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BIOLOGY

Cellulose pulp produced from bulrush fiber

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ABSTRACT: *Brazil continues to use wood as the principal raw material source for the pulp industry; although, non-wood fibers have been revealed to be a competent substitute to produce paper with different and exceptional properties. Keeping this in focus, this study aimed to assess potential of Schoenoplectus californicus fibers (C. A. Mey.) Soják, commonly identified as bulrush or reed, in cellulosic pulp generation, as an alternative fiber source for the pulp and paper industry. On analyzing the chemical composition of reed fibers, extractives of lignin, carbohydrates, uronic acids and minerals were reported. Physico-chemical characteristics of reed-based cellulosic pulp were estimated including viscosity, hexenuronic acids, etc., as well as anatomical features of length, width, etc. From the chemical analyses of the reed the presence of high concentrations of extractives and silica was clear, making them unfit as raw material for cellulosic pulp production. Pulp kraft pulping process produced brown pulps low in viscosity (34.5m Pa.s) and hexenuronic acid content. Reed is thus classifiable as short-fiber source for pulp and paper industries.*

Key words: *Schoenoplectus californicus, caracterização química, processo kraft.*

Produção de polpa celulósica a partir das fibras de junco

RESUMO: *A madeira é a principal fonte de matéria prima utilizada nas indústrias de celulose no Brasil, porém o emprego de fibras não madeireiras pode ser uma alternativa satisfatória para a fabricação de papéis com propriedades diferenciadas e especiais. Nesse sentido, o objetivo desse trabalho foi avaliar o potencial das fibras de Schoenoplectus californicus (C.A. Mey.) Soják, popularmente conhecido como junco, na produção de polpa celulósica, como fonte alternativa de fibras para as indústrias do setor de celulose e papel. Nas fibras do junco foram realizadas análises de composição química, entre elas: teores de extrativos, lignina, carboidratos, ácidos urônicos e minerais. As polpas celulósicas produzidas a partir do junco foram avaliadas no que diz respeito às características físico-químicas (viscosidade, ácidos hexenurônicos, etc.) e anatômicas (comprimento, largura, etc.). As análises químicas demonstraram que as fibras do junco apresentam altos teores de extrativos e sílica, o que as tornam uma matéria prima desfavorável para a produção de polpa celulósica. O processo de polpação kraft do junco resultou em polpas marrons de baixa viscosidade (34,5m Pa.s) e com baixos teores de ácidos hexenurônicos. O junco pode ser classificado como fonte de fibras curtas para as indústrias de celulose e papel.*

Palavras-chave: *Schoenoplectus californicus, caracterização química, processo kraft.*

INTRODUCTION

In Brazil, the cultivated species of eucalyptus and pine provide the major portion of the cellulosic pulp production. However, other potential sources include the non-wood fibers like bamboo (*Bambusa vulgaris*), sugarcane bagasse (*Saccharum officinarum*), flax (*Linum usitatissimum*), jute (*Corchorus capsularis*), Kenaf (*Hibiscus cannabinus*) and abaca (*Musa textilis*). These fibers are usually used to produce printing and writing papers, packaging and special papers (VAN DEN BERG, 2005).

Investigations on the utilization of grasses like bamboo in the generation of cellulosic pulps, has been done earlier by other authors (BARRICHELO & FOELKEL, 1975; AZZINI et al., 1987; BONFATTI J R., 2010;). However, there is a dearth of published works on the family *Cyperaceae*, of which *Schoenoplectus californicus* (C. A. Mey). Soják, commonly identified as reed, is a member, reported in southern Brazil. Data and information on *Cyperaceae* are available, although usually limited to references in floristic surveys, and confined to citations of the family species in local settings.

As *Schoenoplectus californicus* is a species with rapid growth cycles and less expensive than wood, the utilization of its fibers in industrial processes is interesting. In the state of Rio Grande do Sul, south Brazil, reeds occur in great quantities, showing satisfactory growth in the wetlands and humid

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locations, typical of this region. ROESSING (2007) stated that only very few studies are available on the cellular anatomy of the reed and that information on the development of this species is limited to the few cultivators, who grow it empirically and without any scientific basis.

Reeds commonly utilized as ornamental plants also find wide use in handicraft production due to their natural resistance and malleability. As reeds contain a high pulp percentage they are regarded as a potential raw material for paper production (CORDAZZO & SEELIGER, 1988; SOUZA, 2003; SILVEIRA, 2007). As reeds are highly adaptable and grow fast in the Southern region of the state of Rio Grande do Sul, they are promising as a fiber source and is appealing for paper production as an attractive income source to riparian population who can easily cultivate, harvest, and benefit from the fibers.

Therefore, this study aimed to assess the potential of *Schoenoplectus californicus* fibers as an alternative raw material source for the cellulosic pulp production. Pulps produced were estimated in terms of physico-chemical and morphological characteristics of fibers.

MATERIALS AND METHODS

In July, during the cold and rainy period, the aerial portions of *Schoenoplectus californicus* of 1.54m in length and 2.2cm in diameter on average; respectively, were collected from the margins of a flooded land area on the Island of Marinheiros, in the district of the municipality of Rio Grande, in Rio Grande do Sul State.

Stems collected were cut just above the submerged portion, maintaining the cut limit of the part above the water layer, leaving the root portion for the plant to get reestablished and regrow the aerial parts. As these annual plants are developed in the Areas of Permanent Preservation (APP), samples were collected under prior Forest Authorization No. 01/2013 of the State Secretariat of the Environment - SEMA, through the Department of Forests and Protected Areas - DEFAP, agency Pelotas, taking care of the southern state.

Stems collected were hand-chopped and processed into chips ranging between 5 and 7cm in length. A portion of the chips were milled to sawdust using a Willey mill, and stored after sorting them via sifting through 40/60 mesh sieves, in hermetically sealed glass jars for chemical analysis in future.

Due to the significance of chemical analysis and to collect more data on the fibrous raw material in question, chemical characterization of the reed fibers was done by employing the following analyses: the extractive content in acetone (TAPPI T 280 pm-99), total extractive content (TAPPI T 264cm-97-adapted), insoluble lignin (TAPPI T 222 om-98-adapted) and soluble TAPPI A 250) in acid, sugar composition (HPLC-PAD according to WALLIS et al., 1996), inorganic compounds (TAPPI T 211 and TAPPI T 244) and uronic acids (SUNDBERG et al., 1996).

The *Schoenoplectus californicus* fibers produced 45.7 Kappa kraft pulp. Using the electrically heated Regmed® rotary digester provided with a thermometer and manometer, having 4 individual reactors of 2 liters capacity each, baking was done. Cooking time and temperature were monitored using a computational electronic controller at the control center of the other cooking settings. Parameters employed for kraft pulping included the following: Absolutely dry chips = 150g (a.s.); Active alkali load (AA) = 7% (expressed as Na_2O); Sulfidity = 25%; Liquor / reed ratio = 5/1; Maximum temperature = 170°C; Time to achieve maximum temperature = 90 minutes and time to achieve maximum temperature = 30 minutes.

After being baked, fibers were expelled from the reactors and water-washed at room temperature, through a 150 mesh stainless steel screen. Fiber individualization was done in a 25 liter laboratory hydroculator, at an approximately 0.6% consistency. Cellulose pulp debugging was done in a Voith® laboratory scrubber provided with a perforated plate having 0.2mm slits. After washing, defibration and purification, the cellulosic pulp was centrifugally dewatered at about 30% consistency, after which it was stored in a polyethylene bag for further analysis (TAPPI T 236cm-85), including viscosity (TAPPI T230 om-94) and hexenuronic acids (TENKANEN et al., 1999). In terms of the fiber dimensions of the pulps obtained, the length and width, thickness and width of the fiber lumen were assessed based on the Technical Standard of LCP 02 pp-97 and with the help of the Image-Pro Plus® Program.

RESULTS AND DISCUSSION

The results obtained for the reed fibers were compared mainly with work done with eucalyptus wood fibers and bamboo fibers. These comparisons are important because eucalyptus is the main short fiber used in the pulp and paper industries and bamboo, because it is like a reed, monocot (an alternative fiber) already used industrially. It is imperative to identify the chemical properties of the study material for technological research purposes (FONSECA, 2012). ANDRADE (2006) proposed that it was fundamental to evaluate the principal chemical components of wood, due to their great influence on the chemical pulping processes, thus altering the final product quality. For instance, ALMEIDA (2003) stated, that the extractive and lignin content of the wood had a direct influence on the alkali consumption, yield and delignification rate, enabling the determination of the potential of a raw material for cellulose pulp production.

In this study, reed fibers were characterized chemically, to quantify the extractive content in the acetone (3.60%), total extractive content (11.94%), insoluble lignin content (22, 19%) and solubility in acid (2.70%), besides the total carbohydrate (55.89%) and uronic acid (1.85%) concentrations.

The total carbohydrate fraction termed holocellulose (cellulose plus hemicellulose), involves the cellulose fraction; whereas, other sugars (xylans, galactans, mannans and arabinans) represent the hemicellulose fraction (KLOCK et al., 2005). Other constituents of the reed samples included 34.30% glucans, 17.90% xylans, 0.90% galactans, 0.27% mannans and 2.58% arabinans. In *Eucalyptus grandis* and *Eucalyptus urograndis* wood PEDRAZZI (2009) identified the holocellulose levels of 62.10% and 61%, respectively. GONCALEZ et al. (2002) identified holocellulose, lignin and the total extractives of 64.50, 27.30 and 8.20%, respectively, in the *Pinus taeda* wood. PEREIRA et al. (2000); however, in their research on *Eucalyptus benthamii* wood identified the holocellulose, lignin and total extractive contents of 69.70, 24.30 and 6.00%, respectively.

GUIMARÃES JR. et al. (2013); however, in their chemical characterization of the *Bambusa vulgaris* samples concluded that bamboo shows high holocellulose (67.58%) and low lignin (17.31%) contents when compared with the pine and eucalyptus woods.

On comparison of total carbohydrate content of the reed with the content assessed for wood and bamboo, in the researches mentioned above, it is clear that, although the reed fibers have a lower content, it can still find use as a raw material source in the cellulose industries. ALMEIDA (2003), proposed that the holocellulose content has a bearing on the yield of the pulping process, playing a crucial part in the economic aspect of the industry.

However, when the total extractive value in the reed (11.94%) was compared with those of *Pinus taeda* (8.20%) and *Eucalyptus benthamii* (6.00%), high extractive content of the reed exhibited some disadvantages in utilizing it as a raw material source for chemical pulping. BONFATTI JR., (2010) indicated that the undesirable chemical constituents of extractives and lignin affected the cellulose production process; by determining them, the estimates of the yield process and reagent consumption during pulping can be indirectly predicted.

With reference to the lignin concentration, GONCALEZ et al. (2002) and PEREIRA et al. (2000) reported that for reed the value assessed was 24.89%, similar to that of the other raw materials utilized in the pulp and paper industries. Lignin cannot; therefore, be considered as a limiting chemical component in the use of reed fibers during the kraft process of cellulose production.

Reed also revealed a uronic acid value at a concentration of 1.85% on average, while eucalyptus wood registered between 4.70% and 5.00% (PEDRAZZI, 2009). According to ZANUNCIO & COLODETTE (2011), the eucalyptus wood samples contained 4.05% to 5.29% uronic acid in the samples. Therefore, the value of uronic acid occurring in the reed samples was lower than that in eucalyptus wood. The authors emphasized the presence of uronic acids in some hemicelluloses, particularly the xylans. During the alkaline chemical pulping process, uronic acid structure was altered, and changed to hexenuronic acids, which reversed the pulp whiteness and raised the use of chemical reagents in the bleaching stage of the pulp industry.

Thus; although, the reed contains a higher xylan concentration than eucalyptus, the uronic acids occur in lower quantities. COLODETTE (2001) claimed that the chemical structure of the xylan monocotyledons is less acidic than the xylans in eucalyptus wood, causing this hemicellulose to be more resistant to chemical degradation during pulping.

Ash values of 76780mg kg^{-1} were reported in the reed samples, showing on average a high silica content of 37,220mg kg^{-1}. As the total ash value for reed was reported to be 76,780mg kg^{-1}, it could be deduced that among the total inorganic compounds in the samples 48.48% was silica. Other inorganic compounds included magnesium (281mg kg^{-1}), calcium (204.90mg kg^{-1}), manganese (74.40mg kg^{-1}), iron (14.60mg kg^{-1}) and copper kg^{-1}.

Due to the high silica content in reed it is considered unfit as a raw material for cellulose production, because silica is an inorganic component that induces the wearing out of industrial equipment (FOELKEL, 2009). BOECHAT (2010), identified the occurrence of silica in non-wood materials acting as one of the principal reasons for problems in the cellulosic pulp industry.

Pulping process associated with cooking the reed fibers, produced pulp having 45.74 kappa number, 45.85% purified yield and 7.83% reject content. AZZINI et al. (1988) reported yields from the cellulose fibers between 45.67 and 56.78% for *Bambusa tuldoides*. Prior, GUIMARÃES JR. et al. (2013), in their study on *Bambusa vulgaris* pulp recorded nearly 46% purified yield with kappa number between 45 and 55. On comparison bamboo pulps with those reed fibers identified in the current study, it is easily confirmed that yield values in cellulose for a kappa number of around 45 are similar between these two monocotyledons; thus the economics of pulp manufacturing processes to produce paper with the reed as raw material will be unaffected. BONFATTI Jr. (2010) stated that the purified yield shows the quantity of cellulose pulp post the purification process, that is, without the tailings, which is the basic parameter to qualify for pulping and the economics of the process.

With respect to the viscosity and concentration of the hexenuronic acids in the cellulosic reed pulps, the values of 34.50mPa.s and 7.41mmol kg^{-1}, were reported, respectively. In their research, PEDRAZZI (2009) and QUEIROZ et al. (2004) used eucalyptus wood for kraft cooking and the resultant pulps showed viscosities between 63.20 and 69.50mPa.s and a hexenuronic acid content of 60.70mmol/kg on average. Reed pulp had a lower hexenuronic acid content compared with the cellulosic pulps of eucalyptus wood, an expected result as the reed fibers showed a low uronic acid content.

As the hexenuronic acids are not favorable to the cellulosic pulp bleaching process because they utilize reagents like chlorine, chlorine dioxide, ozone or peracids used in this process (ZANUNCIO & COLODETTE, 2011), their presence makes the reed pulp unsuitable for bleaching.

The low viscosity (34.50mPa.s) reported for reed pulp is due to the higher rate of carbohydrate degradation during the pulping process, in which the alkali effectively degraded the holocellulose content of fibers, confirmed by the poorer yield observed in cooking. MOKFIENSKI (2004), showed that the cellulosic pulp viscosity was related to the integrity or intensity of the carbohydrate polymerization (cellulose and hemicellulose) resulting from the cooking.

LAMMI & SVEDMAN (1999) reported that even at optimal levels of the kappa number (16-18), the hardwood kraft pulps may exhibit certain variations in purified yield and quality, several of which could play a role in the variability of the quality of the fibrous raw material, although the others are definitely connected with the cooking process, which influence both chemical characteristics of lignin and carbohydrate fraction and concentration in the pulp, as confirmed in this study.

The morphology of the fibers and their chemical components facilitate predicting the behavior of pulp during the conditions under which the paper production process operates (CARPIM et al., 1987). In table 1 the results of the mean values for the wall thickness length, width, lumen diameter for reed pulps are listed.

From the values evident in table 1, the reed pulp fibers showed an average length of the 0.93mm with an average width of 10.55µm. The mean diameter (3.95µm) and the wall thickness (3.30µm) played no role in characterizing the fiber, but rather revealed the quality and resistance tests of the pulp products, not within the purview of this study.

According to GUO et al. (2010) the lengths ranged from 1.50 to 2mm, whereas the widths were between 17.90 and 19.10µm for the bamboo cellulose pulp fibers. TRUGILHO et al. (2005) in their evaluations of the unbleached cellulose pulp fibers of 15 *Eucalyptus* spp. clones reported mean fiber diameter of 0.92mm and mean width of 15.76µm. In his study, MOKFIENENSKI (2004) morphologically analyzed the bleached pulp fibers of some *Eucalyptus* spp., and reported that in *Eucalyptus globulus* the mean length was 0.77mm and width was 21.00µm, whereas for *Eucalyptus grandis,* the length hovered from 0.89 to 0.98mm and the width from 20.80 to 22.10µm, but for *Eucalyptus urophylla*, the length remained at 0.97mm with the width at 20.81µm.

When the results for reed showing the fiber dimensions of bamboo and eucalyptus pulps were compared, the reed pulp was seen to have fibers with similar lengths to those of eucalyptus pulp fibers but less for the bamboo pulps. As pulps are categorized based on fiber lengths, and eucalyptus is obviously a short fiber species, reed is classified as a fibrous source for short fiber cellulose production. BRACELPA (2010) stated that these fibers are suitable for the production of paper for printing and writing and sanitary uses, and despite their lower resistance, they enable the production of papers with a high degree of softness and absorption.

Conversely, when fiber width is considered, reed revealed poor results when compared with the fibers from the bamboo and eucalyptus pulps, it

Table 1 - Fiber dimensions in chemical pulps of reed.

Average number of fibers measured	Length (mm)	Width (μm)	Lumen diameter (μm)	Cell wall thickness (μm)
1 a 25	0.91	10.34	3.71	3.31
1 a 50	0.90	10.76	3.85	3.45
1 a 75	0.90	10.57	3.94	3.32
1 a 100	0.93	10.55	3.95	3.30

was this characteristic that made the reed fibers unfavorable. CASTANHO & OLIVEIRA (2000) identified that fibers having greater widths have higher chances of collapsing, which facilitates refining, offers a greater contact area between the fibers during paper sheet production and, hence, revealed greater resistance in the paper sheet produced.

CONCLUSION

Results gave sufficient evidence of the high levels of extractives and silica present in the reed fibers. Mat brown pulps could be produced having kappa number of 45.74 and purified yield of 45.85% containing 7.41mmol/kg of hexenuronic acid and low viscosity of 34.50mPa.s. Reed was used as source of raw material for the production of pulp cellulosic of short fiber.

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