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Chemical composition and pulping of banana pseudo-stems

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Abstract

This paper deals with the determination of chemical composition and the study of the pulping potentialities of banana pseudo-stems growing in Madeira Island (Portugal). First, the raw material was both studied as a whole (type I) and as the outer bark part (type II), which is richer in cellulose fibres. Before starting the cooking of banana wastes, the main components of the two types of material were quantified, and showed that the polysaccharide content was high enough (about 60–70%) to justify the pulping investigations. Moreover, the lignin content was very low (approximately 12%). The only discouraging finding was the relative high amounts of ashes and extractives. The pulping of these residues was carried out using soda, kraft and soda-anthraquinone (AQ) cooking processes and the optimal pulping conditions were established. Thus, pulps with a yield of about 37–38% with a Kappa number (Kappa no.) about 30–32 were obtained when cooking in the presence of 0.25–0.35% of anthraquinone at 120 °C for a short cooking time, i.e. 30 min. The longer times and higher temperatures of cooking as well as the use of kraft pulping conditions did not give rise to better performances, comparing to those mentioned above. These severe conditions were detrimental to the hemicellulose preservation.

Keywords: *Musa acuminata* Colla; Agricultural residues; Non-wood pulp; Annual plant; Soda-anthraquinone pulps; Fibres properties

1. Introduction

Over the years, an increasing preoccupation regarding forest preservation and rational use of forest and agricultural residues has occurred. This fact was mainly motivated by the increasing consumption of wood fibre-based products, such as panel, paper and boards. This demand is currently solved by using increasing amounts of recycled fibres. Thus, in some paper grades, more than 50% of raw materials are secondary fibres. Annual plants could also be a new source of lignocellulosic fibres for papermaking and/or composite materials.

In Madeira Island (Portugal) the production of banana, *Musa acuminata* Colla, has a capital economical importance. This agricultural activity generates a large amount of residues, because each plant produces only one bunch of bananas, after its harvesting the bare pseudo-stems are cut and usually left in the

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soil plantation to be used as organic material. Thus, it could be estimated that few tons per hectare are produced annually. As we believe, these crops could and should find a more rational way of utilisation, namely as a source of cellulosic fibres. In this context, we have recently started a research program aiming to deep the knowledge on chemical and structural constitution of banana plants (Oliveira et al., 2002) and the use of its cellulosic fibres in papermaking composition and as reinforcing fibres in composite materials (Faria et al., 2002).

These approaches offer several advantages, since these raw materials can be produced annually and have generally lower lignin contents. They are more easily delignified and require milder and faster cooking conditions comparing with wood fibre sources. The resulting pulp can be used mainly in paper and board, fibre-board and composite materials.

This paper deals with the results of chemical composition of banana crops as well as their soda, soda-anthraquinone (AQ) and kraft pulping optimisation. To the best of our knowledge, the few works dealing with the use of banana crops as a source of fibres concerns varieties which are different from our raw material, namely: *Musa paradisiaca* L., *Musa sapientum* and *Musa cavendishii* (Darkwa, 1998; Soffner et al., 2000; Soffner and Silva, 2000). These studies showed that 9% of active alkali cooking of *M. paradisiaca* L. crops produced pulps of 59% yield and Kappa number (Kappa no.) of 30. The fibres obtained by kraft cooking of the first two species have an average length of about 3.9 mm which explain their high strength properties (Darkwa, 1998). Cooking of *M. cavendishii*, at 120 °C for 2 h, and using CaO (12% (w/w)) as delignified agent gave 50% pulps yield (Soffner et al., 2000; Soffner and Silva, 2000).

2. Experimental

2.1. Material and chemical analysis

The pseudo-stems of *M. acuminata* Colla were harvested from a banana plantation in Funchal (Madeira Island, Portugal). The pseudo-stem of mature plants (after cutting the bananas bunch), randomly selected, were handily separated from foliage and several sheets of the trunk were disconnected. Afterwards they were air dried for 2 weeks. To carry out our experiments the initial raw material was divided in two types: (i) the whole material (type I) and (ii) the outer bark material (type II) which seemed to be richer in fibres. Both types of material were ground and the 60–80 mesh fraction was selected in order to determine its chemical composition, which is in agreement with standard Tappi T264 om-88.

The samples were first submitted to soxhlet extraction with ethanol/toluene (1:2 (v/v)) and water, for 8 h. The determination of the ashes content, extractives, Klason lignin, holocellulose using peracetic method, cellulose following Kurschner–Hoffner approach and hemicelluloses I and II fractions, were performed following standards T211 cm-86, T204, T13 wd 674, Tappi useful method 249, for ashes, extractives and Klason lignin, respectively (Oliveira et al., 2002). The cellulose content was determined following Kurschner–Hoffner approach which consists of treating 5 g of extractives-free samples with 125 ml of alcoholic nitric acid solutions under reflux during four cycles of 1 h. After each cycle, the alcoholic nitric acid solution is removed and a fresh volume is added. The alcoholic nitric acid solution consisted in mixing one volume of 65% (w/w) solution of nitric acid with four volumes of 96% purity ethanol (Browning, 1967). At the end of the four cycles, the cellulose was washed, dried and weighed. Holocelluloses were determined according to peracetic method which consisted of treating 10 g of free extractive banana crops with 500 ml of peracetic acid solution and 25 g of sodium acetate during 70 min at 70–74 °C. The peracetic acid solution was prepared by mixing one volume of 30% hydrogen peroxide solution to an equivalent volume of pure acetic anhydride at 0–4 °C. The solution was then kept at 6 °C for 48 h before titration with $KMnO_4$ in the presence of H_2SO_4. This solution was then diluted to 10% (w/w) and used to isolate holocellulose. At the end of the reaction, the isolated polysaccharides were filtered, washed, dried and weighed.

The holocelluloses were fractionised by sequential extraction of with 5% KOH aqueous solution (hemicellulose I) and 24% KOH aqueous solution (hemicellulose II). The extracted fractions were then dried and weighed.

The polysaccharides of the extractive-free banana pseudo-stem materials were also analysed. In fact,

the sugar composition of holocellulose, cellulose and hemicelluloses were determined by acid hydrolysis according to the TFA method (Fengel and Wegner, 1980) optimised by Gaiolas et al. (2003) followed by high performance liquid chromatography (HPLC) determination. The HPLC measurements were carried out using a Perkin-Elmer 250 Chromatograph equipped with a refractive index detector (HP 1074 A, Hewlett-Packard). The column used was a commercial polysphere CH-PB purchased from Merck. A flow rate of 0.4 ml/min bi-distilled water and a temperature of 80 °C were used. Calibration curves of D-glucose, L-arabinose, D-xylose and D-mannose were obtained using high purity commercial sugars from Merck.

2.2. Pulping

In order to optimise the cooking conditions, chips of banana crops were prepared (approximately 10 cm × 10 cm and 5–7 mm of thickness), and put on rotational thermostated mini-digesters having a volume of 100 ml (20 g oven dried (o.d.) material) controlled by an ES100P apparatus. The effects of (i) raw material type; (ii) temperature: 90, 120 and 160 °C; (iii) sodium hydroxide concentration (w/w with respect to o.d. material): 0, 5, 10, 18, 25 and 50%; (iv) liquor/crops ratio (l/kg): 3/1, 5/1 and 7/1; (v) time at constant temperature: 15, 30, 45, 60, 120, 180 and 240 min; (vi) anthraquinone concentrations (w/w with respect to o.d. material): 0, 0.15, 0.25 and 0.35%; and (vii) sodium sulphite concentrations (w/w with respect to o.d. material): 5 and 18%, were studied. In all experiments, the heating time in order to reach the constant temperature was 1 h.

After cooking, the cooked material was separated from black liquor, disintegrated, screened and washed abundantly with fresh water, using L&W laboratory strainer. The disintegrator used, enabled the pulp screening which allowed, after drying, the determination of both total pulp yields and screening rejects by weighing each fraction. The residual lignin in the pulps was assessed by determining the Kappa no. The degree of polymerisation was calculated from viscosity data (TAPPI 206 m-55). The sugar complex in different pulps was also determined by the method described below (Fengel and Wegner, 1980; Gaiolas et al., 2003).

3. Results and discussion

3.1. Chemical composition

The chemical composition of the two types of raw material is shown in Table 1. The first remark concerns the high amount of ashes (approximately 14%) which is not common for annual plants (Atchison, 1993). The ashes were mainly constituted by K: 33.4%; Ca: 7.5%; Mg: 4.3%; Si: 2.7% and P: 2.2%. The high quantity of ashes is not dramatic in terms of pulping process, since the silicone-based salts are negligible. This ash content is still high for industry processing, in spite of the possibility that 50% of them can be extracted by washing before pulping, as determined in our laboratory.

The second remark is the considerably low amount of lignin, i.e. approximately 12%, to compare with other annual plants, such as *Hibiscus cannabinun* (Pascoal Neto et al., 1996), *Alfa tenassissima* (Belgacem et al., 1986), or *Cyanara cardunculus* L. (Antunes et al., 1998, 2000), and lower than wood-based materials (Atchison, 1993). In spite of the high content of ashes, this raw material is worth pulping, mainly because of its relatively low lignin content.

The quantity of extractives in ethanol/toluene and water were 14.1 and 8.1%, for the whole material

Table 1
Chemical composition of banana crops (% w/w with respect to o.d. material)

	Ashes	Extractives			Lignin	Holocellulose[a]	Cellulose[a]
		Diethyl ether	Ethanol/toluene	Water			
Type I	13.9	0.6	4.6	8.9	12.0	60.1	34.5
Type II	14.6	0.4	2.3	5.4	12.7	65.2	40.2

[a] Corrected taking into account residual lignin. The relative errors of these data are in the range of ±2–3%. Type I: the whole material. Type II: the outer bark material, which seemed to be richer in fibres.

(type I) and outer back material (type II), respectively, corresponding mainly to polar extractives (95–96% of the total extractives). These values are relatively high when compared with wood and some other annual plants. Nevertheless, in some cases the amount of extractive components can be very high as for example for *C. cardunculus* L., 15–18% (Antunes et al., 2000), or *Arundo donax*, 14–23% (Pascoal Neto et al., 1997).

Table 1 also reports the results concerning the polysaccharide contents in both types of the residue studied here. Thus, the amount of holocellulose and cellulose for the type II material was higher than that of type I, which confirmed our visual observation concerning the outer bark material. Both values were reasonable, if one takes into account the high content of ashes and extractive components, and lead to promising perspectives concerning the use of these crops as a source of cellulose fibres, thus justifying the investigation dealing with the optimisation of the pulping process. The holocellulose content of the crops under investigation was higher than 60%, with respect to o.d. material, meaning a hemicellulose/cellulose ratio about 1:2 which is common to other vegetal species. This ratio is very important if one considers the capital role which hemicelluloses play in papermaking.

The results of the monosaccharides composition analysis from pseudo-stem are shown in Table 2. Glucose is the predominant monomer in this raw material with 74.0% followed by xylose, 13.1%; arabinose, 9.1%; galactose, 2.5% and mannose, 1.3%. The values found for glucose were higher than those obtained for other annual plants like *H. cannabinun* (Pascoal Neto et al., 1996) or *C. cardunculus* L. (Antunes et al., 2000). The composition of holocellulose is close to that of initial raw material; nevertheless, the loss of some arabinoses, glucoses and xyloses shows the same selectivity of the isolation method.

Table 2
Monosaccharides composition of *Musa acuminata* Colla pseudo-stem, holocellulose and hemicellulose (molar proportion, %)

Sample	Neutral sugar content (molar proportions, %)				
	Glu	Xyl	Gal	Ara	Man
Type I					
Pseudo-stem	74.0	13.1	2.5	9.1	1.3
Holocellulose	80.2	10.8	1.7	5.7	1.6
Hemicellulose I	29.3	44.1	6.4	20.2	nd
Hemicellulose II	74.6	12.7	1.9	8.5	2.3
Type II					
Holocellulose	81.4	10.8	1.4	5.2	1.2
Hemicellulose I	23.0	51.3	6.1	19.6	nd
Hemicellulose II	71.5	19.2	2.0	5.4	1.9

The relative errors of these data are in the range of ±2–4%.

The holocellulose fractionation by sequential extraction of with 5% KOH aqueous solution (hemicellulose I) and 24% KOH aqueous solution (hemicellulose II) was also analysed, and showed that the hemicellulose I is composed in major proportion by xylose (44%), while hemicellulose II is composed mainly by glucose (75%). The hemicellulose is composed by glucose, xylose, arabinose, galactose and mannose with molar proportion of 29:44:20:6:0, respectively for hemicellulose I and 75:13:8:2:2 for hemicellulose II.

Although the different contents of holocellulose and cellulose in the two types of material, the holocellulose and hemicellulose were constituted by the same monosaccharides albeit in different molar proportions (Table 2).

To the best of our knowledge, there are no detailed studies, on the quantification and characterisation of chemical constitution of the *M. acuminata* Colla; variety of banana plant, available in the literature. To fulfil this gap, we have recently started a research program aiming to deep the knowledge on chemical and structural constitution of the different morphological parts of banana plant.

3.2. Optimisation of pulping process

Before producing pulp from *M. acuminata* Colla pseudo-stem in a large scale and to avoid extensive consumption of raw material, delignification was carried out using rotational mini-digesters, which gave very suitable pulps to be investigated in terms of Kappa no., viscosity and sugar composition. The results obtained are shown in Figs. 1–3 and summarised in Tables 3–7. These data could be interpreted by following the next criteria.

3.2.1. Influence of material type

To study the behaviour of the two types of materials, we cooked banana pseudo-stem using 18% of sodium hydroxide, 0.15% of AQ and liquor/crops ratio: 5/1, at

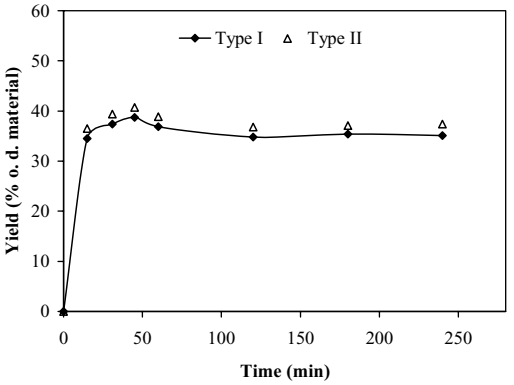

Fig. 1. Screened pulp yields as a function of cooking times for *Musa acuminata* Colla pseuto-stem crops. Cooking conditions: 18% of NaOH and 0.15% of anthraquinone (w/w with respect to o.d. material), a liquor/crops ratio: 5/1, 60 min of heating from room temperature to 120 °C and maintaining the delay indicated in figure.

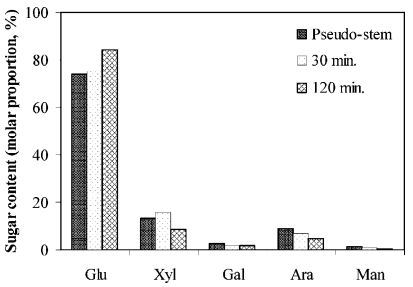

Fig. 2. Sugar composition as a function of cooking time.

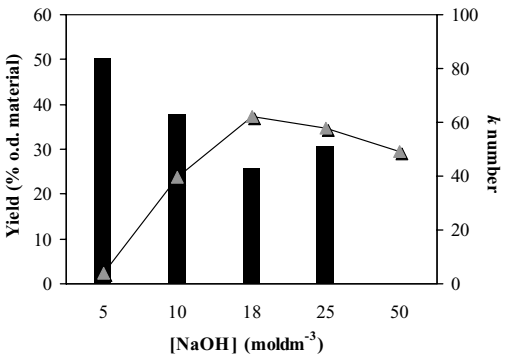

Fig. 3. Effect of the NaOH concentration on the screened pulp yield and Kappa no. (continuous line corresponds to the screened pulp yield whereas bars are relative to Kappa no.). Cooking conditions: a liquor/crops ratio: 5/1, 60 min of heating from room temperature to 120 °C and maintaining the reaction for 30 min.

Table 3
Kappa no., viscosity and degree of polymerisation as a function of delignification time

	Exp. no.	Time (min)	Kappa no.	Viscosity (g/cm^3)	Degree of polymerisation
$P_{120}t_{30}$	1	30	33.1	791	1254
$P_{120}t_{60}$	2	60	32.8	614	948
P_{120}	3	120	31.5	534	812

Cooking conditions: 18% of NaOH and 0.15% of anthraquinone (w/w with respect to o.d. material), a liquor/crops ratio: 5/1, 60 min of heating from room temperature to 120 °C. The relative errors of these data are in the range of ±4–5%.

different time at 120 °C (Fig. 1). As expected, type II material with higher cellulose content, reveals higher yields in pulp fibres. Nevertheless, the gain of the yield was modest, which is not significant enough to justify the addition of the crops separation. Thus, we decided to pulp the whole material and to study the other cooking variables. The Kappa no. for 30 min was 33.1 and 30.2, for types I and II, respectively.

3.2.2. Influence of time process

The delignification kinetics of *M. acuminata* Colla pseudo-stem was also studied, and is shown in Fig. 1. As it can be seen (i) the maximum yield was reached at 30–45 min, which is a very short cooking time; (ii) for times higher than 45 min the cooking time did not affect significantly the pulps yield. From Table 3 we can see that increasing the reaction time, decreased modestly the Kappa no. of the pulps, but at the same time induced polysaccharide degradation, as seen by

Table 4
Monosaccharide composition of pulps, obtained with different cooking conditions, from *Musa acuminata* Colla pseudo-steam (molar proportion, %)

Exp. no.		Neutral sugar content (molar proportions, %)				
		Glu	Xyl	Gal	Ara	Man
13	P_{90}	72.9	9.6	4.1	11.6	1.8
3	P_{120}	84.5	9.0	1.2	4.3	1.0
15	P_{160}	80.4	11.1	1.7	5.5	1.3
11	P_{10}	75.2	9.9	3.5	9.7	1.7
12	P_{25}	83.9	8.4	1.6	5.5	0.6
7	$P_{120}AQ_{0.25}t_{30}$	75.4	15.6	1.6	6.7	0.7
18	$P_{120}AQ_{0.25}t_{120}$	84.4	8.7	1.7	4.6	0.6
10	$P_{120}S_{18}$	81.2	9.7	1.6	6.4	1.1

The relative errors of these data are in the range of ±3–4%.

Table 5
Pulping conditions of *Musa acuminata* Colla and the properties of the obtained pulps (w/w with respect to o.d. material)

	Exp. no.	NaOH (%)	AQ (%)	Na_2S (%)	Yield (%)	Kappa no.	Viscosity (g/cm^3)	Degree of polymerisation
P_5	4	5	0	0	2.3	83.7	–	–
P_{10}	5	10	0	0	23.7	63.1	451	674
P_{120}	3	18	0	0	37.4	43.0	564	863
$P_{120}AQ_{0.15}$	6	18	0.15	0	40.2	33.1	791	1254
$P_{120}AQ_{0.25}$	7	18	0.25	0	38.2	32.4	870	1393
$P_{120}AQ_{0.35}$	8	18	0.35	0	37.0	30.6	–	–
$P_{120}S_5$	9	18	0	5	26.2	56.2	650	1009
$P_{120}S_{18}$	10	18	0	18	28.2	50.3	613	989
P_{25}	11	25	0	0	34.6	51.5	550	839
P_{50}	12	50	0	0	29.5	–	–	–

Cooking conditions: liquor/crops ratio: 5/1, 60 min of heating from room temperature to 120 °C and maintaining the reaction for 30 min. The relative errors of these data are in the range of ±4–5%.

Table 6
Yield and pulp characteristics obtained with different temperatures of soda process

	Exp. no.	Temperature (°C)	Time (min)	Yield (%)	Kappa no.	Viscosity (g/cm^3)	Degree of polymerisation
P_{90}	13	90	30	13.4	58.0	422	626
$P_{90}T_{180}$	14	90	180	28.0	–	–	–
P_{120}	3	120	30	35.8	43.0	564	863
P_{160}	15	160	30	28.6	42.5	499	754

Cooking conditions: 18% of NaOH (w/w with respect to o.d. material), a liquor/crops ratio: 5/1, 60 min of heating from room temperature to final temperature and maintaining the delay indicated in the table. The relative errors of these data are in the range of ±4–5%.

the decrease of the viscosity and consequently the decrease of polymerisation degree. This indicates that cooking with shorter time might offers pulps with better quality.

Besides the determination of the individual sugar content of *M. acuminata* Colla pseudo-stem, holocellulose and cellulose, reported in Table 2, and already discussed in Section 3.1, we also studied the sugar composition of pulps, obtained with different cooking conditions, as summarised in Table 4. From these data a first remark could be drawn: in different pulps the major monosaccharides present are glucose, xylose and arabinose. Galactose and mannose are also present although in minor extent (less than 6% of the total of monosaccharide).

The effect of pulping process time on the sugar composition could be observed by exp. nos. 7 and 18 (Table 4). These results show that the longer time cooked pulp has lower proportions of xylose, galactose and arabinose (Fig. 2), indicating that for longer cooking times, a higher degradation of hemicelluloses took place. This factor could be a justification for the decrease of the viscosity parameter with time, as discussed above.

Table 7
Yield and pulp characteristics obtained with different ratio of liquor/crops of soda process

	Exp. no.	Liquor/raw material (l/kg)	Yield (%)	Kappa no.	Viscosity (g/cm^3)	Degree of polymerisation
$P_{120}LB3/1$	16	3/1	21.1	53.9	546	833
P_{120}	3	5/1	35.8	43.0	564	863
$P_{120}LB7/1$	17	7/1	23.4	50.5	497	750

Cooking conditions: 18% of NaOH (w/w with respect to o.d. material), 60 min of heating from room temperature to 120 °C and maintaining the reaction for 30 min. The relative errors of these data are in the range of ±4–5%.

3.2.3. Influence of the sodium hydroxide concentration

The sodium hydroxide varied from 0 to 50% (w/w of o.d. material). The conditions used and some characterisations of the obtained pulp are summarised in Table 5. In the absence of sodium hydroxide, pulping did not occur. The increase in the sodium hydroxide concentration from 5 to 18% produced an increase in the screened pulp yield, from 2.3 to 37.4%, and a decrease in Kappa no. of pulps from 84 to 43, as shown in Fig. 3. This is probably due to the very weak alkali level in the first reaction system. For higher sodium hydroxide concentration, up to 25%, a decrease of the pulp yield was observed. This is due to the cellulosic fibres degradation, as stated by decreasing viscosity, and did not yield additional delignification because Kappa no. increased, possibly due to self-condensation reactions of lignin macromolecules.

Despite the fact that the yields are lower than those obtained from wood species and from some other annual plants (Atchison, 1993), the overall yield of pulp obtained with our raw material can be considered as acceptable, if one takes into account the fact that original material contains high amounts of ashes.

With respect to sugar composition we can see, by the data in Table 4, that pulps obtained at minor concentration of sodium hydroxide (10%) presented a very similar monosaccharide composition to raw material. For 18 and 25% of sodium hydroxide we can observe an increase in glucose proportion indicating a preferential extraction of cellulose in stronger alkaline conditions.

3.2.4. Influence of anthraquinone and sulphite addition in pulp process

The effect of the addition of AQ on the pulp yield of our material has also been studied, as shown in Table 5 (exp. nos. 6, 7 and 8). The addition of catalytic amounts of about 0.15% (w/w o.d. material), $P_{120}AQ_{0.15}$, revealed a slight increase of the pulp yield and a significant decrease of the Kappa no. The Kappa no. of the unbleached pulps reached a value of about 30, which is very interesting for our plant in particular, and for annual plants in general. The Klason lignin content in pulps was also determined and the results showed the same tendency as Kappa no.

With the addition of sulphite, 5 and 18% (w/w o.d. material) the yields were lower than those obtained in the absence of this additive and the Kappa no. became significantly higher. However, the pulps thus obtained possessed a higher viscosity and consequently a higher degree of polymerisation, as shown in Table 5 (exp. nos. 9 and 10). Due to the quite modest gain of yield with the addition of catalysts, AQ and Na_2S, we decided to carry out the delignification processes only with sodium hydroxide.

The sugar composition (Table 4, exp. nos. 7 and 10) showed that the catalytic effect of anthraquinone and sulphite provoked a preferential extraction of cellulose.

3.2.5. Influence of temperature and liquor/crops ratio

Tables 6 and 7 show the values obtained for yield, Kappa no., viscosity and degree of polymerisation, as a function of the variation in cooking temperature and liquor/banana crops ratio, respectively. The highest yield was reached at 120 °C and liquor/banana crops ratio of 5/1. Pulping at higher temperature significantly decreased the pulp yield and the viscosity, without significantly decreasing the Kappa no. liquor/banana crops ratio of 3/1 or 7/1 decreased considerably the pulp yield and increased the Kappa no.

The amount of each monosaccharide (Table 4) in the pulp obtained at lower temperature (90 °C) is similar to that observed for raw material, but with higher temperatures (120 and 160 °C) a decrease of glucose proportion was observed indicating a preferential degradation of cellulose.

4. Conclusions

The main objective of this study was to establish the suitability of *M. acuminata* Colla as a potential source of lignocellulosic fibres for paper and composites materials. By the present study in chemical composition and pulping optimisation process we conclude that: (i) regardless the high amount of ashes a good yield of pulping is obtained with an acceptable Kappa no.; (ii) the banana pseudo-stem separation in two parts did not present any significant increase in the pulp yield, which justify an additional separation operation in the crops, although the holocellulose content was found to be higher in the outer bark part; (iii) the optimal conditions of cooking are soft comparing to those used for vegetal species; (iv) the

monosaccharides composition study, of the different pulps obtained, show that in soft conditions the extraction of cellulose and hemicelluloses are similar, but in harder conditions the extraction of cellulose is preferential. The suitability of these fibres in the papermaking, boards and composite materials areas are under investigation and will be reported shortly.

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