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Production of Cellulosic Pulp from Reed (*Phragmites australis)* to Produce Paper and Paperboard

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Abstract: Due to the global increase in the demand for paper and cardboard, and depletion in some areas of wood resources (main raw material in the paper industry), it has been necessary to investigate new raw materials to substitute the conventional ones. In this work, *Phragmites australis*, commonly known as reed, has been studied. It has been characterized the chemical composition of the raw material by determining the ash content, α-cellulose, holocellulose, lignin and their extractables in ethanol, 1% soda and hot water. Subsequently three pulping processes have been conducted, using the “soda method" under three different operating conditions. The cellulosic pulps and the papersheets made from the pulps were analysed in order to determine the chemical and physical properties following the relevant TAPPI, UNE and ISO standards. Comparing the results obtained, it can be possible to conclude that the pulp obtained under the following operating conditions: 175°, 90 minutes and 14% of NaOH, presents the best results.

Keywords: *Phragmites australis*, Cellulosic Pulp, Soda, Paper, Paperboard

1. Introduction

There are many resources that nature offers us, some of them are not renewable like fossil fuels. However, many others are, such as plant biomass, a type of resource that using and managing it properly can offer great benefits both from an environmental and economic point of view. In this sense, it is convenient to recall within the 12 Principles of Green Chemistry proposed by [1], the seventh, called "Use of Renewable Feedstocks" that revolves around that the raw material must be preferably renewable, technically and economically feasible.

The largest source of renewable raw material in the world is the lignocellulose biomass. This work is focused on the use of *Phragmites australis*, commonly known as reed, as raw material to produce cellulosic pulps for the manufacture of paper and / or paperboard, because it has high content of cellulose (33-36%) and hemicellulose (20-22%) [2].

Phragmites australis is the most widely distributed angiosperm, it is a wild plant that reproduces easily naturally due to its system of rhizomes or stolons. It is found mainly in freshwater environments (marshes) and marine wetlands. Featuring a wide geographic distribution around the world, from warm to tropical zones. It is a perennial plant that grows from 2 to 4 meters in height, preferably in the warm season, being the optimum temperature of growth of 30-35°C. It grows more favourably in argillaceous soils, tolerates very well the moderate salinity being able to grow in brackish water [3, 4].

The reed has become a problem in wetlands because its occurrence has increased considerably, as it causes a decrease in plant species richness [5] reductions and microphotography [6] and reduction of biodiversity [7]. It is a promising raw material because of its high potential for production and composition, providing a large amount of biomass with annual yields of 40-63 tonnes per hectare, and also has become a very important source of biomass due to its huge reserves and increased cultivation [3]. The reed (*Phragmites australis*) has a dry yield of 4-8 t / ha / year [8].

The *Phragmites australis* has been success-fully employed to purify waste water, using reed ponds [9] and natural marsh [10]. It can also be used for papermaking [11], construction material, electricity [12] and bioethanol [13].

2. Materials and Methods

2.1. Raw Materials

The common reed, *Phragmites australis,* used in this work were provided by Center for New Water Technologies, located in Carrión de los Céspedes (Seville, Spain).

Once harvested, were dried and kept at constant temperature. Dried reed was prepared according to the TAPPI standard T-257 and was characterized according to TAPPI standards to determine α-cellulose (T-9m54), lignin (T-203os61), holocellulose (T-222), ash (T-211), ethanol extractable (T-204), solubility in 1% soda (T-212) and solubility in hot water (T-257). The analyses for the chemical characterization were done in triplicate and the mean standard deviation were calculated [14].

Figure 1. *The common reed (Phragmites australis).*

2.2. Pulping Process and Pulp Characterization

The reed was pulped in a 15L reacted heated by an outer jacket heater and under stirring by rotating the reaction vessel via a motor, following three different process, these conditions of pulping have been chosen because of the similarity of the reed with other raw materials that have been previously studied. Table 1 shows the temperature, time, and concentrations of reagent (soda) used in the pulping experiments. The liquid/solid ratio was always 10:1.

Table 1. *Operating conditions of the different process.*

	Temperature (°C)	Soda concentration (%)	Time (minute)
Pulping 1	155	8	30
Pulping 2	165	11	60
Pulping 3	175	14	90

After each process was completed, cooked material was unloaded from the reactor, washed with water at room temperature to remove residual cooking liquor and fiberized in a disintegrator at 1200 rpm for 30 min, which was followed by beating in a Sprout-Bauer refiner. Finally, the fiberized material was passed through a filter of 0.16 mm pore size to remove uncooked particles.

The pulp obtained were characterized according to the TAPPI methods: beating degree (T-227), yield (gravimetric method), α-cellulose (T-9m54), lignin (T-203os61), holocellulose (T-222), ash (T-211), ethanol extractable (T-204), solubility in soda (T-212) and solubility in hot water (T-257), Kappa number (T-236cm-85) and viscosity (T-230om-94) [14].

Figure 2. *Cellulose pulp of reed (Phragmites australis).*

2.3. Formation Paper Sheets and Characterization

Ten paper sheets were prepared from each cellulose pulp on an ENJO-F-39.71 sheet machine according to TAPPI standard T205ps-95. The paper sheets were conditioned in a weather chamber at 25°C and 50% humidity for 48h before performing the mechanical test.

Once conditioned, this paper sheets were characterized in terms of tensile index, burst index and tear index following the TAPPI standard T494om-96, T403om-97 and T414om-98, respectively [14].

3. Results and Discussion

3.1. Characterization of Raw Material

The results obtained for the characterization of *Phragmites australis* are shown in table 2, together with the values found in literature for other raw materials [15].

Table 2. *Chemical characterization of reed (Phragmites australis) and different alternatives raw materials.*

	Ethanol extraction (%)	**Soda extraction (%)**	**Solubility in hot water (%)**	**Ash (%)**	**Holocellulose (%)**	**α-cellulose (%)**	**Lignin (%)**
Phragmites australis (Reed)	7.82	21.84	18.05	7.51	62.56	30.86	18.25
Rice straw	0.56	57.70	7.30	9.20	60.70	41.20	21.90
Leucaena leucocephala	4.64	18.44	3.98	-	75.92	44.43	22.43
Arundo donax	7.30	26.80	4.73	-	70.20	40.46	22.34
Paulownia fortunei	5.50	31.50	9.60	-	70.70	37.40	22.40
Olive pruning	10.36	30.04	8.16	1.36	61.47	35.67	19.71
Cotton stalks	1.42	20.34	3.33	2.17	72.86	58.48	21.45
Pine	2.57	8.00	1.99	0.54	69.60	55.90	26.20
Eucalyptus	1.15	12.4	2.84	0.57	85.50	52.80	20.00

It is observed that the ash content is high (7.51%), similar to that of rice straw, which can cause corrosion problems in the work equipment. The content of α-cellulose of the reed is lower (30.86%) than the rest of the raw materials. However, the content of hemicellulose is similar to that of other materials studied, except for eucalyptus, which has higher hemicellulose values. The lignin content is the lowest (18.25%) of those found in the studied materials, which makes the reed less hard material and therefore the cooking treatment does not have to be so aggressive to be able to isolate the cellulose.

3.2. Chemical Characterization of Cellulose Pulp

Table 3 shows the results obtained from the characterization of the three pulps obtained and compared with other alternative raw materials.

Table 3. *Chemical characterization of cellulose pulp.*

	Ethanol extraction (%)	**Ash (%)**	**Hemicellulose (%)**	**α-cellulose (%)**	**Lignin (%)**
Pulp 1 *Phragmites australis* (Reed)	1.58	3.83	20.45	60.57	11.37
Pulp 2 *Phragmites australis* (Reed)	1.44	3.25	18.81	67.54	5.18
Pulp 3 *Phragmites australis* (Reed)	1.53	3.78	17.36	69.25	3.94
Oats	0.89	2.50	16.40	69.20	13.10
Corn	0.86	1.36	20.00	71.00	8.90
Barley	1.31	2.64	18.30	69.90	10.90
Wheat	3.37	2.47	25.71	59.20	7.72

It is observed that the results are similar in the three pulps, seeing that the highest content of α-cellulose (69.25%) and the lowest of lignin (3.94%) was obtained for pulping 3, which presents the most drastic operating conditions

The amount of hemicellulose is similar to all raw materials. This content is important because the presence of the hemicellulose favours the swelling of the fibres, which increases the plasticity, flexibility, and ease of bonding between them, resulting in an improvement of the density and the mechanical and physical properties of the sheets.

The α-cellulose content is similar to the other raw materials studied. The lignin content is appreciably lower than the other materials, except for pulp 1 which is slightly higher than the others pulps of reed.

3.3. Characterization of the Pulps and Paper Sheets

Table 4 shows the values of yield, beating degree, Kappa number and viscosity for the different pulps.

Table 4. *Characteristics of the pulps.*

	Yield (%)	**Viscosity (mL/g)**	**Kappa number**	**Beating Degree (ºSR)**
Pulp 1	33.9	700.9	101.5	14.0
Pulp 2	30.5	1014.8	74.6	20.0
Pulp 3	30.2	1101.5	57.7	25.5

The pulp 1 presents a higher yield (33.9%) than the other two, although the differences are not significant.

The viscosity of the pulp 3 is the highest (1101.51 mL/g). This is important, because in a bleaching process there will be further degradation of the cellulose in the fibres, with a consequent further decrease in the viscosity value.

The Kappa number is a measure of delignification process. Hight values of Kappa number imply that the pulp may not be well suited for carrying out bleaching of the pulps because of high cost, because the Kappa numbers is related to the amount of lignin present in the pulp. The pulp 3 presents the lowest value (57.7) for the Kappa number, because it is the pulp that has a lower content of the lignin and will be easier to bleach.

The pulp 3 has the highest beating degree, which makes it present a greater cross-linking between the fibres.

The values of the properties of the paper sheets (tensile index, stretch index, burst index, tear index and brightness) are shown in the table 5.

Table 5. *Characteristics of the paper sheets.*

	Rupture length (m)	Tensile Index (Nm/g)	Tear Index (mNm^2/g)	Burst Index (kPam^2/g)	Brightness (%ISO)
Pulp 1	1891	18.54	2.66	1.18	18.7
Pulp 2	2895	28.37	2.98	1.93	20.0
Pulp 3	2945	28.86	2.91	1.85	21.2

The paper sheets produced with the pulp 3 present the best result for the rupture length (2945 m), tensile index (28.86 Nm/g) and brightness (21.2% ISO). The results of the burst and tear indexes show some similar results between the three pulps, with slightly higher values of pulp 2, although the differences are not significant.

4. Conclusions

From the results obtained in the characterization of *Phragmites australis* (reed), it is concluded that it is an alternative raw material to those already used in the production of paper, presenting properties similar to those found in literature for other alternative raw materials.

The cellulosic pulp obtained under the following conditions: 175°, 90 minutes and 14% NaOH (on dry matter) and with a liquid / solid ratio is the one with the best properties of both the pulp and paper sheets obtained.

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