Potential of Elephant Grass for Pulp Production

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Elephant grass (*Pennisetum purpureum*) (EG) is a fast-growing plant with high biomass productivity in the range of 30-45 bone dry t/ha/yr. This high productivity qualifies EG as a potential raw material for bleached pulp production. This study evaluated elephant grass as a raw material for paper pulp production. This was accomplished through determining its full chemical and morphological characterization, pulpability at kappa numbers 15 and 20 via the kraft and soda-AQ processes, and its pulp bleachability to 90% ISO brightness. The results were compared with those of a commercial hybrid eucalyptus wood clone (*Eucalyptus urophylla* x *Eucalyptus grandis*) (EUCA) that is widely planted in Brazil. Concerning its chemical composition, the elephant grass presented a high ash (60,100 mg/kg) and total extractives content (14.8%). However, the elephant grass showed good potential for pulp production. The kraft process was the ideal cooking process at kappa number 20, producing the highest screened yield (47.9%), bleachability (0.163 Δkappa/TAC), and good viscosity (812 dm^3/kg). For EUCA, the ideal cooking process was the kraft process at kappa number 20, resulting in a screened yield of 52%, bleachability of 0.217 Δkappa/TAC, and final viscosity of 886 dm^3/kg. This high productivity qualifies EG as a potential raw material for bleached pulp production.

Keywords: Pennisetum purpureum; Elephant grass; Eucalypt; Soda-AQ; Kraft; Pulp

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INTRODUCTION

Hardwoods and softwoods have been the world's main raw materials for pulp production because they deliver suitable product quality. However, the cost of these raw materials has greatly increased in the last decade due to many factors, including the newly created demand for biorefinery applications, and researchers have been searching for new raw materials that can compensate for the lack of low cost wood. The biomass quality is of extreme importance for pulp production, that is, high yield, low cost, and high quality. It is difficult to find raw materials that are better than wood as a source of fiber for pulp production. High productivity plants, such as elephant grass (30 to 45 bone dry t/ha/yr), can potentially supply low-cost biomass to meet the current demand (Vilela *et al.* 1998, 2001a,b, 2002a,b, 2003a,b; Paulino *et al.* 2007; Mazzarella 2007). However, the quality of such raw material for pulp production is not yet fully known.

Among the many nonwoody raw materials used to make pulp, which represents a market share of about 8 to 10% of the world's pulp production, there is no reference for the commercial use of elephant grass (González *et al.* 2008; Alaejos *et al.* 2004; Hammett *et al.* 2001; Jiménez *et al.* 2006; Johnson 1999; Rezayati-Charani *et al*. 2006).

Elephant grass has beneficial characteristics for pulp production, such as high fibers production (similar to sugar cane) and its chemical composition (Prinsen *et al*. 2012; Del Río *et al*. 2012; Xie *et al*. 2011). Some works have shown contents of 40%, 30%, and 17.7% for cellulose, hemicelluloses, and lignin, respectively (Madakadze *et al*. 2010). These values are good for pulp production, especially the low lignin content, suggesting high pulpability of this material in cooking processes. The aforementioned studies indicate that EG has potential for paper production, but a more thorough investigation is required, particularly comparing its potential with that of the well-known eucalyptus wood, which is planted in Brazil and many other parts of the world.

The aim of this study was to evaluate elephant grass as a raw material for paper pulp production through its full chemical and morphological characterization, pulpability at kappa 15 and 20 *via* the kraft and soda-AQ processes, and its bleachability to 90% ISO brightness. The results were compared with those of a commercial hybrid eucalyptus wood clone (*Eucalyptus urophylla* x *Eucalyptus grandis*) that is widely planted in Brazil.

EXPERIMENTAL

Materials

A 500 kg sample of 150-day-old elephant grass (*Pennisetum purpureum*) (EG) harvested at a Federal University of Viçosa experimental station and a 500 kg sample of eucalyptus (*Eucalyptus urophylla* x *Eucalyptus grandis*) (EUCA) wood collected at seven years of age were supplied by a Brazilian forest company and used throughout the study. The samples were evaluated for their moisture content at the moment of harvesting according to TAPPI T264 cm-97 standard procedure. The EG sample was manually chipped, producing wet chips about 5 cm long. The chip thickness was quite variable, since it depended on the grass thickness, which varied from 1 cm to 4 cm in diameter. The grass chips were air-dried to a moisture content of about 15% and stored in large plastic bags. The eucalypt sample was chipped in a laboratory chipper (Chogokukikai model) equipped with 3 knives and 2 screens (40 and 13 mm). Both EG and EUCA chips were well mixed (260 m^3 rotary mixer) and the EUCA sample was screened according to SCAN-CN 40:94 procedure. For the EUCA sample, the chips retained in the 3 mm and 7 mm screens were collected and mixed again, air dried to about 15% moisture content, and stored in large plastic bags.

Methods

The sampling for physical, chemical, and morphological analyses was done using the so-called quartering technique. Chips were used to measure bulk and basic density according to SCAN CN-46:92 and SCAN CM-43:95 standard procedures, respectively.

Biomass productivity

The biomass productivity was calculated using the medium annual increase (MAI) and basic density with the following equation:

$$\text{Biomass productivity } \left(\frac{\text{ton/ha}}{\text{yr}}\right) = \text{MAI } \left(\frac{\text{m}^3\text{/ha}}{\text{yr}}\right) \times \text{basic density } \left(\frac{\text{ton}}{\text{m}^3}\right) \quad (1)$$

Sample preparation for morphological analysis

About 200 grams of biomass were sliced into toothpick-type material and macerated using nitro-acetic acid solution in order to prepare individual fibers for morphological analysis. To make the maceration, a solution of five parts acetic acid and one part nitric acid were mixed and added to the biomass material until they were completely immersed; it was then placed under a hood for 6 h at 100 °C. The reaction was stopped by washing the material, which was dispersed in distilled water. Next, the material was gently mixed in a magnetic stirrer (slowly and steadily) for 60 min so that all of the fiber bundles were separated. Morphological characterization of fibers, vessels, and fines was carried out on a pulp suspension. For the analysis of the morphological characteristics of fibers, the pulp suspension was deposited on a glass slide and analyzed by the CyberMetrics analyser; this operation was done automatically using a Sample Maker apparatus. The Sample Maker allowed for the automatic deposition of the fibers on a glass slide with or without metallic calibrated wires. This apparatus eliminated the effect of the operator and applied a constant pressure, which was the same for all the preparations. The glass slide was then introduced into the CyberMetrics analyser. The fibers were analysed by a microscope lens coupled to a CCD camera (charged coupled device). The fiber characteristics were measured with the following software packages: CyberFlex, CyberBond, and CyberSize. These analyses allowed reliable measurements of thousands of fibers, vessels, and fines to determine the main morphological and dimensional characteristics of the pulp components.

Sample preparation for chemical analysis

For chemical analysis, about 1 kg of EG and EUCA biomass was sampled and ground in a Wiley type mill to produce sawdust of variable size. This sawdust was screened according to TAPPI standard T257 cm-85. The sawdust that passed through the 40 mesh screen and was retained in the 60 mesh screen was selected for chemical analysis. The sawdust was air-dried and conditioned in a temperature and humidity controlled room (23 ± 1 °C, 50 ± 2% RH) until equilibrium moisture content was achieved (~10%). This sawdust (raw sawdust) was used for chemical analysis. The analyses of ash, silica, chloride, iron, copper, manganese, potassium, calcium, and magnesium were carried out directly on the raw sawdust, according to the Standard Methods for the Examination of Water and Wastewater (1998), except for chloride, which was determined according to the TAPPI T256 cm-07 standard procedure. The biomass extractives contents were determined in the raw sawdust using three different methodologies: (1) in acetone; (2) ethanol/toluene (1:2) and; (3) ethanol/toluene (1:2) →ethanol →hot water solvent series, according to TAPPI T280 wd-06, TAPPI T204 cm-07, and TAPPI T264 cm-07 standard procedures, respectively. In order to determine the main cell wall components of the biomass, a 200 g sample free of extractives was prepared using TAPPI T264 cm-07 standard procedure. This sample (extractive-free sawdust) was conditioned in a temperature- and humidity-controlled room (23 ± 1 °C, 50 ± 2% RH) until equilibrium moisture content was achieved (~10%). The contents of uronic acids, acetyl groups, and sugars (glucans, mannans, galactans, xylans, and arabinans) in the extractive-free biomass were determined according to Scott (1979), Solar *et al.* (1987), and Wallis *et al.* (1996). The acid-insoluble lignin, acid-soluble lignin, and syringyl/guaiacyl (S/G) ratio were determined according to TAPPI T222 om-11 standard procedure and by nitrobenzene oxidation (Goldschmid 1971; Lin and Dence 1992).

Pulping processes and conditions

Two pulping processes were used to convert EG and EUCA into pulps, namely, kraft and soda-AQ processes. The EG and EUCA cooking trials were done in an M & K digester with two individual reactors of 10 L each, equipped with a forced liquor circulation system, and electrically heated with temperature and pressure control. The digester was coupled to a cooling system (coil system with residual liquor, surrounded with water at room temperature working as a cooling mechanism) to ensure the cooling of the liquor after the cooking simulation. The experimental conditions used in the cooking process were established based on the technical knowledge of the Pulp and Paper Laboratory of the Federal University of Viçosa-Brazil. With the exception of the alkaline charge, the other cooking conditions were kept constant and are shown in Table 1. Eight cooking experiments were performed for each sample, using different active alkali charges to establish the delignification curves for each sample. Eight cooking trials were performed for each sample to establish the delignification curve. After determination of the delignification curve, four samples of pulp were prepared for each raw material from the two processes and two kappa numbers per process, 15 and 20.

Table 1. Conditions for Pulping Processes of EG and EUCA with Kappa Numbers 15 and 20

Parameter	Kraft process	Soda-AQ process
Biomass, kg	1	1
Active alkali as NaOH, % on o.d. chips*	Variable	Variable
Sulfidity as NaOH, % on o.d. chips	26	0
Anthraquinone charge, % on o.d. chips	0	0.05
Liquor to chips ratio, L/kg	4/1	4/1
Maximum temperature, °C	170	170
Time to maximum temperature, minutes	90	90
Time at maximum temperature, minutes	50	50
*The optimum AA for achieving kappa numbers 15 and 20 was determined by varying the AA charge from 15.5-23.1% and 13.8-27.7% for the kraft and soda-AQ processes.		

Fiber individualization was achieved in a "hydrapulper" (capacity 15 L), followed by fines screening (Voith laboratory cleaner) equipped with perforated plates with 0.2-mm openings. The material retained on the sieve (rejects) was dried and weighed. The clean pulp was dewatered in a centrifuge to a consistency of about 30%, weighed, and stored in polyethylene bags for further analysis. With the known weights of the sieved and retained materials, the reject content and the screened cooking yield were determined. The hexenuronic acid content, heating value, and solids content were determined according to Vuorinen *et al*. (1996), TAPPI T684 om-11, and CPPA H1, respectively.

Bleaching sequence and conditions

The pulps cooked at kappa numbers 15 and 20 were bleached by the elemental chlorine-free sequence (ECF) O-D-P-D to 90% ISO brightness. The oxygen delignification (O stage) was run at 10% consistency, 100 °C, 60 min, 700 kPa pressure,

20 kg NaOH/odt pulp, and 20 kg O_2/odt pulp. The first chlorine dioxide stages (D) were carried out at 10% consistency, end pH 3.5, 85 °C, 120 min, with a kappa factor of 0.20 for pulps with kappa number 15 and 0.24 for pulps with kappa number 20. The P stages were run at 10% consistency, end pH 10.5, 85 °C, 120 min, with hydrogen peroxide doses of 0.5 by pulp weight. The second chlorine dioxide stages (D) were carried out at 10% consistency, end pH 5.5, 70 °C, 120 min, with variable amounts of chlorine dioxide to achieve the desired brightness. The bleached pulps were evaluated for their brightness, brightness stability, and viscosity. The brightness stability expressed as post color number was calculated according to TAPPI TIS 017-10.

RESULTS AND DISCUSSION

Biomass Productivity

Two very important factors regarding biomass use for pulp production are the moisture content and the density, since they affect harvesting, transportation, and utilization costs (Leite *et al.* 2010). In Table 2, the biomass productivity results of EG and EUCA are presented. The samples analyzed in this study showed average moisture contents of 73 and 55% and densities of 216 and 480 kg/m^3 for EG and EUCA, respectively. These values are considered satisfactory for pulp production (Gomide *et al.* 2005; Gomide *et al.* 2010). A high density is always favorable in pulp production because it increases pulp mill throughput, but it may penalize pulping yield due to poor white liquor penetration when conditions are not properly optimized (Batalha *et al.* 2012).

These results point out the great difficulty in the industrially processing of EG materials and to the need to develop methods for compacting them to increase their density before utilization. The MAI for the EG sample was 148.1 m^3/ha/yr, but its low density (216 kg/m^3) resulted in a productivity of only 32 tons of material per ha/yr, which was much lower than the EUCA sample. The MAI of the EUCA sample was 80.9 m^3/ha/yr, giving a productivity of 38.8 tons of wood per ha/yr. This productivity was much higher than the average obtained in commercial plantations in all Brazilian Territory (~20-30 t/ha/yr) (Bracelpa 2013). However, the EG biomass productivity is quite satisfactory when compared, for example, with the Brazilian average for eucalyptus.

Table 2. Biomass Productivity for EG and EUCA

Sample code	Average annual increment, m^3/ha/yr	Biomass basic density, kg/m^3	Biomass productivity, bone dry ton/ha/yr	Chip bulk density, kg/m^3
EG	148.1	216	32.0	85
EUCA	80.9	480	38.8	183

Morphological Characteristics

Another important aspect of raw material for pulp production is its morphological characteristics. The strength and morphology of fibers have a strong impact on the physical properties of paper (Seth and Page 1988). In Table 3, the morphological characterizations of EG and EUCA are presented. A lower fiber content per gram of pulp in the EG sample was observed. The width, length, coarseness, fines content, and macro fibrillation index showed values that were close to the eucalyptus sample. However, EG

showed a high vessel content. For the pulping process, the vessel elements are desirable, since they make the penetration of cooking liquors easier. However, for the production of special kinds of paper, such as printing papers, they are considered undesirable because the vessels on the surface of the paper sheet tend to be pulled, thereby causing printing failures known as "vessels picking" (Lindström and Fardim 2012). In general, it is possible to say that the fibers have potential for paper production, since the requisite characteristics for paper quality have been met.

Table 3. Morphological Characterization of EG and EUCA Materials

Sample	*EG	**EUCA
Fibre content, millions/g of pulp	14.9	28.1
Mean fibre arithmetic length, µm	695.5	562
Mean length-weighted fibre length, µm	1131	733
Mean area-weighted length, µm	1114	733.5
Mean fibre width, µm	21.5	16.4
Mean fibre coarseness, mg/m	0.09	0.06
Mean fibre curl index, %	6.8	4.5
Macro--Fibrillation index, %	0.42	0.54
Broken fibre content, %	26.1	17.7
Fine content, % in area	11.9	12.4
Mean fine area, μm^2	1,987	1,647
Mean fine length, µm	62	64.5
Vessel content, nb/g of pulp	11,628	6,771
Mean area-weighted length, mm	0.45	0.43
Mean vessel width, µm	191.1	194.3

Chemical Characteristics

Table 4 shows the extractives contents of the EG and EUCA samples extracted using the three techniques that were given in the methodologies: (1) ethanol/toluene → ethanol → hot water solvent series, (2) ethanol/toluene 1:2, and (3) acetone. In order to measure the biomass cell wall components, it was necessary to remove all extractives present in the material. The TAPPI T264 cm-07 standard procedure (ethanol/toluene 1:2 → ethanol → hot water) was sufficient for removing all polar and apolar extractive fractions. Although this procedure was intended to free the wood from extractives, it also served to quantify the total amount of extractives present in the biomass, since the main cell wall components (cellulose, hemicelluloses, and lignins) were not soluble in any of the solvents that comprised the series. Extraction with ethanol/toluene only extracts waxes, fats, resins, phytosterols, and non-volatile hydrocarbons. Extraction in acetone (TAPPI T280 wd-06) serves to quantify those extractives that are more relevant to the pulping operation and pitch formation in the pulp. The acetone-extractable content of wood is a measure of such substances as fatty acids, resin acids, sterols, waxes, and non-volatile hydrocarbons. Because acetone is both more polar and water-miscible than dichloromethane or benzene-ethanol, the quantity of acetone-extractable material, especially in wood, may be higher than that found from the other solvents. This procedure does not give the same results as ethanol-toluene or dichloromethane

extractions. In his work, Barbosa *et al.* (2005) showed that acetone is the best solvent for the evaluation of wood's lipophilic extracts content.

Biomass extractives are quite troublesome since they cause many difficulties in the operation of industrial facilities. For example, unexpected lost time in operations may be necessary to clean the equipment and instruments due to stickiness and tackiness. In addition, the deposition of substances called pitch in the pulp may occur (Barbosa *et al*. 2005), decreasing the pulp value or even causing its rejection by the market. In her study, Cruz *et al.* (2006) showed that waxes, fats, and long chain alcohols are the main compounds associated with pitch formation.

The evaluated EUCA material showed acceptable acetone extractives, ethanol/toluene (1:2) extractives, and total extractive contents for pulp mills (Gomide *et al.* 2010). For pulp production, EG showed a high content of acetone extractives and ethanol/toluene extractives. Additionally, pitch formation and pulp dirtiness are much more likely in raw materials containing high contents of these extractives. As a consequence, EG also presented a high total extractives content (14.8%). Such materials are likely to result in a low yield during the cooking process.

Table 4. Extractives Content of EG and EUCA Materials

Sample code	Acetone extractives, %	Ethanol/toluene (1:2) extractives, %	Total extractives, %
EG	3.9	6.8	14.8
EUCA	0.8	1.5	2.3

The mineral contents of raw materials are detrimental for industrial utilization since they cause corrosion and deposits on equipment, reduce the biomass heating value, and decrease mill throughput. In general, the amount of inorganics present in eucalyptus woods is very low and acceptable for most applications (Moreira 2006). The total inorganics measured by complete biomass combustion (ash content) of EUCA was 1,550 mg/kg biomass (Table 5). It reached 60,100 mg/kg for EG. The high mineral content in the grass is explained by its fast metabolism at a young age. The mineral contents in biomass tend to decrease with age, due to the decreased biomass deposition rate as a function of time (Morais 2008). The same trend observed for total inorganics (ash content) was also verified for the individual components, such as silica, chloride, calcium, potassium, and magnesium, with the EG always presenting higher values than the EUCA sample. Calcium, magnesium, and silica are undesirable in most industrial processes because of their ability to cause deposits in equipment during evaporation of liquid streams and combustion of solid streams. However, potassium and chloride are particularly dangerous due to their ability to decrease the ash melting point during combustion, thus causing sticky ash problems in recovery boiler systems (Wessel *et al.* 2002). In addition, chlorides are highly corrosive and troublesome for most equipment, regardless of metallurgy. In regard to transition metals (Fe, Cu, and Mn), there were also significant differences between the EG and EUCA materials. For EG, these contents were 8.8, 11.2, and 11.1 mg/kg for copper, iron, and manganese, respectively. For the EUCA materials, the copper, iron, and manganese contents were 1.1, 9.3, and 18.2 mg/kg, respectively. Transition metals are particularly important in operating systems where oxygen-derived chemicals are used for processing biomass. These metals are aggressive toward oxygen-oxygen bonds, thus degrading any peroxide form and subsequently forming highly reactive free radical intermediates that negatively affect the integrity of

carbohydrate chains. Concerning the high grass inorganic content, some pretreatments and processes may be used for removing it. For example, in alkaline pulping processes, with the purpose of removing metals which are not soluble in alkaline solution, a pretreatment using acid leaching of the raw material has presented good results (Del`Antonio *et al.* 2011). For removing metals which are soluble in alkaline solution aiming a feasibility of the process, for example, the dissolved silica in the black liquor could be removed from the recovery cycle by precipitating it from weak black liquor with flue gases or by two-stage causticising (Finel 2003).

Table 5. Metal Contents, Cl^-, and SiO_2 of EG and EUCA Materials

Sample code	Inorganics, mg/kg biomass								
	Ash	Cu	Fe	Ca	Mn	Mg	K	Cl^-	SiO_2
EG	60,100	8.8	11.2	423	11.1	490	21,194	6,631	15,167
EUCA	1,550	1.1	9.3	378	18.2	104	369	434	-

Table 6 presents the contents of sugar, acetyl groups, uronic acid, lignin, and syringyl/guaiacyl ratio of lignin. The lignin contents for EG and EUCA biomasses were 18 and 27.2%, respectively. These values are acceptable for pulp production. The low lignin content of EG is advantageous for potentially improving pulping ease and yield (Gomes *et al.* 2008). However, its low lignin S/G ratio and low sugar content work to its detriment, since these characteristics are not desirable for pulping processes, as low lignin S/G ratio implies higher difficulty of delignification, and added to its low sugar content results in yield loss (Gomes *et al.* 2008). The low sugar content of EG reflected its very high extractive and mineral contents.

Table 6. Chemical Composition of EG and EUCA Materials

Sample code	*Sugar composition, %					Acid-soluble lignin, %	Total lignin, %	Lignin S/G ratio	Acetyl group, %	Uronic acid group, %
	Gluc.	Xyl.	Galac.	Man.	Arab.					
EG	38.2	9.6	0.8	0.6	0.2	2.2	18	0.8	1.9	1.2
EUCA	49.4	12	1.2	0.9	0.3	4.2	27.2	2.7	1.9	4.0
*Sugar composition: Gluc.= glucans; Xyl. = xylans; Galac. = galactans; Man. = mannans; Arab. = arabinans										

In general, both the EG and EUCA hemicelluloses were largely comprised of xylans with some minor components (mannans, galactans, and arabinans) of little importance. EG showed a lower uronic acid content than EUCA, which can be negative for this raw material, since uronic acids protect xylans, preserving them during alkaline pulping processes (Magaton *et al.* 2008).

Pulpability of EG and EUCA by the Kraft and Soda-AQ Processes

The pulpabilities of EG and EUCA were compared at kappa numbers 20 and 15, using the kraft and soda-AQ processes. Kappa number targets of 15 and 20 were chosen because they are widely used for the production of bleachable-grade hardwood pulps. The kappa number 15 is more conventional, but the kappa number 20 is desirable for fiber line yield improvements; however, dealing with it is more challenging in the mill

screening and bleaching areas due to the high lignin content, which affects these operations. The kraft process is more conventional, while the soda-AQ process is desirable when black liquor is being used for biorefinery purposes.

In order to achieve these kappa number targets for both processes, the raw materials were cooked at various active alkali doses, and a delignification curve was determined whereby kappa number was plotted against active alkali charge (Figs. 1 and 2 for EG and EUCA, respectively) and yield (Figs. 3 and 4, for EG and EUCA, respectively). For both processes (soda-AQ and kraft), increasing the alkali charge decreased the kappa number and increased the screened yield.

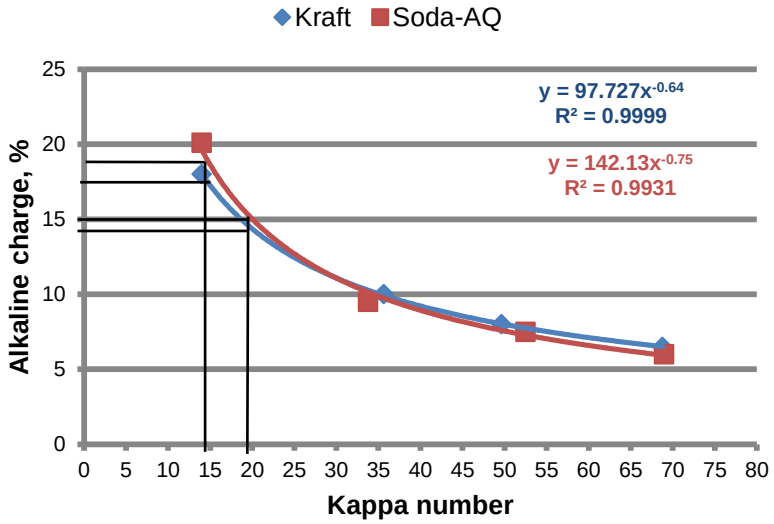

Fig. 1. Delignification curve by kraft and soda-AQ processes for EG. Pulping conditions: 26% of sulfidity as NaOH only used to Kraft process; 0.05% of anthraquinone charge only used to Soda-AQ process; 4/1liquor to chips ratio; maximum temperature of 170 °C; time to maximum temperature of 90 min.; time at maximum temperature of 50 min.

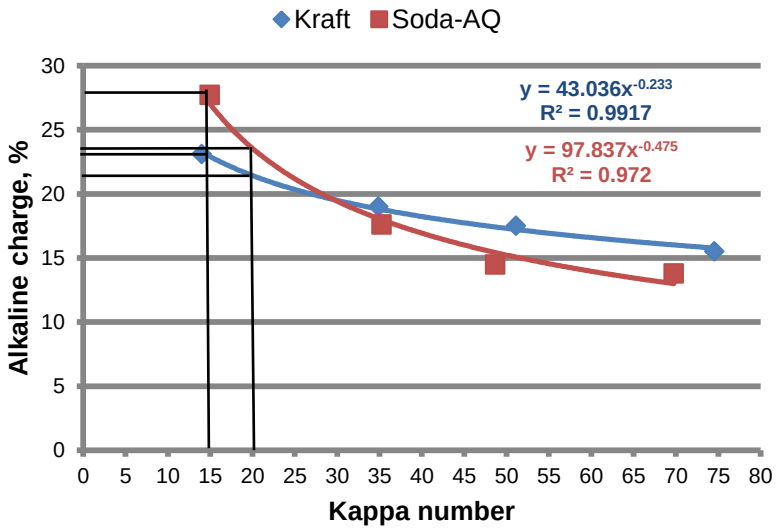

Fig. 2. Delignification curve by kraft and soda-AQ processes for EUCA. Pulping conditions: 26% of sulfidity as NaOH only used to Kraft process; 0.05% of anthraquinone charge only used to Soda-AQ process; 4/1liquor to chips ratio; maximum temperature of 170 °C; time to maximum temperature of 90 min.; time at maximum temperature of 50 min.

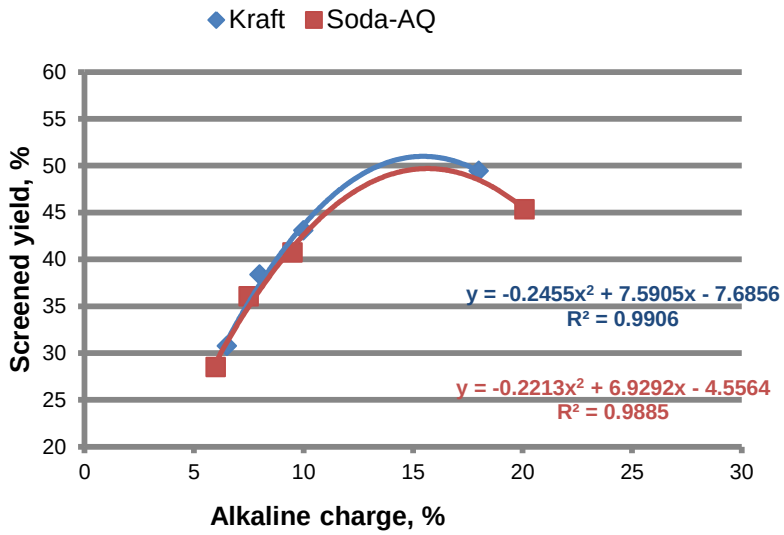

Fig. 3. Alkaline charge *versus* screened yield by kraft and soda-AQ processes for EG. Pulping conditions: 26% of sulfidity as NaOH only used to Kraft process; 0.05% of anthraquinone charge only used to Soda-AQ process; 4/1liquor to chips ratio; maximum temperature of 170 °C; time to maximum temperature of 90 min.; time at maximum temperature of 50 min.

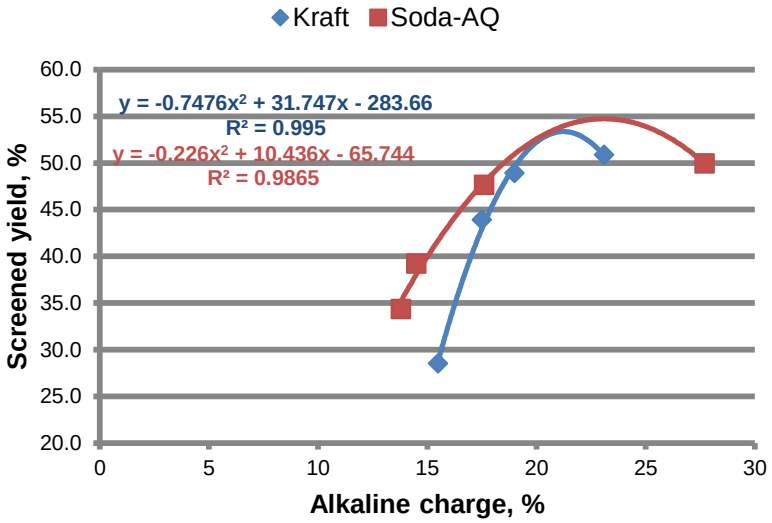

Fig. 4. Alkaline charge *versus* screened yield by kraft and soda-AQ processes for EUCA. Pulping conditions: 26% of sulfidity as NaOH only used to Kraft process; 0.05% of anthraquinone charge only used to Soda-AQ process; 4/1liquor to chips ratio; maximum temperature of 170 °C; time to maximum temperature of 90 min.; time at maximum temperature of 50 min.

Kraft pulping of EG and EUCA

The key to the kraft process is the recovery furnace, which is quite efficient at recovering the pulping chemicals NaOH and Na_2S. Currently it is the most commonly used process for pulp production (Bose *et al.* 2009; Moshkelani *et al.* 2013). Table 7 presents the results for EG and EUCA using the kraft process with the two kappa numbers. For the kraft process, increasing the alkali charge resulted in expected behavior:

lower total yield, higher yield of screened accepts, lower rejects, lower kappa number, lower viscosity, and higher residual alkali. The EG samples showed a lower alkali charge demand than EUCA samples. However, the EG samples presented a higher alkaline consumption during the whole pulping process, especially to the process aiming to achieve a kappa number of 15, probably due to more condensed lignin (low lignin S/G ratio). High consumption of alkali charge is a characteristic that is highly unfavorable, since it is detrimental to the yield, carbohydrate chains, and recovery boiler and increases the production cost (Gomide *et al.* 2004). Overall, for EG, the low screened yield values confirmed the significant presence of extraneous and non-cellulosic materials, as indicated by the analytical data. The screened yield values of EG were in close agreement with those found in the literature for the kraft process (Madakadze *et al.* 2010). For eucalypts, the values for screened yield were also in close agreement with those found in the literature using the kraft process (Gomide *et al.* 2005).

Concerning the brightness values, the results of EG and EUCA were similar between each kappa number evaluated, as expected. The highest values were obtained for kappa number 15. Another parameter evaluated was the hexenuronic acid content, for which a lower content in EG pulps than EUCA was observed. This fact can be explained by the low uronic acid content in EG material. In addition, this pulp characteristic also helps explain the low screened yield during the EG pulping process, since uronic acids work as protection for xylan, preserving it during the pulping process (Magaton *et al.* 2008).

The heating value of black liquor has been associated with lignin content, in that it increases with increasing lignin content (Biermann 1996). For both raw materials at kappa number 20, a high value was observed, which may be explained by a lower carbohydrate loss in pulps with a high kappa number. Another parameter that confirmed this fact is that the black liquor solids and organic solids were always high in the process ending in a kappa number of 15 for both raw materials.

Table 7. Cooking Results of EG and EUCA by the Kraft Process for Kappa Numbers 15 and 20

Parameters	EG kappa 15	EG kappa 20	EUCA kappa 15	EUCA kappa 20
Alkaline charge as NaOH, %	18	15	26	20
Screened yield, %	47.4	47.9	50.4	52.0
Rejects, %	1.1	5.6	0.1	0.4
Viscosity, dm^3/kg	1,100	1,359	939	1,100
Brightness, % ISO	32.9	31.1	33.3	31.1
HexA, mmol/kg	13.2	11.1	39.5	46.4
Black liquor residual AA, g/L	4.1	8.0	16.1	15.8
Black liquor solids, %	11.3	7.4	13.1	10.1
Black liquor organics, %	54.0	52.5	54.2	49.4
Black liquor inorganics, %	46.0	47.5	46.8	51.6
Black liquor heating value, cal/g	3,395.0	3,554.0	3,657.0	3,920.3

Soda-AQ pulping of EG and EUCA

Soda pulping is a chemical process for making wood pulp with sodium hydroxide as the cooking chemical. In the soda-AQ process, anthraquinone (AQ) is used as a pulping additive to decrease the carbohydrate degradation (Gomide *et al.* 1980; Bose *et al.* 2009). Table 8 presents the results of EG and EUCA cooked using the soda-AQ processes resulting in kappa numbers 15 and 20. For the soda-AQ process, increasing the alkali charge resulted in expected behavior: lower total yield, higher yield of screened accepts, lower rejects, lower kappa number, lower viscosity, and higher residual alkali. The lower viscosity of the EG may be explained due to low selectivity of the soda-AQ process, since AQ is efficient only to avoid the peeling reactions (Jeronimo *et al*. 2000), especially for the EG material in which the carbohydrate degradation seems to be larger, probably due to its low carbohydrate content. In addition to the kraft process, the pulp yield in the soda-AQ process was satisfactory for both EG and EUCA among all kappa numbers evaluated (Khristova *et al.* 1998; Jeronimo *et al.* 2000), with a lower value for EG, again reflecting its high extractive and mineral content.

The results of brightness values for EG and EUCA cooked by the soda-AQ process followed the same tendency as the kraft process; the values were similar between each kappa number evaluated, and the highest values were obtained from a kappa number of 15. Similar to the kraft process, the hexenuronic acid content for EG pulps was lower than EUCA pulps. This can be explained by the low uronic acid content in EG material. This pulp characteristic of elephant grass also helps explain the low screened yield during soda-AQ pulping, since the uronic acids help to preserve xylan during pulping (Magaton *et al.* 2008).

Similar to the kraft process, the heating value of black liquor obtained by soda-AQ processes for EG and EUCA were evaluated, and the same tendency was observed; at kappa number 20, the pulps showed a high value. These results indicated less carbohydrate loss in pulps with a high kappa number using the soda-AQ process. This was also confirmed by the contents of black liquor solids and organic solids, which were always high in processes ending in kappa number 15 for both raw materials.

Table 8. Cooking Results of EG and EUCA by Soda-AQ Process for Kappa Numbers 15 and 20

Parameters	EG kappa 15	EG kappa 20	EUCA kappa 15	EUCA kappa 20
Alkaline charge as NaOH, %	19.5	15	29.5	22
Screened yield, %	46.0	47.5	49.6	51.6
Rejects, %	1.4	3.8	0.2	0.6
Viscosity, dm^3/kg	875	883	972	1,028
Brightness, % ISO	31.6	28.5	33.0	29.6
HexA, mmol/kg	15.2	16.3	21.8	45.7
Black liquor residual AA, g/L	10.4	7.7	24.7	19.7
Black liquor solids, %	11	9.3	12.5	11.7
Black liquor organics, %	53.4	56.1	50.2	56.4
Black liquor inorganics, %	46.6	43.9	49.8	43.6
Black liquor heating value, cal/g	3,510.5	3,696.0	3,602.3	4,072.6

Kraft versus soda-AQ pulping of EG and EUCA

By comparing the kraft and soda-AQ processes for EG and EUCA (Table 7 and 8, respectively), it was possible to observe that the screened yield and viscosity were higher in kraft than in soda-AQ, which may be attributed to the high alkaline charge required in soda-AQ process to achieve the desired kappa number. An interesting point about the viscosity is that anthraquinone had a positive effect in preserving the carbohydrates, since viscosity values were close between the kappa numbers 15 and 20 for each raw material evaluated.

Comparing the heating value of black liquor among the four pulps obtained by the two processes, it was also possible to observe a highest heating value of black liquor and lowest brightness for the soda-AQ process. In their work, Bose *et al.* (2009) analyzed pulps by periodate and permanganate oxidations and suggested that the residual lignin from non-sulfur processes contained more condensed structures than kraft residual lignin. The low reactivity of these structures is believed to be responsible for the lower brightness of soda-AQ pulps than kraft pulps. Likewise, the highest result for heating value of black liquor from the soda-AQ process may be explained as stated previously, that is, the heating value has been associated with residual lignin content of the black liquor, being the most condensed lignin which is rich in carbon-carbon bonds; so more energy may be provided from this black liquor.

Between the pulping processes of EG and EUCA pulps, the soda-AQ process has lower yields and worse pulp quality than the kraft process. However, the soda-AQ process would be an excellent replacement for the kraft process if the pulp quality, yield, and delignification rate could be improved, since the black liquor from the soda-AQ process has a high heating value and great potential for applications in biorefineries. The absence of sulfur compounds in the soda-AQ black liquor facilitates its further fractionation into valuable components. Regarding nonwoody raw materials, *e.g.*, corn stalk, sugarcane bagasse, rice straw, many studies have investigated pulp production by an alkaline pulping process. The results have indicated a good potential use of these kinds of raw materials (Won and Ahmed 2004; Aziz and Zhu 2006; Behin and Zeyghami 2009; Zhao *et al.* 2011; Jahan *et al.* 2012), with pulping yield equal to or higher than the obtained yields in this study. For example, a yield of 51.8% has been reported for corn stalk processed by Soda-AQ pulping process ending at kappa number 21 (Jahan and Rahman 2012).

Oxygen Delignification Stage of EG and EUCA Pulps

The pulps produced by two processes and two kappa numbers were submitted to the oxygen delignification process that was described in this paper methodology. Table 9 shows the results of the oxygen delignification stage (O stage). The overall oxygen delignification stage performance was measured by the decrease in kappa number and the increase in brightness. The decrease in kappa number was evaluated using the kappa drop concept, which is the percentage of the subtraction between the kappa number of entrance in O stage and the kappa number after O stage. It was observed that the kappa drop decreased with increasing kappa number, a result likely explained by the decreased content of lignin containing free phenolic hydroxyl groups and increased hexenuronic acid (HexA) content with increasing kappa (Colodette *et al.* 2007).

The EG sample showed the best performance in kappa drop. It was also possible to observe that the kappa drop was higher in the soda-AQ process. This fact may be explained by a lower hexenuronic acid content in the EG pulp (Table 7). It has been

documented in the literature that the oxygen stage is more efficient on pulps with lower hexenuronic acid content since only small amounts of hexenuronic acids are removed during oxygen delignification (Ventorim *et al*. 2006; Eiras *et al.* 2003). Another relevant point that helps to explain the higher efficiency of the pulp obtained by the soda-AQ process is a possible higher content of free phenolic hydroxyl groups (Lai *et al.* 1999), which are the main sites for oxygen reactions (Colodette *et al.* 2007). It is known that in soda-AQ pulping, anthraquinone (AQ) oxidizes the reducing end groups of carbohydrates, thus stabilizing them against terminal depolymerization reactions (peeling reactions) in alkaline media, providing less carbohydrate loss. The reduced form, anthrahydroquinone (AHQ), cleaves part of the β-aryl ether linkages in lignin. Thus, the molecular mass of the residual lignin is reduced and new phenolic hydroxyl groups are formed. Both effects render the lignin more soluble (Kleen *et al.* 2002). Since phenolic hydroxyl groups are essential to lignin dissolution in alkali, a higher content of this functional group in the residual lignin may be partly related to the residual lignin being more condensed. Lai *et al.* (1999) showed that, for woody samples, the tendency of alkaline lignin condensation reactions would increase in this order: high sulfidity < kraft < soda-AQ < soda cooks.

Table 9. Oxygen Delignification Performance of EG and EUCA Pulps

		Kappa 15		Kappa 20	
		*Kappa drop, %	Brightness gain, % ISO	*Kappa drop, %	Brightness gain, % ISO
Kraft	EG	61.9	12.0	50.9	7.5
	EUCA	47.4	20.1	43.8	16.2
Soda-AQ	EG	73.7	14.5	69.4	14.7
	EUCA	54.0	18.3	47.3	16.6

*Kappa drop: Percentage of the subtraction between the kappa number of entrance in O stage and the kappa number after O stage.

Bleachability of EG and EUCA Pulps

The pulps were bleached to a target brightness of 90% ISO as described in the methodology section of this work. In this work, bleachability was defined as the ratio between kappa number entering the bleaching process and total active chlorine (TAC) required for attaining a brightness of 90% ISO. Total active chlorine was defined by the following equation:

$$TAC=[(ClO_2\times 2.63)+(H_2O_2\times 2.09)] \quad (2)$$

where,

ClO_2 = chlorine dioxide dosage in kg/odt
H_2O_2 = hydrogen peroxide dosage in kg/odt

In the equation above, the factors 2.63 and 2.09 are simple conversions of ClO_2 and H_2O_2 into active Cl_2 based on their oxidation equivalents. The EG pulp showed lower bleachability than EUCA, probably due to its high content of metals (Fig. 5). It was observed that the bleachability of EUCA was influenced by the type of process used to

produce the pulp. The best performance was observed for the pulps from the kraft process at kappa number 20. The worst performance was observed for the pulps from the soda-AQ process at kappa number 15. Concerning the raw material type, EUCA showed better performance compared to EG.

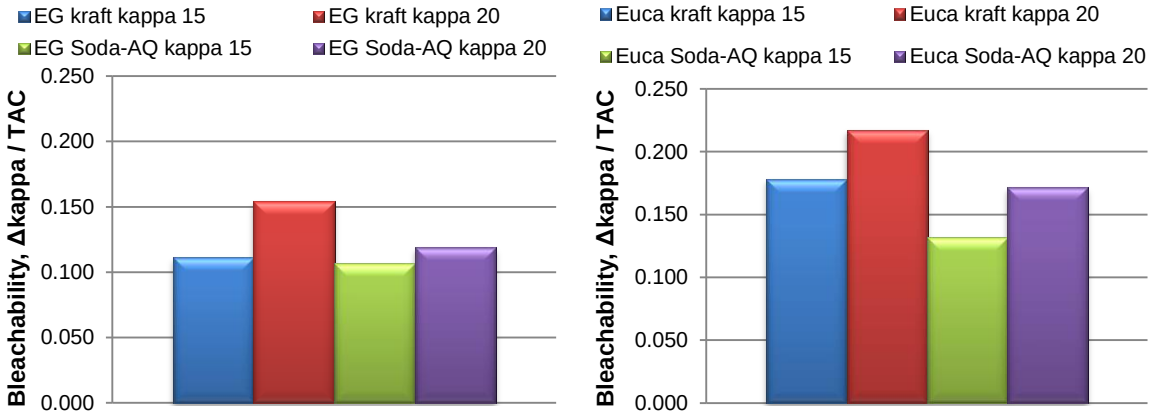

Fig. 5. Bleachability of EG and EUCA pulps in each process studied

Among the EUCA pulps, the best bleachability was found for the kraft process at kappa number 20 (44.1 TAC kg/odt). For EG, the best bleachability was also found for the kraft process at kappa number 20 (50.6 TAC kg/odt). It is important to remember that the EG used in this study did not undergo any improvement processes before pulp production, so the TAC results might have been better if the raw material used had a lower metal content, for example. The total active chlorine (TAC) requirements to bleach the pulps to 90% ISO are shown in Fig. 6. Although the bleachability of the kraft pulp at kappa number 20 for the EG and EUCA pulps was the highest, the TAC was not the lowest due to the effect of the kappa number value. The lowest TAC was achieved from the EUCA kraft pulp at kappa number 15. In general, the highest values of TAC were seen for the soda-AQ pulps of kappa number 20, especially the EUCA sample.

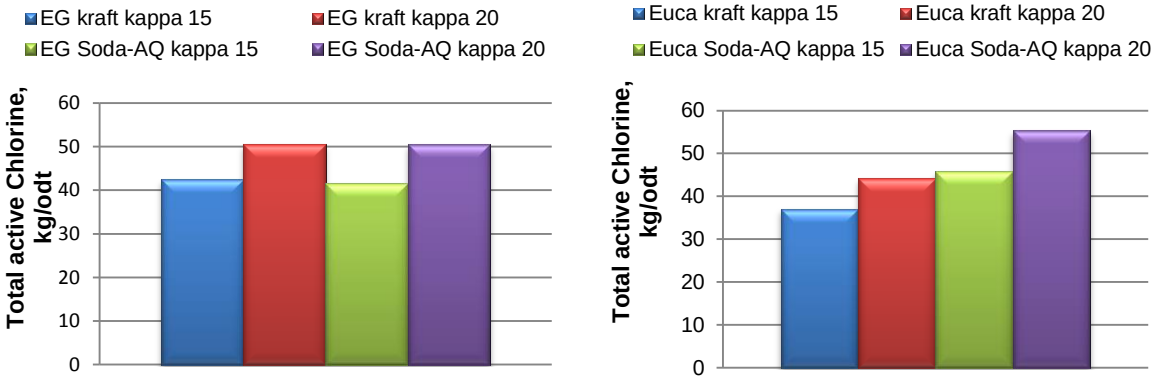

Fig. 6. Total active chlorine of EG and EUCA pulps during the bleaching process

Figure 7 shows the viscosities for bleached EG and EUCA pulps. Among all samples studied, the bleached EG pulp showed the highest viscosity (886 dm^3/kg). For EUCA, only the kraft process at kappa number 20 achieved satisfactory viscosity for

most pulp applications (720 dm^3/kg) (Gomide *et al.* 2005). On the other hand, the bleached EG pulp had viscosity values satisfactory for most pulp applications from the two processes and all kappa numbers evaluated (Gomide *et al.* 2005). Figure 8 shows the results of brightness stability expressed as post color number. The brightness stability increased with increasing kappa number (increasing TAC), as previously reported (Eiras *et al.* 2003). This can be explained by the increased use of oxidizing compounds, thereby removing larger quantities of the compounds that produce the brightness instability. The pulping and the biomass raw material had no observable effects on brightness stability.

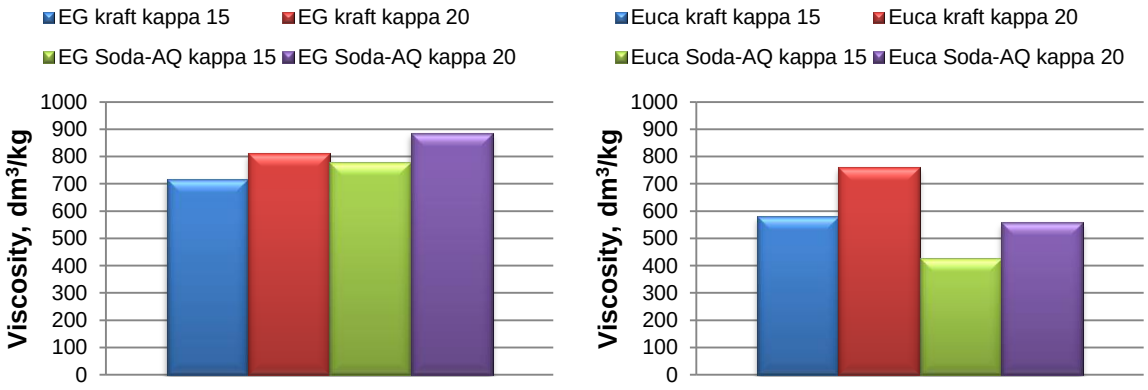

Fig. 7. Viscosity of EG and EUCA pulps during the bleaching process

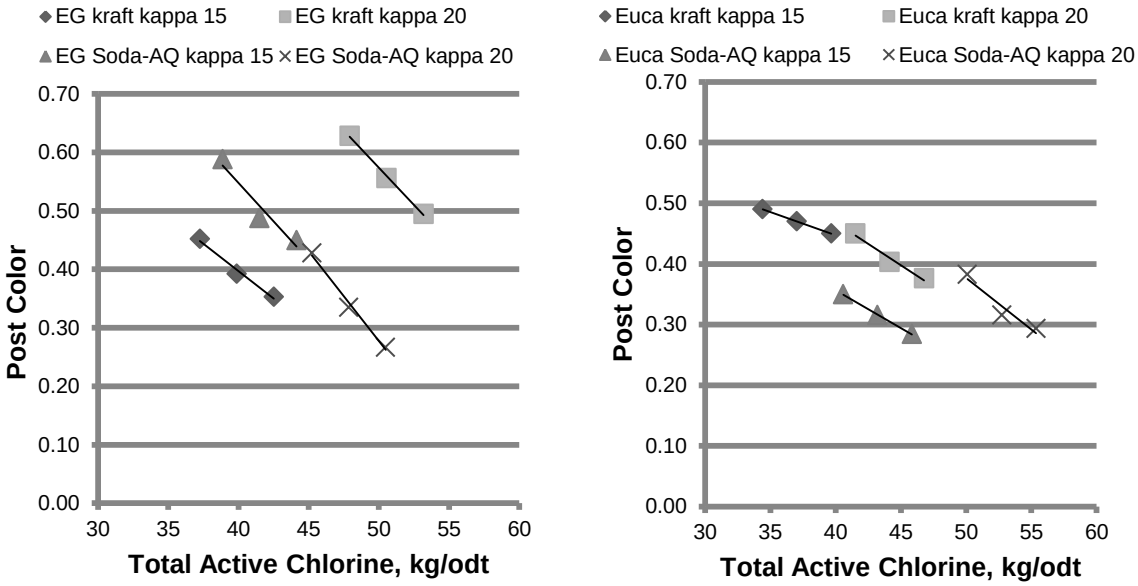

Fig. 8. Post color number of EG and EUCA pulps during the bleaching process

CONCLUSIONS

1. The soda-AQ process presented the lowest screened yield and bleachability among all samples.

2. Elephant grass showed good potential for pulp production. The ideal cooking process was the kraft process at kappa number 20 with a screened yield of 47.9%, bleachability of 0.163 Δkappa/TAC, and viscosity of 812 dm^3/kg.
3. For EUCA, the ideal cooking process was also the kraft process at kappa number 20 with a screened yield of 52%, bleachability of 0.217 Δkappa/TAC, and final viscosity of 886 dm^3/kg.
4. EG showed a high vessel content (11,628 vessels/g of pulp), which is a negative characteristic for some specific paper productions.
5. EG presented a high ash (60,100 mg/kg) and total extractive content (14.8%), which are undesirable and need to be improved in order for this raw material to be used in pulp production.

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