtained from *E. globulus* bark with 4% of calcium carbonate.

Temperature, °C	Sulphite, %	Refined pulp yield, %	Cellulose (glucans), %	Hemicellulose (xylan/ mannan), %	Acid soluble Lignin, %	Acid insoluble Lignin, %	Total lignin, %
		Calcium carbonate concentration, 4 %w/v					
160	5	60.5	48.8	13.6	1.7	13.1	14.8
	8	59.8	46.0	12.9	1.5	12.4	13.9
	12	57.1	44.5	11.2	1.3	10.7	12.0
	16	54.4	42.3	9.8	1.4	9.9	11.3
170	5	60.8	46.9	13.0	1.9	15.2	17.1
	8	56.8	44.1	12.1	1.8	13.5	15.3
	12	54.6	43.5	10.7	1.1	12.2	13.3
	16	51.8	42.3	9.2	1.3	10.9	12.2

Concerning the chemical composition of the pulp, it is observed that the content of glucans varied from 38.6 to 48.8 g glucan/100 g, where the higher concentration is presented when the reaction conditions are less severe (160 °C, 5% of sulphite and 4% of carbonate) (Table 3). As shown in Tables 2 and 3, it is possible to observe that there is a decrease in the glucan contents at higher sulphite charge. For lignin, the concentration varied from 11% to 17%. The lower concentration of lignin (11.3%) was obtained using 160 °C, 16% of sulphite and 4% of carbonate (Table 3).

In contrast, the higher lignin content (17.1%) resulted using 160 °C, 5% of sulphite, and 2% of carbonate (Table 2). The xylan/mannan (hemicelluloses) content varied from 7.7% to 13.6%, where the higher concentrations resulted using 4% of carbonate (13%-13.6%) (Table 3), and the lowest concentration was obtained using 2% of carbonate (7.7%) (Table 2). Considering the chemical characterization of bark pulp, it is possible to determine the importance of the neutralizing agent, which with a dosage of 4% is able to maintain a high yield of glucans, xylan, and mannan in the pulp due to its ability to neutralize acidic agents that could disintegrate cellulosic material. This observation is supported by the research carried out by Ahmadi et al. (2010), who reported the use of the sodium carbonate as neutralizing agent at a concentration of 4% recommended to neutralize the reaction and to stabilize the cellulose structure in an NSSC pulping.

As observed in Tables 2 and 3, the sum of the percentages of glucans, xylan/mannan, and lignin, in addition to the cells lost by washing, there is a remaining fraction, which is not addressed on these tables. Such a relevant fraction should be expressed by the presence of extractive components and inorganic compounds. The bark, unlike the wood, contains much more extractives and ashes. Most of the bark extractives are similar to those contained in wood, however, the polyphenols and suberins contained in the extractives from bark are of higher molecular weight. Among these polyphenols, tannins can be found. These tannins could cause problems in the paper manufacture, in the equipment, and therefore in the physical-mechanical properties of the sheets themselves. In addition, the presence of extractives could generate problems such as: staining, resin, and undesired precipitations (Dutt and Tyagi, 2011; Miranda et al., 2012; Neiva et al., 2018). The amount of extractives can be dissolved using a neutral sulphite process (Young, 1994; Vásquez et al., 2008).

At the morphological level is observed that the stage of refining causes a decrease in the average fibre length in approximately 30%, going from 1.058 mm average in fibre without treatment to 0.73 mm in fibre treated with 2% of carbonate, 5% of sulphite at 160 °C, and to 0.76 mm in fibre treated with 2% of carbonate, 16% of sulphite at 170 °C. The concentration of fines increases in comparison with untreated fibres going from 4.8% to 5.6% and 5.8% average for pulp obtained at 160 °C and 170 °C, respectively.

One point to take in consideration in the refining stage of the NSSC pulping is the type of raw material that is introduced into the equipment. In all circumstances, it is important that the material introduced between the refining discs is uniformed in size, water content, and in its composition (Mokvist and Johansen, 1993). Therefore, the use of bark in the refiner should not cause a greater deterioration than regular use.

3.3. Paper sheets mechanical properties

The strength properties of the paper sheets elaborated with the pulps obtained from NSSC process are presented in Figure 1.

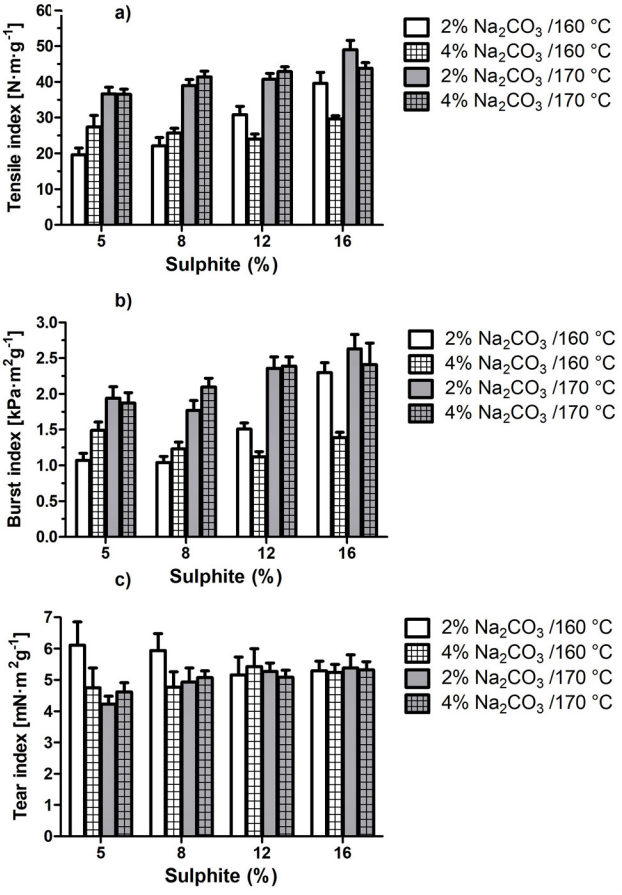

Figure 1. Strength properties of paper sheets prepared from *E. globulus* bark pulps using 2% and 4% of sodium carbonate at 160°C and 170°C in different sulphite concentration: (a) tensile index, (b) burst index, (c) tear index.

It is observed that the tensile index improved when the pulps were produced at 170 °C and 16% of sulphite resulting in 49 Nm/g and 44 Nm/g using 2% and 4% of carbonate, respectively as shown in Figure 1a. These results are higher than those reported by Fica (2015) regarding the recycled paper, where a highest tensile strength value of 38 Nm/g was obtained. However, a lower resistance is observed compared to paper sheets made from *Eucalyptus nitens* wood pulp using an NSSC process with values above 50 Nm/g at 170 °C and 15% of sulphite as described by Pereira *et al.* (2005).

This result can be explained by the differences in the average lengths of the fibres. The fibres from pulps obtained in this research at 170 °C using 2% of carbonate, with 12% and 16% of sulphite show average of fibre lengths of 0.78 mm and 0.76 mm, respectively. This result is similar as reported by Pirralho et al. (2014) who described fibre lengths of 0.72 mm from *E. globulus* wood. Morphological properties of fibres such as length and width could be considered critical properties to achieve efficient fibre-fibre bonding. Nevertheless, the large proportion of parenchyma cell and vessels probably play a role in the behavior of the bark pulps (Fernandes et al., 2014).

The burst index showed good results in pulps obtained at 170 °C as shown in Figure 1b. It was observed that as the sulphite concentration increases, the burst resistance rates are higher using a lower amount of carbonate. In contrast, when there is an increase in carbonate, only at higher temperatures does an upward trend follow, since at 160 °C the burst rate remains constant. With 2% of carbonate, the best burst indexes values were achieved in pulps at 170 °C using 12% and 16% of sulphite resulting in 2.4 and 2.6 $kPam^2/g$, respectively. With 4% of carbonate, the higher burst indexes were obtained in pulps processed at 170 °C with sulphite concentrations of 8%, 12%, and 16%, which resulted in values of 2.1, 2.4, and 2.4 $kPam^2/g$, respectively. These burst indexes are consistent and slightly higher than those described by Rudi et al. (2016) who reported values between 1.7 to 2.1 $kPam^2/g$ using an NSSC pulp on sunflower stalk. However, these values are lower when compared to the burst resistance reported by Pereira et al. (2005) who reported a value of 2.9 $kPam^2/g$ using NSSC pulp on *E. nitens* wood.

As shown in Figure 1c, tear resistance of pulps obtained at 170 °C increased with the sulphite concentration, while the opposite effect was observed when the temperature was 160 °C. For pulps with 2% of carbonate, the tear rates had two high values at each temperature with opposite sulphite concentration: at 160 °C with 5% sulphite, a tear index of 6.1 mNm^2/g was obtained and 5.38 mNm^2/g at 170 °C with 16% of sulphite. With 4% of carbonate, there were no significant differences in the results for both temperatures, being the higher values of 5.4 mNm^2/g at 160 °C and 12% of sulphite, and 5.3 mNm^2/g at 170 °C and 16% of sulphite. The tear index values obtained in this research are similar in comparison to those reported by Fernandes et al. (2014) who show a tear index value of 5.0 mNm^2/g using pulp from *E. globulus* bark.

The tear index has correlation with the chemical characterization of bark pulp. An NSSC less severe facilitates the highest tear index in comparison with an NSSC with the lowest temperature and sulphite concentration that have a higher quantity of lignin and hemicelluloses. This allows a structure more stable, with higher strength and rigid properties to the tear tests. Tear indexes are lower using higher sulphite concentration with a low quantity of lignin (11.5%) in the NSSC process. A small quantity of lignin and hemicelluloses allows a more rigid cellulose-cellulose bond. This phenomenon is consistent with that reported by Reyes et al. (2015), where the tear index in correlation to the decreasing of lignin and hemicellulose concentrations is discussed when the reaction conditions of the chemithermomechanical pulping mechanism are more severe using wood from *P. radiata*.

Figure 2 (a and b) shows CMT and RCT results, respectively. The NSSC pulps are considered optimal for preparing corrugated cartons due to their great stiffness (Casey, 1980; Cathie & Guest, 2001), resulting from their lignin content and the higher amount of hemicelluloses. The paper sheets tested by CMT shows a higher performance at the highest reaction temperature and sulphite concentrations. The results obtained with 4% of carbonate at 160 °C do not vary significantly when the sulphite concentration increases. The higher CMT values were 1.61 Nm^2/g and 1.63 Nm^2/g, obtained with 2% of carbonate at 170 °C using 12% and 16% of sulphite, respectively. According to the CMT index, the results shown in this research, present lower values than those reported by other authors such as Kasmani et al. (2014) in pulp from the mixing of the old corrugated container with virgin neutral sulphite semi-chemical pulp, where it was obtained a value of 2.73 Nm^2/g.

The RCT test depends on the force and pressure applied on the edge of the carton along the cylinder axis. The result is usually proportional to the CMT results (Casey, 1980). The paper sheets tested by RCT showed a higher result at the highest reaction temperature (170 °C) and at higher concentrations of sulphite as is shown in Figure 2b. The results obtained with 4% of carbonate at 160 °C showed a slight decrease in the property of RCT at higher concentrations of sulphite. The higher RCT values were obtained with 2% of carbonate at 170°C: 1.66 Nm^2/g and 1.74 Nm^2/g using 12% and 16% sulphite, respectively. In contrast, at 160 °C the higher value found was 1.54 Nm^2/g with 12% of sulphite. The results shown for RCT tests were higher when comparing them to those reported by Kasmani et al. (2014) where a higher value of 1.13 Nm/g was obtained in sulphite pulp obtained from old corrugated pulp with virgin fibres.

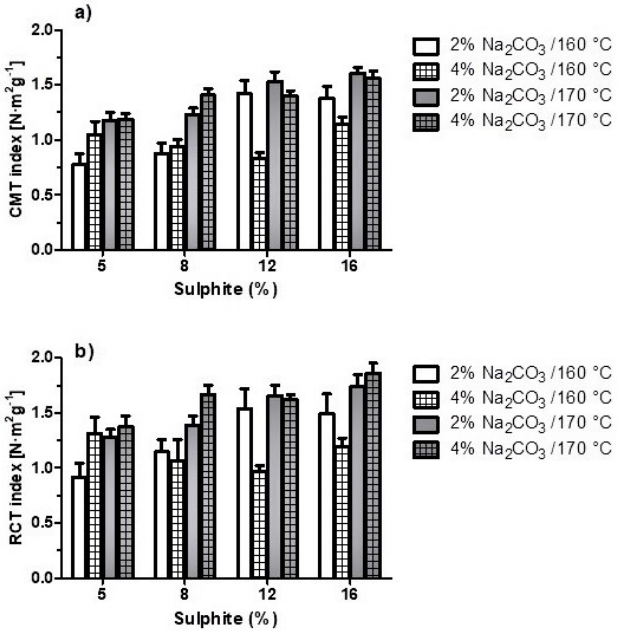

Figure 2. CMT (a), and RCT (b) properties of paper sheets produced from *E. globulus* bark pulps elaborated using 2% and 4% of sodium carbonate at 160°C and 170°C in different sulphite concentration.

4. CONCLUSIONS

Eucalyptus globulus bark has a high content of cellulose and hemicellulose with ratios that are slightly lower than in wood of *E. globulus*, which makes it an interesting raw material for the pulp industry, as an alternative fibre. The NSSC pulping is effective in getting pulp with high yield and good chemical characteristics. The conditions researched showed that 170°C, 2% of sodium carbonate, and 16% of sulphite are the best conditions to obtain higher mechanical performance of paper sheets. The properties of hand sheets produced under this condition are suitable to produce liner-paper with similar values to those offered in the market for recycled paper (testliner), providing advantages with the use of virgin fibres compared to the recycled fibres. *E. globulus* bark would have the potential to be used in the liner paper industry.

ACKNOWLEDGMENTS

This research was conducted with the support provided by the Innovation Fund for Competitiveness of the Chilean Economic Development Agency (CORFO) under Grant no. 13CEI2-21839. A. Andrade thanks to National Agency for Research and Development (ANID)/Scholarship program (Doctoral grant No 220-21202153).

SUBMISSION STATUS

Received: 17 Jul. 2020
Accepted: 22 Feb. 2021
Associate editor: Fernando Gomes

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