

CELLULOSE CHEMISTRY AND TECHNOLOGY

INTERNATIONAL JOURNAL FOR PHYSICS, CHEMISTRY
AND TECHNOLOGY OF CELLULOSE AND LIGNIN

EDITED BY THE ROMANIAN ACADEMY

39 ⇨ 2005

5-6 ⇨ SEPTEMBER-
DECEMBER

REPRINT



© 2005, EDITURA ACADEMIEI ROMÂNE
Calea 13 Septembrie nr.13, 050711 București
sect. 5, tel. 4021-3188106, 4021-3188146
Fax: 4021-3182444, e-mail: edacad@ear.ro

CELLULOSE CHEMISTRY AND TECHNOLOGY

PULP AND PAPER PROPERTIES FROM DWARF CAVENDISH PSEUDO-STEMS

N. CORDEIRO*, M. N. BELGACEM**, D. CHAUSSY** and J. C. V. P. MOURA***

*Centro de Estudos da Macaronésia, Universidade da Madeira, 9000-390 Funchal, Portugal

**Ecole Française de Papeterie et des Industries Graphiques, BP65, 38402 St. Martin D'Hères,
France

***Departamento de Química, Universidade do Minho, 4700-320 Braga, Portugal

Wood is one of the most complex natural materials and Professor Cristofor Simionescu is one of the few scientists who got a real insight about its chemistry and technology. On behalf of all young scientists in this field, I would like to express my gratitude for the fundamental and applied knowledge you have built for science. We hope to read you and follow your precious advice.
All the best! M.N.B.

Received October 8, 2004

Pseudo-stem from *Musa acuminata* Colla, var. *cavendish* was studied for its possible use as a source of pulp fibres. The chemical composition, i.e. the hollockulose (60%) and the low lignin content (12%), encouraged its potential use in papermaking applications. A systematic study was conducted, aiming at fibre's characterisation and investigation of the physical and mechanical properties of the virgin pulps, as well as of those arising from two different blends (with eucalyptus and pine fibres). Pulping was carried out by soda, kraft and soda-anthraquinone cooking processes. The pulps obtained were fractionated and the morphology was determined in terms of fibre lengths and diameter of both global and fractionated fibres. It was revealed that the pulp contained a high amount of short fibres (70% of the fibres, w/w %, lengths less than 0.5 mm), alongwith fibres with a wide distribution of lengths (0.20-4.19 mm). Isotropic handsheets from unrefined pulps were made and their physical and mechanical properties investigated. Scanning electron microscopy showed a fibrillation phenomenon. Finally, these fibres showed interesting potentialities in terms of burst index and breaking length, both when used alone or in combination with other common pulps.

Key words: *Musa acuminata* Colla, non-wood pulp, fibres, paper properties.

Cellulose Chem. Technol., 39, 5-6, 517-529 (2005)

INTRODUCTION

In the last century, wood became the dominant fibre source on the pulp and paper market. Technological innovations resulted in the economic preference of wood over agricultural fibres, previously used in industry. The agricultural sources of fibre for papermaking are being re-evaluated in Europe and North America as an option to improve the competitiveness of the pulp and paper industry, particularly since the sources of wood fibre become increasingly scarce. Agricultural fibres in North America include residues from food remainings such as cereal straw, annual dedicated fibre crops, such as hemp, kenaf and fibre flax, and more recently, perennial warm season grasses, such as switchgrass.¹ Although non-wood fibres have been used for papermaking for two millennia, the use of non-wood fibres is limited to about 9% of world's pulp production.² As a result of the actual environmental problems, there is an increasing interest in non-wood fibres as a source for pulp and paper production. The main part of non-wood fibre pulp is produced from short fibre sources such as straw and bagasse. These pulps are comparable with hardwood pulps normally used in printing and writing paper grades, because of their ability to give good surface properties. The quality of the short fibre pulps is dictated mainly by their coarseness, stiffness and optical properties of the ensuing paper mat, rather than by the strength properties of the paper-base materials.³⁻⁵

In the island of Madeira, due to the edaphic conditions, there is a reduced forest area of pine and eucalyptus, which results in a total dependence on these types of raw materials and on their by-products. However, high amounts of agricultural residues are produced from the commercialization of bananas, which has a capital importance in this island. *Musa acuminata* Colla *var cavendish* is the main variety used for banana production, since it is responsible for 50 to 60% of its total volume. These crops can be valorised as a source of fibres by conventional cooking and/or mechanical treatment, in order to isolate the lignocellulosic materials. In this context, a research program has been recently initiated, for getting deep knowledge on the chemical and structural composition of banana plants,^{6,7} as well as on the use of these fibres in papermaking composition⁸⁻¹⁰ and in biocomposite materials.^{11,12}

To the best of our knowledge, the few works dealing with the use of banana crops as a source of fibres concern varieties quite different from our raw material, namely: *Musa paradisiaca* L. and *Musa sapientum*.^{13,14} Such studies showed that 9% of the active alkali cooking of *M. paradisiaca* L. crops produced pulps of 59% yield, and a Kappa number of 30. The fibres obtained by kraft cooking have an average length of about 3.9 mm, which explains their high strength properties.¹³ According to literature data,¹⁵ the pseudo-stem of this plant evidences long fibres (*ca.* 5 mm).

In a previous paper,⁹ the chemical composition of banana pseudo-stem from *Musa acuminata* Colla *var cavendish* and their soda, soda-anthraquinone and kraft pulping optimization were studied. The present paper is a continuation of the previous work, aiming at accessing its papermaking potentialities. Thus, a systematic study was conducted for examining fibres' bleachability, their morphology and the physical properties of their paper-based product. In fact, the morphological characterization of different fractions is presented, as well as the physical and mechanical features of paper. Some trials of blending the banana-based fibres with more conventional ones (*Eucalyptus globulus* and *Pinus pinaster*) will be reported.

EXPERIMENTAL

The pulps were made from banana pseudo stems (*Musa acuminata* Colla *var cavendish*), as described elsewhere.⁹ Briefly, the effects of the (i) raw material type; (ii) temperature; (iii) sodium hydroxide concentration; (iv) liquor/crops ratio; (v) time at constant temperature; (vi) anthraquinone (AQ) concentrations; and (vii) sodium sulfite amount, were studied, as summarized in Table 1, which gives a partial report on the most relevant data. The sample selected here as a starting material for papermaking concerns the P₁₂₀, a soda pulp obtained at 120° with a NaOH concentration of 18% , giving a pulp yield of about 37%.

TABLE 1

Cooking conditions^a and pulp characteristics of pseudo-stem from *Dwarf Cavendish* (% w/w, o.d. material)

	Temperature	NaOH	Additive	Yield, (%)	Polymerisation degree
P ₁₂₀	120 °C	-	-	37.4	863
P ₁₂₀ AQ _{0.15}		18%	0.15% AQ	40.2	1254
P ₁₂₀ AQ _{0.25}			0.25% AQ	38.2	1393
P ₁₂₀ S ₁₈			18% Na ₂ S	28.2	989
P ₁₀		10%	-	23.7	674
P ₂₅		25%	-	34.6	839
P ₉₀	90°C		-	13.4	626
P ₁₆₀	160°C	18%	-	28.6	754

^a Cooking conditions: liquor/crops ratio: 5/1, 60 min of heating from room to final temperature, and 30 min at this constant temperature.

The relative errors of these data occur in the ±4–5% range.

The pulps, fractionated with a Bauer-McNett classifier, gave four fibres fractions, namely: (i) those retained on a screen with a 50 mesh opening size (F₅₀); the pulp fraction retained between screens of 50 and 100 mesh (F₅₀₋₁₀₀); those collected between screens of 100 and 140 (F₁₀₀₋₁₄₀); and the fine fraction passing through a 140 mesh screen (F₁₄₀). Fibre length, weight and diameter were determined by a Morfi fibre image analyser equipment of integral pulps and of different pulp fractions. Drainage resistance (°SR) was determined for non-refined pulps concerning banana-based fibres, whereas the pulps obtained from *Eucalyptus globulus* and *Pinus pinaster* were refined up to 40°SR, using a Valley-beater, on following the standard procedure.

For testing paper's properties, standard isotropic handsheets from integral pulps and the fraction retained on a screen with opening size of 50 mesh from P₁₂₀, were made. The basic weight of the handsheets prepared was about 60 g/m². Bleaching of selected pulp was carried out with a conventional procedure, following a QPQP sequence. Handsheet papers made from 20 and 50% (w/w) banana crops pulp with *Eucalyptus globulus* and *Pinus pinaster* pulp were also studied.

The paper samples were characterized in terms of their physical and mechanical properties using equipment and procedures according to TAPPI (Technical Association of the Pulp and Paper Industry) standards. The morphology of the handsheet papers was analyzed by scanning electron microscopy (SEM) on a JEOL JSM-6100, whereas contact angle measurements were carried out with a home-made apparatus designed to acquire dynamic images, as described in a previous publication.¹⁶

RESULTS AND DISCUSSION

Fibre fractionation and morphology characterization

Pseudo-stem pulp fibres from *Musa acuminata* Colla *var cavendish* were fractionated with a Bauer-McNett classifier, different fractions (P₉₀, P₁₂₀ and P_{120S}₁₈) being collected, as shown in Table 2. From these data, some concluding remarks could be drawn. In soda cooking processes, increase of temperature from 90 to 120°: (i) increased the amount of fibres retained on 50 mesh screen (F₅₀), which corresponds to relatively long fibres, from 8% to 37%; (ii) decreased the amount of F₁₀₀₋₁₄₀ with about 52%, and (iii) decreased the fine fraction (lower than 140 mesh screen, F₁₄₀) from 62 to 25%. It is worth noting that the tested samples were made of fibres collected after cooking, disintegration and washing. The uncooked materials were kept out these tests. In other words, for example, the fibre tested in P₉₀ pulps showed only a 13% yield. The data show that pulping at low temperatures allowed a more efficient extraction of the short fibres, and that the energy given for the cooking process was not high enough to extract long fibres, as shown by the very low yield and the very high amount of uncooked materials.⁹

TABLE 2

Bauer-McNett classification of pulps obtained by soda (at 90 and 120°) and Kraft processes at 120° (% w/w o.d. material)

	F ₅₀	F ₅₀₋₁₀₀	F ₁₀₀₋₁₄₀	F ₁₄₀
P ₉₀	8.0	8.3	21.6	62.1
P ₁₂₀	36.8	27.6	10.4	25.2
P _{120S} ₁₈	48.0	32.3	9.4	10.3

The kraft process yielded high amounts of long fibres, since the sum of the F₅₀ and F₅₀₋₁₀₀ contents was about 80% of the integral pulps. Thus, as expected, addition of sodium sulfite played an important role in polysaccharide preservation, leading to the extraction of higher amounts of long fibres. These values agree with the polymerization degree obtained for these pulps, as reported in Table 1. In fact, the DP of these pulps followed the same trend as their length.

Table 3 summarises the morphological characteristics of the different fractions under investigation. The first remark concerns the fibre length of integral pulps, which could be considered as relatively high (1.5 mm is between softwood and hardwood fibres¹⁷). Then, quite logical tendencies were observed here, namely a systematic decrease of the fibre length as a function of the collected fractions. Finally, the addition of anthraquinone or sodium sulfite led to the extraction of higher amounts of long fibres, exceeding 1.45 mm. As mentioned above, this result can be attributed to the protective capacity of the polysaccharides of these two additives. Thickness of the integral pulps was around 30–40 µm, independently on the extraction mode. These values are similar to those obtained from wood-based fibres and agree with the fibre length of these materials.

TABLE 3
Fibre morphology of different pulps from "Dwarf Cavendish" pseudo-stem, using a Morfi equipment

		Weighed average length (mm)	Arithmetic average length (mm)	Diameter (µm)
P ₁₆₀	Integral	1.33	0.97	32.0
	F ₅₀	1.35	0.83	27.4
	F ₅₀₋₁₀₀	1.87	1.25	25.3
	F ₁₀₀₋₁₄₀	1.06	0.95	34.7
	F ₁₄₀	0.49	0.48	36.8
	F ₁₄₀	0.55	0.43	28.9
P ₉₀	Integral	0.83	0.55	39.2
	F ₅₀	1.76	1.25	35.0
	F ₅₀₋₁₀₀	1.09	0.74	33.1
	F ₁₀₀₋₁₄₀	0.8	0.57	31.3
	F ₁₄₀	0.64	0.49	29.4
P _{120S} ₁₈	Integral	1.50	1.00	33.2
	F ₅₀	1.81	1.48	25.4
	F ₅₀₋₁₀₀	1.08	0.73	26.3
	F ₁₀₀₋₁₄₀	0.64	0.47	26.4
	F ₁₄₀	0.52	0.41	32.1
P ₁₂₀ AQ _{0.15}		1.49	1.22	31.2
P ₁₂₀ AQ _{0.25}		1.78	1.27	30.3
P ₁₀		1.29	0.77	35.9
P ₂₅		1.60	1.05	36.2

Fig. 1 shows the average weighed fibres length distribution for the integral pulp investigated here (P₁₂₀), as well as that of the four fractions obtained by Bauer-McNett classification. According to the Fig., the Bauer-McNett classification seemed to be successful, since the length distribution of the different

fractions studied was sharp and had a close distribution. Moreover, it should be specified that:

- (i) fraction F_{50} was mainly composed (70%) of fibres with lengths ranging from 1.14 to 2.71 mm;
- (ii) fraction F_{50-100} showed widely distributed fibre lengths, with a contribution of around 20% for the first four classes. Thus, the fibre length distributions of this fraction were of 20, 23, 23 and 16% for the 0.20-0.31, 0.31-0.48, 0.48-0.74 and 0.74-1.14 mm, respectively;
- (iii) fraction $F_{100-140}$ showed a sharp distribution of the fibre lengths, centred around 0.31-0.48 mm, corresponding to 30% of the total fraction;
- (iv) fraction F_{140} showed that about 70% of the fibre had a length below 0.48mm.

Fig. 2, plotting the light microphotography of the P_{120} pulp, shows that cell types could be distinguished, as in most non-wood pulps.¹⁸ Other cell types were also detected, including epidermal cells with hook-like serrulate margins, parenchyma cells ranging from ball-shaped to barrel-shaped, vessel elements with or without pitting and with open ends.

Handsheet paper from P_{120} was examined by scanning electron microscope, in both surface and cross-section, as shown in Figs. 3 and 4, which reveal that fibres displayed fibrillation phenomena much more pronounced than in usual wood-based fibres. The cross-section of these papers was also studied, as reported in the microphotographs presented in Fig. 4, leading to two main conclusions: (i) the porosity of these materials is much reduced, and (ii) the existence of some residual mineral crystals (Fig. 4b and 4c) is evidenced. This result was confirmed by the determination of ashes in virgin pulps, which was around 15%.

Paper Properties

Isotropic handsheet papers from P_{120} (unbleached and bleached), P_{90} , the fraction F_{50} of P_{120} , and sheet paper from *Eucalyptus globulus* and *Pinus pinaster* with 0, 20 and 50% unbleached P_{120} banana pulp, were prepared and characterized by standard methods. Table 4 summarizes the results obtained on the physical and mechanical properties. The integral pulps from banana crops (bleached and unbleached) presented very high values of drainage resistance, i.e., 67°SR. These abnormally high values, if one considers that these pulps are unbeaten, could be attributed to fibers' high flexibility and also to the relatively high amount of fines. As a matter of fact, the fibre flexibility of three different pulps was determined in laboratory conditions, the values obtained being of about 25, 50 and 75%, for *Pinus pinaster*, *Cynara carunculul* and banana fibres (P_{120}), respectively. Nevertheless, for a long fibre length fraction (F_{50}), the drainage rate was close to that of conventional pulps with similar morphology. This property has a direct incidence on sheet density and, consequently, on all physical and mechanical properties. Thus, a low permeability (less than 15 mL/min) of the paper sheets made from banana-based integral pulps was systemically observed, as well as in the papers in which they were incorporated, even at low levels (20%) (Table 4). Even

for fraction F_{50} , constituted mainly of fibres with lengths higher than 1 mm, it is surprising to observe the weak value of Bendsten permeability (35 to compare with about 120 mL/min for conventional pulps).

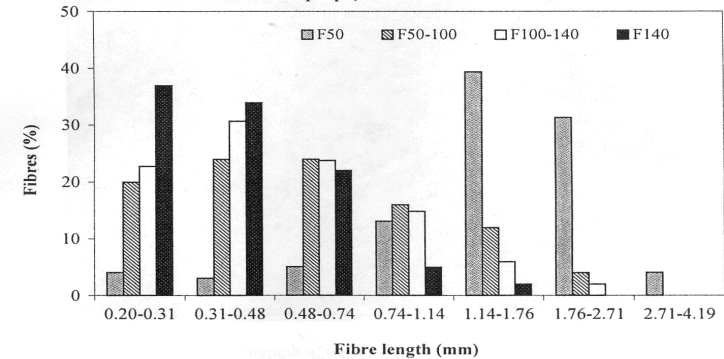


Fig. 1. – Weighed average length fibre distribution at different temperatures (P_{90} , P_{120} , P_{160}).



Fig. 2. – Micrograph (10X) of P_{120} pulp from *Dwarf Cavendish* pseudo-stem: (A) fibre and (B) ground tissue cell.



Fig. 3.(A) – SEM microphotograph of top surface of handsheets made of *Dwarf Cavendish* pseudo-stem: (A) $\times 1500$;

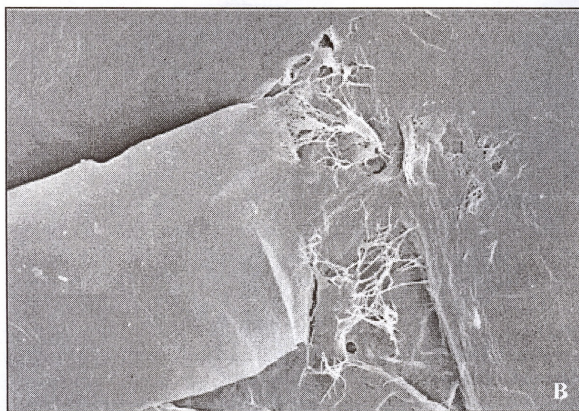


Fig. 3.(B) – SEM microphotograph of top surface of handsheets made of *Dwarf Cavendish* pseudo-stem: (B) $\times 1500$;

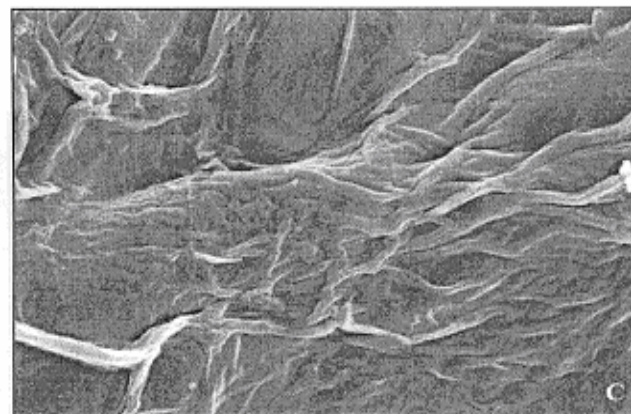


Fig. 3.(C) – SEM microphotograph of top surface of handsheets made of *Dwarf Cavendish* pseudo-stem: (C) $\times 4000$.

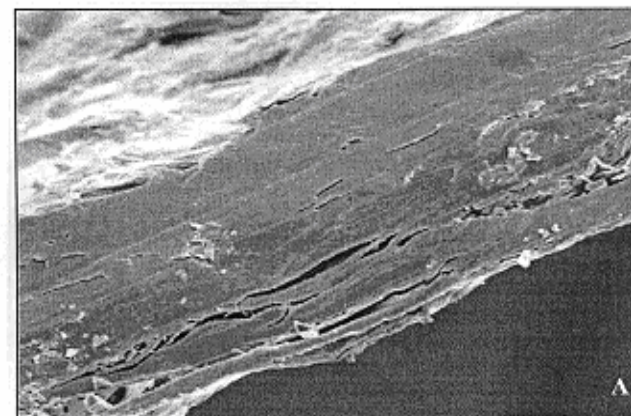


Fig. 4.(A) – SEM microphotograph of the cross-section of handsheets made from *Dwarf Cavendish* pseudo-stem: (A) $\times 1000$;

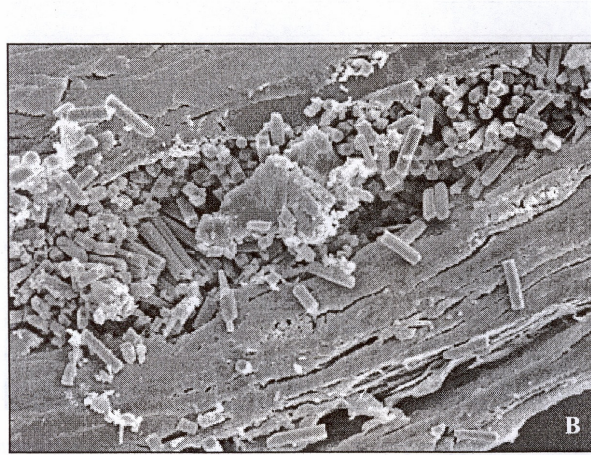


Fig. 4.(B) – SEM microphotograph of the cross-section of handsheets made from *Dwarf Cavendish* pseudo-stem: (B) $\times 1500$;

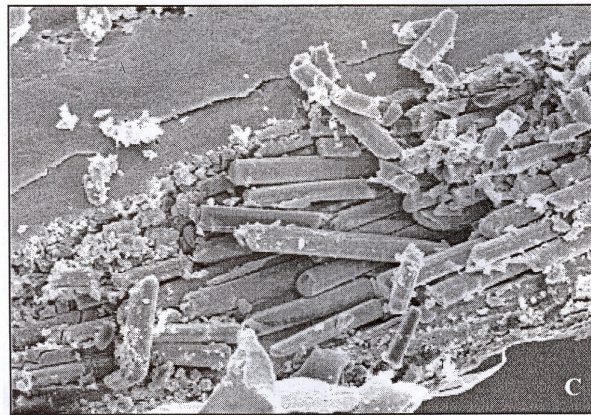


Fig. 4.(C) – SEM microphotograph of the cross-section of handsheets made from *Dwarf Cavendish* pseudo-stem: (C) $\times 2000$.

TABLE 4
Physical and mechanical properties of handsheets based on *Dwarf Cavendish* pseudo-stems

	P ₁₂₀		P ₉₀		P ₁₂₀		Euc. <i>P.120</i>		Pinun		pin/P ₁₂₀	
	Bleached		F ₅₀		Euc. <i>P.120</i>		50/50		pinaster		pin/P ₁₂₀	
	Unbleached	Bleached	Unbleached	Bleached	80/20	50/50	80/20	50/50	80/20	50/50	80/20	50/50
Drainage resistance (°SR)	67	–	67	21	40	60	65	40	59	66	66	66
Basis weight (g.m ⁻²)	55	55	83	76	69	66	66	75	69	66	66	66
Density (g.cm ⁻³)	0.58	0.67	0.69	0.68	0.71	0.70	0.76	0.71	0.60	0.70	0.70	0.70
Bendtsen permeability (mL/min)	7.86	5.68	n.d.	34.9	117.8	15.0	8.52	120	10.0	7.3	7.3	7.3
Tear index (mN.m ² .g ⁻¹)	1.8	1.2	1.9	3.9	2.9	2.8	2.3	5.5	4.5	3.5	3.5	3.5
Burst index (kPa.m ² .g ⁻¹)	4.77	4.39	3.29	3.29	6.29	5.35	4.24	3.39	3.98	4.36	4.36	4.36
Z-Span tensile (dry) (km)	10.3	9.40	8.26	13.3	15.8	14.3	14.2	16.4	15.1	15.1	15.1	15.1
Young modulus (GPa)	2.78	2.65	2.63	2.85	2.44	2.74	2.96	2.67	2.90	2.68	2.68	2.68
Tensile index (N.m.g ⁻¹)	68.5	82.4	61.9	117.2	59.6	99.0	63.2	71.9	117.3	75.8	75.8	75.8
Elongation at break (%)	5.7	5.7	4.1	4.8	5.4	5.0	5.3	5.4	5.5	5.1	5.1	5.1
Breaking length (km)	7.0	8.4	6.3	11.9	6.1	10.1	6.4	7.3	12.0	7.7	7.7	7.7

The mechanical properties of the handsheets from pure banana fibres, as well as those made by their incorporation into wood pulps (*Eucalyptus globulus* and *Pinus pinaster*) were studied, as summarised in Table 4. The results obtained showed that:

- (i) bleaching of the banana fibres increased the tensile properties and breaking length of the handsheets;
- (ii) increasing the temperature increased the tensile properties and breaking length, as already observed by Kokta *et al.*;¹⁹
- (iii) as expected, the F50 of the banana fibres decreased the °SR and increased the tear index and breaking length. It is worth noting that sheet density remained constant because of the high fibre flexibility (see above);
- (iv) the modulus and elongation at break did not show any clear tendency;
- (v) the paper made from pulps blends showed a sort of synergy effect in the case of using 20% banana fibres. This is a quite unexpected result, since the literature reports that mixing of pulps gives a linear dependency of the properties²⁰ or a negative trend.²¹ In fact, in our study, mixing of 20% banana with eucalyptus or pine fibres gave rise to a substantial increase in the breaking length for both eucalyptus- and pine-based handsheets. In other words, the breaking length of these two blends was expected to be around 7 km, but, in reality, the values found were of 10.1 and 12.0 km, for eucalyptus- and pine-based handsheets, respectively. Surprisingly, this effect was not observed for the burst index.

The contact angles of the handsheet papers from P₁₂₀ were measured with five different liquids, in order to determine both the dispersive and the polar contribution of these materials to surface energy, according to the Owens-Wendt method.²² The calculated total surface energy gives a value of 41 mNm⁻¹, with 32 and 9 mNm⁻¹ for γ_s^D and γ_s^P , respectively, a result very close to that obtained for other similar raw materials.²³

CONCLUSIONS

This work deals with the study of the potential use of industrial crops as a source of pulp for papermaking purposes. The fibrous material obtained, characterized thoroughly, showed that fibres' average length was relatively high, but with a high proportion of short fibres, i.e., 70% of the fibres had a length lower than 0.5 mm. This explains the high °SR for unbeaten pulps (67°SR). As expected, the increase of cooking temperature was found to have a positive effect on the mechanical properties. The fibres obtained from banana crops were naturally highly fibrillated, although they were not submitted to refining processes. Moreover, part of the mineral crystals initially present in the starting materials remained onto the fibres, which gives naturally filled papers. The paper made from

pulp blends showed that the addition of 20% banana fibres had a good synergy on the mechanical properties of the resulting sheets. This encouraging result opens the way for valorising such crops in different pulps formulations, including those based on recycled fibres, since they displayed a good burst index and breaking length.

ACKNOWLEDGEMENTS. The authors would like to thank to the Portuguese Foundation for Science and Technology (FC1) which has sponsored this work. Many thanks also to the Department of Science and Technology of Paper - University of Beira Interior, for allowing the use of their facilities.

REFERENCES

- ¹ P. Girouard and R. Samson, *84th Annual Meeting, Technical Section, CPPA: Preprints B.*, 105 (1998).
- ² C. Ververis, K. Georghiou, N. Christodoulakis, P. Santas and R. Santas, *Ind. Crops and Prod.*, **19**, 245 (2004).
- ³ K. G. Satyanarayana, K. Sukumaran, A. G. Kulkarni, S. G. K. Pillai and Rohatgi, *Composites*, **17**, 329 (1986).
- ⁴ W. H. Zhu, B. C. Tobias, R. S. P. Coutts and G. Langfors, *Cem. Conc. Compos.*, **16**, 3 (1994).
- ⁵ L. A. Pothan, S. Thomas and Neelakantan, *J. Reinf. Plast. Comp.*, **16**, 744 (1997).
- ⁶ L. Oliveira, N. Cordeiro, A. J. D. Silvestre, I. C. Torres and D. Evtuguin, *Ind. Crops Prod.*, in press, 2004a.
- ⁷ L. Oliveira, N. Cordeiro, A. J. D. Silvestre, I. C. Torres and D. Evtuguin, *Ind. Crops Prod.*, submitted, 2004b.
- ⁸ N. Cordeiro, M. N. Belgacem, I. C. Torres and J. C. V. P. Moura, *Proceedings of the 28th EUCEPA Conference*, Lisboa, Portugal, 333 (2003).
- ⁹ N. Cordeiro, M. N. Belgacem, I. C. Torres and J. C. V. P. Moura, *Ind. Crops Prod.*, **19**, 147 (2004).
- ¹⁰ S. Schott, D. Chaussy, E. Mauret, I. Desloges, A. Antunes, N. Cordeiro and M. N. Belgacem, *Proceedings of the International Conference on Chemical Technology of Wood, Pulp and Paper*, Bratislava, Slovakia, 167 (2003).
- ¹¹ H. Faria, L. Oliveira, N. Cordeiro and M. N. Belgacem, *Proceedings of the International Congress in Production, Processing and Use of Natural Fibres*, Potsdam, Berlin, 94 (2002).
- ¹² H. Faria, L. Oliveira, N. Cordeiro, M. N. Belgacem, and A. Dufresne, *J. Appl. Polym. Sci.*, submitted, 2004.
- ¹³ N. A. Darkwa, *Proceedings of the 1998 Pulping Conference*, Montreal, Que., Canada, TAPPI Press, 645 (1998).
- ¹⁴ M. L. A. P. Soffner, F. G. Jr. Silva, J. O. Brito and L. L. Pereira, *Proceedings of the Fourth International Nonwood Fibre Pulping and Papermaking Conference*, Jinan, China, **2**, 535 (2000).
- ¹⁵ M. H. Williams, M. Vesik and M. G. Mullins, *Can. J. Bot.*, **67**, 2154 (1989).
- ¹⁶ P. Aurenty, V. Lanet, A. Tessadro and A. Gandini, *Rev. Sci. Instrum.*, **68**, 1801 (1997).
- ¹⁷ J. E. Atchison, "Nonwood plant fiber pulping", Progress report n°19, Tappi Press, Atlanta, 1 (1991).
- ¹⁸ W. J. Hua and H. N. Xi, *Appita*, **41**, 361 (1998).
- ¹⁹ B. V. Kokta, A. Ahmed, H.-Y. Zhan and M.-C. Barbe, *Paperi ja Puu*, **71**, 1044 (1989).
- ²⁰ P. Krkoska, P. Misovec and A. Blazec, *Cellulose Chem. Technol.*, **23**, 455 (1989).
- ²¹ E. Retuainen, *Paperi ja Puu*, **74**, 419 (1992).
- ²² D. K. Owens and R. C. Wendt, *J. Appl. Polym. Sci.*, **13**, 1741 (1969).
- ²³ G. M. Dorris and D. G. Gray, *J. Colloid Interface Sci.*, **77**, 353 (1980).