Effect of Kappa Number Variation on Modified Pulping of *Eucalyptus*

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This work evaluated the impact of brownstock pulp kappa number variations on modified pulping process, oxygen delignification, bleaching, and the physical characteristics on bleached pulp. Wood chips of 6-year-old *Eucalyptus grandis* x *Eucalyptus urophylla* from Brazil were used. A modified pulping process was performed with the purpose of achieving two delignification levels on brownstock pulp: kappa 18 and kappa 15. Pulps were submitted to oxygen delignification and elemental chlorine-free (ECF) bleaching to achieve 89.5 ± 0.5% of ISO brightness. Subsequently, pulps were refined in four levels (0, 750, 1500, and 3000 rotations) for physical mechanical tests. Delignification increased after increasing the cooking H factor and alkali charge. As a result, delignification negatively impacted the pulping yields (from 56.1% to 55.0%) and the pulp viscosity (from 1,317 cm^3/g to 1,227 cm^3/g). Pulps with an initial kappa of 15 presented more efficient oxygen delignification and lower consumption of bleaching reagents. The final viscosity of these pulps were lower (899 cm^3/g against 963 cm^3/g), however, than that of the pulps with an initial kappa of 18. Furthermore, the pulps with a higher unbleached kappa demanded less refining energy and had lower capillarity. The other physical properties were not influenced by the brownstock delignification level.

Keywords: Kraft pulping; Hardwood; Delignification level

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INTRODUCTION

The kappa number is one of the most important parameters measured in chemical pulping. It indicates the pulp delignification degree and is defined as the volume in mL of 0.1 N potassium permanganate solution consumed by 1 g of moisture-free pulp in an acidic medium (Saleem and Akhtar 2002). The main components of hardwood pulp kappa number are hexenuronic acids and lignin (Costa and Colodette 2002), and it is dependent on many cooking variables, such as pulping time and temperature (H factor), and applied alkali charge (Wan Rosli *et al*. 2009), depending also on the chemical composition of wood (Cardoso *et al.* 2002).

In a pulp mill, the brownstock kappa number influences some of the most important parameters of pulp production, as pulping yield and quality, alkali charge, solids generation, and bleaching chemical consumption.

The first direct impact of kappa number is on pulping yield. Delignification degree and yield are inversely related variables, *e.g*., increasing the pulp delignification degree and producing pulp with lower kappa decreases the pulping yields (MacLeod 2007; Vivian *et al.* 2014). On the other hand, a low degree of delignification (higher kappa) provides higher yields (Wan Rosli *et al*. 2009), positively impacting the production capacity of an industrial plant.

The quality of pulp is also related to its kappa number. An increasing of delignification and reducing of kappa promotes higher pulp degradation, which results the production of pulp with higher fines from fibers fragmentation and lower viscosity (Shin and Strömberg 2007; Pedrazzi *et al*. 2010).

Brownstock kappa number presents direct impact on the subsequent stages of pulp production, especially oxygen delignification and bleaching. Some studies indicate that oxygen delignification performance increases with higher lignin content in pulp and decreases with increased hexenuronic acids (Eiras *et al.* 2003; Ventorim *et al.* 2006). This difference is explained by the fact that the oxygen delignification primarily removes the lignin with minimal removal of hexenuronic acids (Ventorim *et al.* 2006). Furthermore, a higher kappa number increases the consumption of chemical reagents during bleaching, increasing the costs of this production stage (Forsström *et al.* 2006).

In addition to pulp quality, oxygen delignification and bleaching steps, kappa number plays an important role to recovery area of pulp mills. Increasing kappa number promotes a reduction of alkali charge, which means that less white liquor must be produced at causticizing (Almeida 2003). As the yields are improved when higher kappa is considered, lower load is sent to chemical recovery boiler. This fact is considered a benefit, since many times the capacity of the boiler firing represents a bottleneck for the increase in factory production (Gomide *et al*. 2011).

Conventional pulping processes have undergone some changes since the 1980s due to efforts to reduce environmental impacts, increase yields, and improve pulp quality. These efforts were responsible to create new pulping processes, known as modified pulping, that are based on better alkali and energy distribution during the cooking, allowing the implementation of ECF and TCF bleaching sequences (Silva Júnior and McDonough 2002).

The basic principles of modified pulping processes are: low and uniform alkali concentration during the pulping, high sulfide concentration, low content of dissolved lignin on pulping liquor and low temperature (Miyanish and Shimada 2001).

The use of modified pulping technologies allows a reduction on pulping temperature and alkali charge, production of pulp with better physical resistance and viscosity, improvement of washing efficiency, lower bleaching chemicals consumption, and reduction of specific wood consumption (Silva Júnior and McDonough 2002).

In this context, the objective of this work was to evaluate the impacts of kappa variation on the modified kraft pulping process, oxygen delignification, pulp bleaching, and pulp physical characteristics.

EXPERIMENTAL

Materials

Eucalyptus grandis and *Eucalyptus urophylla* wood chips from Brazilian trees six years in age were collected and screened in a laboratory screener. The thickness fraction between 4 and 6 mm was used for the experiments. The wood characterization is shown in Table 1.

Table 1. Wood Characteristics

Basic Density (g cm^{-3})	Total Extractives (%)	Total Lignin (%)	Ash (%)	Holocellulose (%)
0.469	3.06	28.09	0.45	68.85

Pulping

The modified kraft pulping was performed in a laboratory digester equipped with metering pumps for multiple extractions of black liquor and white liquor injections. The chips were steamed and submitted to an impregnation for 30 min. After that, the first extraction of black liquor and injection of white liquor was performed, starting the first pulping phase. The duration of this phase was 110 minutes, followed by the second extraction of black liquor and injection of white liquor. Immediately this second change of cooking liquor, the second pulping phase was performed for 125 min. The pulping conditions were adjusted to reach two delignification levels on pulps: kappa numbers 18 and 15. Sulfidity of 30% and liquor to wood ratio of 4 L/kg were used. The other fixed conditions of all treatments are shown in Table 2.

Table 2. Pulping Fixed Conditions

Phase	Retention time (min)	Temperature (°C)	Alkali charge distribution (%)
Steaming	15	100	-
Impregnation	30	120	50
1st pulping phase	110	variable	30
2nd pulping phase	125	variable	20

Liquor and Pulp Analysis

The pulping liquor was collected every 30 min, before and after each extraction and injection, respectively, and at the pulping end. After cooking, the pulp was washed with distilled water to remove the liquor and to carry out other analysis. The residual alkali of the liquor was determined according to SCAN-N 2:88 (1988) and the solids content through TAPPI T625 (2014) and T650 (2015). Yields were calculated based on pulp and wood dried masses. The kappa number, hexenuronic acids, and viscosity of unbleached pulps were determined according to TAPPI T236 (1999), T282 (2013), and T230 (2007). Lignin kappa was calculated according to Li and Gellerstedt (1997), considering that 11.6 µmol/g of hexenuronic acids is equivalent to 1 point of kappa. Specific wood consumption (WSC) was determined by the relationship between wood basic density (BD) and the pulping screened yield (SY), and was calculated as follows:

$$WSC = \left(\frac{1}{BD \cdot SY}\right) \times 0.9 \tag{1}$$

Dry solids formed during the modified pulping were calculated considering the total pulping yield (TY), screened yield (SY), and active alkali (AA) and were calculated as follows:

$$solids = \frac{(1 - TY) + AA}{SY} \times 0{,}9 \qquad (2)$$

Oxygen Delignification and Bleaching

An oxygen delignification was performed using a laboratory reactor with 290 g of dried pulp. After that, elemental chlorine-free (ECF) bleaching was performed to obtain a brightness of 89.5 ± 0.5% ISO on bleached pulps through the A/D Eop D P sequence (Table 3), which is a bleaching sequence with acid extraction and chlorine dioxide delignification (A/D), oxidative extraction with hydrogen peroxide (Eop), bleaching with chlorine dioxide (D), and bleaching with hydrogen peroxide (P). After each stage, the pulp was washed with distilled water to complete the filtrate removal. All stages were performed in triplicate.

Table 3. Bleaching Conditions

Conditions/Charges	Bleaching Stages				
	O_2 delig.	A/D	Eop	D	P
Consistency (%)	12	12	10	10	10
Temperature (°C)	95 + 100	90 + 85	85	75	80
Time (min.)	10 + 60	120 + 13	60	120	120
Pressure (bar)	5.5	-	5	-	-
H_2O_2 (kg/a.d. ton)	-	-	5	-	3
ClO_2 (kg/a.d. ton)	-	kf = 0.2*	-	5 - 10**	-
NaOH (kg/a.d. ton)	15	-	8	-	5
O_2 (kg/a.d. ton)	18	-	4	-	-
H_2SO_4 (kg/a.d. ton)	-	6	-	-	-

*Chlorine dioxide charge using kappa factor (kf): *(delignified pulp kappa x kf x 10) / 2.63*
**Variable conditions; the charge depends on the brightness of each sample

Acid Extraction and Chlorine Dioxide Delignification (A/D)

This stage was performed in polyethylene bags in a thermostatic bath. The pulp sample was mixed with hot water, and the desired quantity of sulfuric acid and chlorine dioxide. The initial pH was measured, and the bag was sealed and inserted in the bath. After the reaction time, a filtrate sample was collected for the pH measurement.

Oxidative Extraction with Hydrogen Peroxide (Eop)

This stage was performed in a laboratory reactor. The pulp was mixed with hot water and the desired quantity of sodium hydroxide and hydrogen peroxide. The initial pH was measured, and oxygen was injected into the reactor. After that, to complete the total pressure of this stage, nitrogen was injected. After the reaction time, a filtrate sample was collected, and its pH was measured.

Bleaching with Chlorine Dioxide (D)

This stage was the same as the A/D method.

Bleaching with Hydrogen Peroxide (P)

This stage was performed in polyethylene bags in a thermostatic bath. The pulp was mixed with hot water and the desired quantity of hydrogen peroxide and sodium hydroxide for the pH control. The initial pH was measured, and the bag was sealed and inserted into the bath. After the reaction time, a filtrate sample was collected for the pH measurement.

Delignified and Bleached Pulps Analysis

The kappa numbers of oxygen-delignified pulps and the viscosity of oxygen-delignified and bleached pulps were measured following the same standards previously mentioned. Final pulp brightness was measured according to ISO 2470 (1999). Delignification efficiency calculus considers the kappa reduction after the oxygen delignification.

The total active chlorine and pulp bleachability were also calculated according to the equations below,

$$\text{Total Active Chlorine} = D \times 2.63 + P \times 2.09 \quad (3)$$

$$\text{Bleachability} = \frac{\text{kappa number before oxigen delignification}}{\text{Total Active Chlorine}} \quad (4)$$

where D is the total consumption of chlorine dioxide (ClO_2) on bleaching (kg/a.d. ton), and P is the total consumption of hydrogen peroxide (H_2O_2) on bleaching (kg/a.d. ton).

Refining and Physical Tests

Bleached pulps were refined in a PFI mill at four levels: 0, 750, 1500, and 3000 rotations. These pulps were evaluated through their physical properties in each refining level according to SCAN Standards.

RESULTS AND DISCUSSION

Pulping

The pulping results are shown in Table 4. For an increase in delignification level (kappa reduction), a raising of the alkali charge and also of the H factor were necessary. In this work, the rise in the H factor was achieved by increasing the maximum cooking temperature, while the time was the same in all experiments.

The increase in applied active alkali charge and in H factor also resulted in a decrease in yield and in pulp with lower viscosity, both effects that were related to higher pulp degradation with these cooking modifications. Pulps with higher delignification levels present higher hexenuronic acids content, showing that the formation of these compounds is increased under more severe cooking conditions (Colodette *et al.* 2002). As already known, the formation and degradation of hexenuronic acids are directly related to pulping conditions, mainly the H factor and alkali charge (Pedroso and Carvalho 2003; Gustavsson 2006). Antes and Joutsimo (2015) studied different kappa levels for *E. nitens* and *E. globulus* pulps and reported that the impact of HexA on the overall kappa number is more significant for pulp at kappa 14 than for pulp at kappa 18.

The lignin kappa is a useful index to indicate the amount of lignin present in pulp, excluding the hexenuronic acids from the kappa number (Fatehi *et al*. 2009). This parameter is related to pulp bleachability. In this work, the difference between the regular kappa numbers was 16.5%, while the difference between lignin kappa numbers was 24.6%.

The specific wood consumption was based on wood basic density and on pulping yields, indicating the wood volume necessary to produce a specific pulp mass (Magaton *et* *al*. 2009; Sable *et al*. 2012; Segura and Silva Júnior 2016). On lower kappa pulping, the yield reduction was responsible for the increase of wood specific consumption.

Table 4. Pulping Results

Parameter	Kappa 18	Kappa 15
Kappa Number	17.7	14.9
Active Alkali (%)	17.5	19.0
Maximum Temperature (°C)	151	153
H Factor	680	805
Total Yield (%)	56.1	55.0
Screened Yield (%)	56.1	55.0
Hexenuronic Acids (µmol/g)	45.8	52.4
Lignin Kappa	13.8	10.4
Viscosity (cm^3/g)	1,317	1,227
Specific Wood Consumption (m^3/a.d. ton)	3.42	3.49
Dry solids (tons/a.d. ton)	0.98	1.05

Dry solids, which represent the amount of solids available for burning in the recovery boiler (Segura and Silva Júnior 2016), is a parameter influenced by the kappa number of the pulps. Higher kappa represents lower solids generation that is a benefit considering the recovery boiler as production bottleneck. The alkali profile during the cooking is presented in Fig. 1.

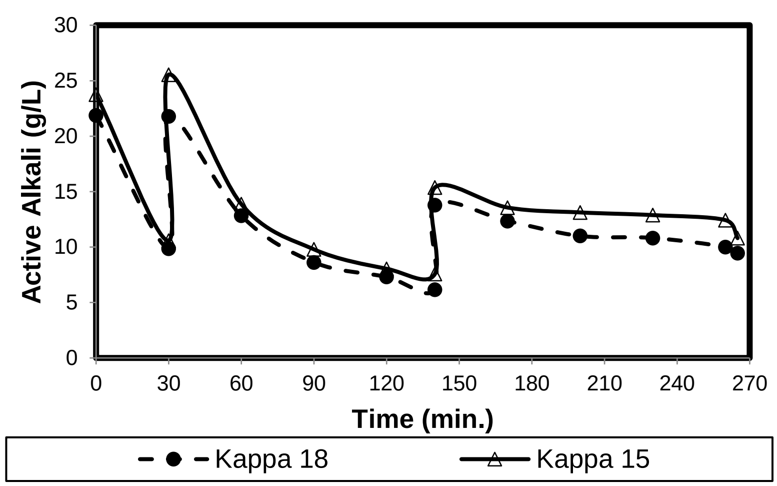

Fig. 1. Alkali profile

During the impregnation, there was high alkali consumption caused by the neutralization of acid composites formed by the wood-alkali reaction. After the first injection, there was an increase in active alkali. The alkali consumption rate remained high for 30 min followed, when the initial delignification take place, followed by a reduction in the alkali consumption rate. The second injection again increased the alkali concentration of the cooking liquor. From this moment, the final delignification occurred with a low rate of alkali consumption, representing the residual delignification phase (Almeida *et al*. 2014). The impregnation phase was responsible for 37% of the total alkali consumption during pulping, while the consumptions after the first and second liquor injections were 50% and 13%, respectively.

The black liquor total solids content during the cooking is presented in Fig. 2.

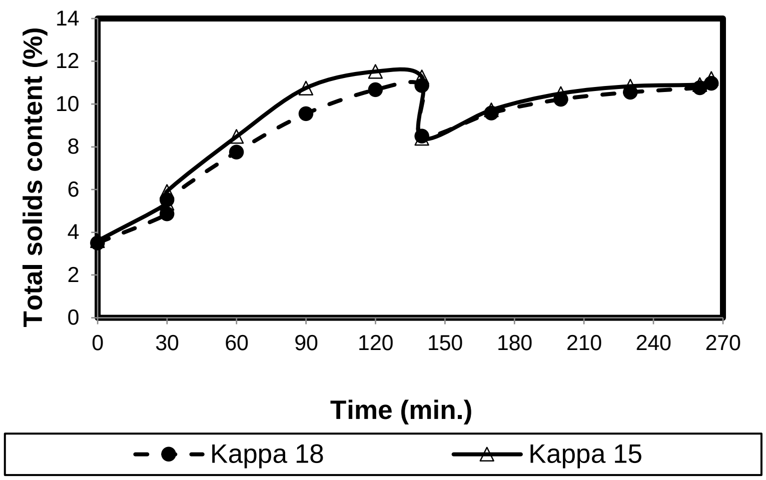

Fig. 2. Liquor total solids profile

During the first 30 min of cooking, when impregnation was being performed, an increase in black liquor solids content was observed. After 30 min, the first liquor change had finished: the black liquor, rich in organic solids, was partially replaced by clean white liquor. After this change, despite the black liquor removal, the total solids content was increased due to the injection of the white liquor in the inorganic solids.

The main delignification phase started with the increase of temperature and liquor total solids. During these 110 min, the liquor solids content increased, decreasing when the second liquor change was done. From this moment to the pulping final, the solids on liquor slightly increased, due to the residual delignification.

Oxygen Delignification and Bleaching

The oxygen delignification was commercially introduced in the 1970s with the main objective of reducing the amount of residual lignin in pulp. The results of oxygen delignification are shown in Table 5.

Pulps with the unbleached kappa of 18 maintained a higher kappa after the oxygen delignification (11.5 against 9.5). These pulps also presented lower brightness (45.0 % ISO to 47.8 % ISO), higher viscosity (1,144 cm^3/g against 1,038 cm^3/g), and lower hexenuronic acids content (38.7 µmol/g to 48.1 µmol/g). The oxygen delignification efficiency reflected the pulp kappa reduction after this step, and the unbleached kappa number presented a direct influence on this parameter. In the present work, the pulps with an initial kappa of 15 had higher delignification efficiency than the pulps with an unbleached kappa of 18, which confirmed the results of Lai *et al.* (1998). These authors stated that the delignification efficiency was improved when the pulp kappa number decreased. According to Colodette *et al.* (2007), the lignin of the higher kappa pulps showed lower amounts of free phenolic groups, which were the main targets of the oxygen reactions. In this way, the higher kappa pulp delignification efficiency is in general lower.

Table 5. Oxygen Delignification Results

Parameter	k = 18	k = 15
Kappa number	11.5	9.5
Oxygen delignification efficiency (%)	34.8	36.6
Brightness (% ISO)	45.0	47.8
Hexenuronic acids (µmol/g)	38.7	48.1
Lignin Kappa	8.2	5.3
Viscosity (cm^3/g)	1,144	1,038

After the delignification, an ECF bleaching was performed. The main objective of the bleaching was to increase the pulp brightness removing some pulp chromophore compounds such as lignin, resins, metallic ions, and non-cellulosic carbohydrates (Singh 1979) using different reagents. Most of these reagents are oxidants, and they provided more stable pulp brightness. The bleaching results and chemical consumptions are shown in Table 6.

Table 6. Bleaching Results

Parameter	k = 18	k = 15
Brightness (% ISO)	89.7	89.6
Viscosity (cm^3/g)	963	899
Chlorine dioxide consumption, as active chlorine (kg/a.d. ton)	16.8	12.2
Hydrogen peroxide consumption (kg/a.d. ton)	8.0	8.0
Bleachability (kappa number / TAC)	0.290	0.305

The initial kappa number had a direct influence on the bleaching process and bleached pulps characteristics. Pulps with an initial kappa of 18 presented higher final viscosity, which reflected the pulping conditions and followed the viscosities of unbleached pulps. On the other hand, these pulps demanded higher chemical application to achieve 89.5 ± 0.5% ISO brightness than the pulps with an initial kappa of 15. As bleachability can be defined as the ease with which a determined pulp can be bleached (Martino *et al*. 2013), in this study it was calculated considering a ratio between the kappa number after the delignification and the Total Available Chlorine, pulps with a higher initial delignification degree (lower kappa) were more easily bleached.

Refining and Physical Tests

The bleached pulps were refined in four levels (0, 750, 1500, and 3000 revolutions) and had their physical mechanical properties evaluated. The drainability of the pulp was increased with the refining intensity. Pulps with a higher unbleached kappa number needed less refining energy when compared to pulps with a lower initial kappa number.

The tension index and burst index of the pulps at different refining levels are observed in Fig. 3, tear index and Klemm capillarity are presented in Fig. 4, while pulp specific volume is presented in Fig. 5.

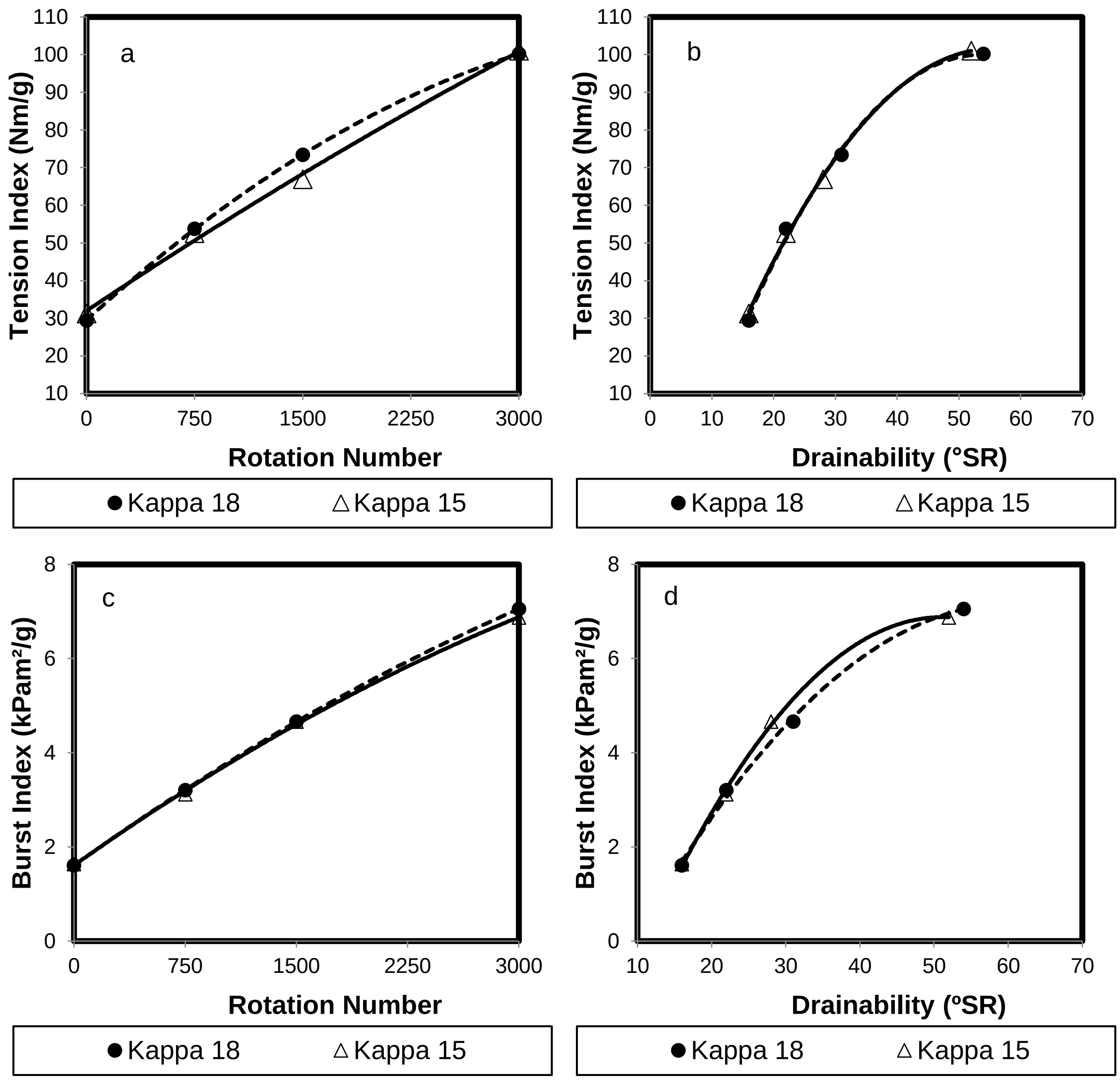

Fig. 3. (a, b) Tension index, (c,d) Burst index of bleached pulps

The tensile strength index and burst index are important properties for papermaking pulps because they indicate the probability of sheet break during production and have an impact on paper quality. As expected, the tensile index and burst index of all of pulps increased with the refining increase, achieving maximum values on drainability between 40 and 50 °SR. There were no observed differences between the pulps with different unbleached kappa numbers for these properties.

The tear index also increased with refining increase, reaching the maximum level at drainability of 40 °SR, and decreasing after this level for all pulps. According to Sarto *et al*. (2015) the tear resistance is related to the fibers' transverse dimensions, which are of fundamental importance in short fiber pulps.

Based on the results of this work, it can be concluded that the excessive refining is prejudicial for the tear index, matching to the results found by Cowan (1995) and Mansfield and Kibblewhite (2000). This property was not influenced by the unbleached kappa number of the pulp.

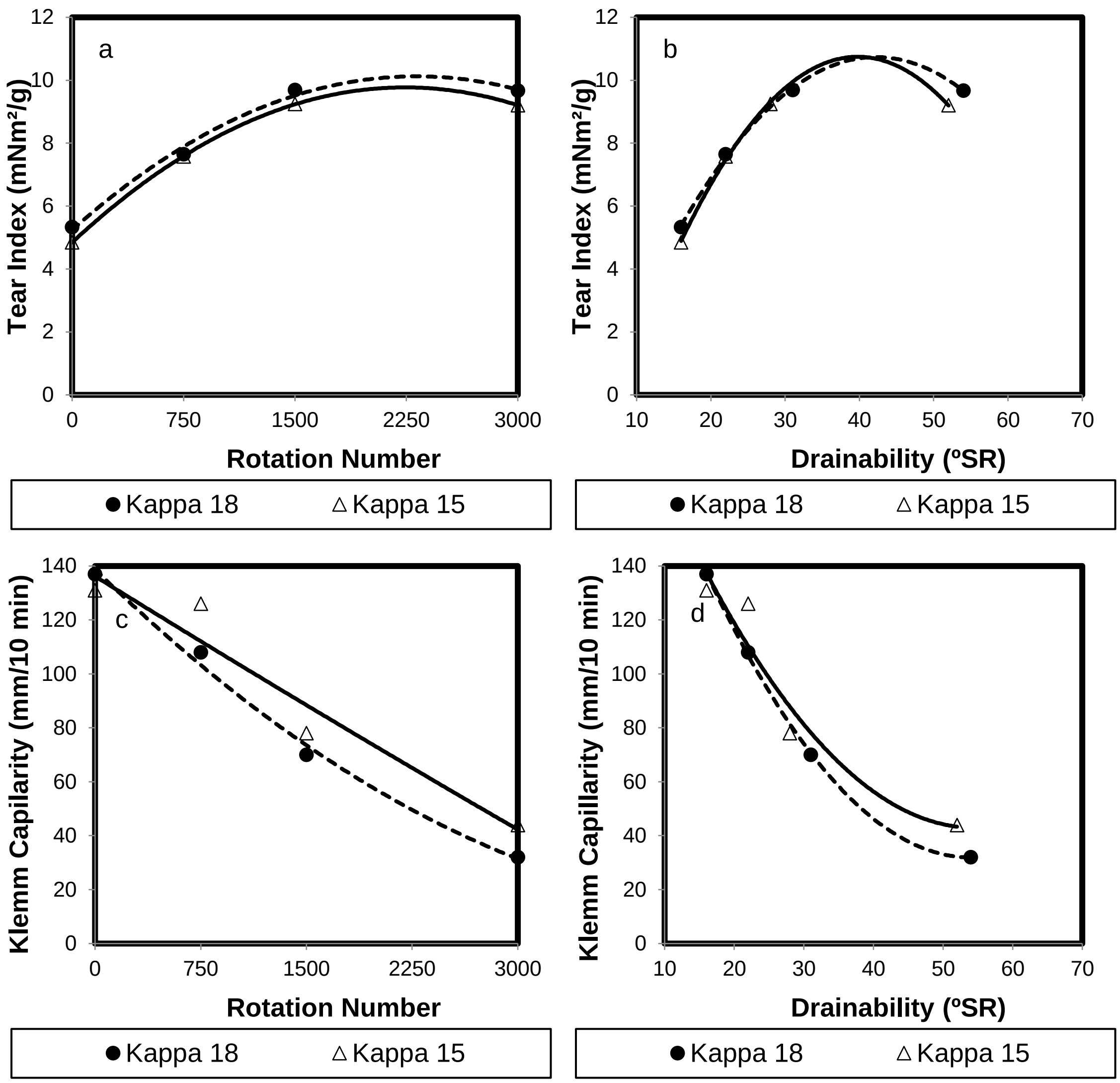

Fig. 4. (a, b) Tear index, (c, d) Klemm capillarity and (e, f) Specific volume of bleached pulps

The Klemm capillarity is an important property with respect to the pulp drying process and the use of pulp in absorbent paper production (tissue) (Sarto *et al*. 2015). In this work, this property decreased with the refining evolution, stabilizing at 50 °SR. It was observed that pulps with an initial kappa of 15 presented higher capillarity than pulps with an unbleached kappa of 18. This result indicates that bleached pulp with initial kappa 15 has less united and greater amounts of empty spaces than pulp with initial kappa 18 (Gomes *et al*. 2011).

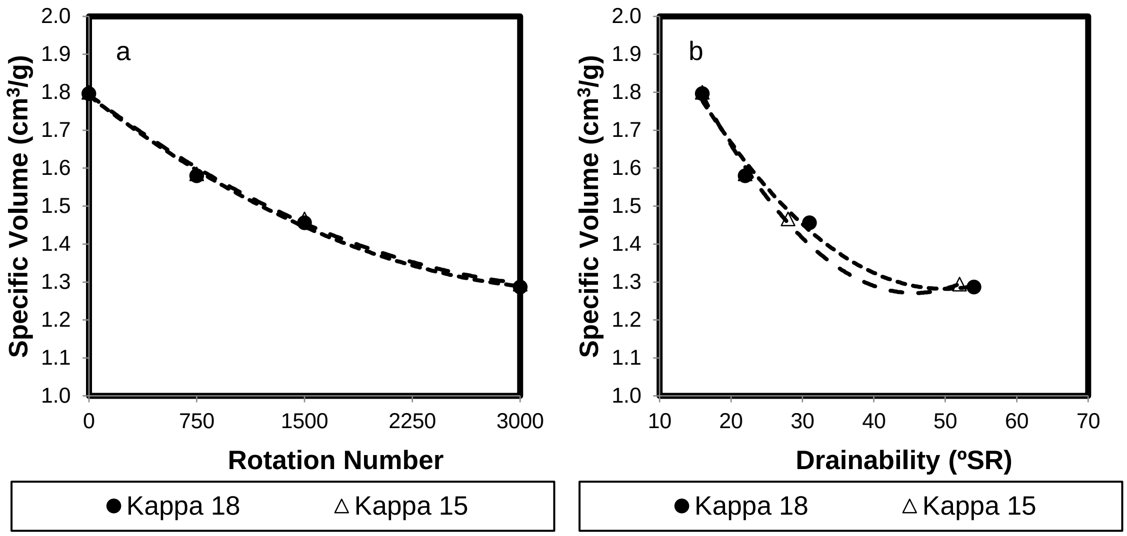

Fig. 5. Specific volume of bleached pulps (a, b)

The specific volume, also known as "bulk" is a quite prized property by paper manufacturers and is directly related to fiber morphology and refining intensity (Sarto *et al*. 2015). For all pulps, the bulk was reduced with the refining, stabilizing at 40 °SR. There were no observed differences between the pulps with different initial kappa numbers.

CONCLUSIONS

1. The delignification increase demanded high H factor and alkali change, which reduced the pulping yields and pulp viscosity, thus increasing the specific wood consumption.
2. Oxygen delignification was directly influenced by kappa variation; it was more efficient in pulp with a lower unbleached kappa given the studied kappa ranges.
3. The more delignified pulps presented lower reagent consumption on bleaching. Otherwise, the bleached pulps with an unbleached kappa of 15 presented lower viscosity than pulps with an initial kappa 18.
4. The more delignified pulps demanded higher refining energy.
5. Pulps with an initial kappa of 15 presented higher capillarity than pulps with an unbleached kappa of 18. The other physical properties of the pulps were not influenced by initial kappa variation.
6. The reduction of the brownstock kappa number from 18 to 15 was not justified based on pulping results. Also, the physical properties were slightly influenced by unbleached kappa variation considering the studied kappa ranges.

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