Primary and secondary fines from *Eucalyptus globulus* kraft pulps. Characterization and influence

Kerwords

<u>E. globulus</u> kraft pulps, primary and secondary fines, fibres and fines measurements, fines content, pulp drainability and handsheet properties.

Abstract

The main topic of this study is the characterisation of primary and secondary fines in a chemical kraft pulp. The former were removed from an unbeaten pulp and the latter from the corresponding decrilled pulp after beating, being their size distributions measured with the Coulter-Multisizer in terms of an equivalent volume diameter. Additionally, some morphological parameters of the fibres were also determined, such as length, width and curl. Handsheets were made with the whole and the decrilled and the unbeaten and the beaten pulps in order to investigate the role of both kinds of fines separately.

Introduction

This study is part of a more comprehensive work concerning the beating of *E. globulus* kraft pulps, namely the evaluation of its effects on the physical characteristics of the fibrous material and on the final paper properties.

The main effects of beating are /1/: i) external fibrillation, as a result of primary wall and secondary layers removal; ii) internal fibrillation, as a result of the breakage of intermolecular bonds; and iii) fibre shortening, as a result of the cutting action during beating. Fibrillation originates an increase in fibre-to-fibre bonding ability through an enhancement in the area available for bonding and in fibre swelling. In addition, fibrillation also increases fibre flexibility and collapsibility, thus improving fibre contormability in the network. On the other

hand, both external fibrillation and fibre shortening produce fines which are essentially sheet-like fibre wall fragments and fibrilar material (fibrils, microfibrils bundles and fibre ends) removed by the rubbing and crushing actions during beating. These are known as secondary fines while those initially present in the pulp and resulting from the chipping and the cooking processes comprising short or broken fibres, parenchyma cells, vessels and other cells from the stem of the trees, are known as primary fines /2–5/.

It is widely recognised that fines originate an increase in fibre-to-fibre bonding, due to their bridging effect /4,6,7/ and their swelling ability /5,8/ (which improves Campbell forces but in turn adversely affects the drainage characteristics of the pulp). Fines also decrease air permeability and increase sheet density by filling interstices between fibres and bringing fibres close together, and contribute to a better stress distribution over the whole surface of the bond 15-71. As a consequence, they play an outstanding role on the mechanical and optical properties of paper /1,3-10/. Nevertheless, little is known about their morphological characteristics or about the main differences between primary and secondary fines regarding size, shape and influence on the quality of the end-product, specially as far as chemical pulps are concerned. The lack of information is particularly significant for hardwood kraft pulps.

This work is mainly focused on the determination of sizes, size distributions and shapes of both primary and secondary fines of an *E. globulus* kraft pulp. Besides, it also includes the study of the influence of fines on the handsheet strength and optical properties. For this, a systematic procedure comprising beating, separation of fines before and after beating, and preparation of handsheets with and without fines was undertak-

en. Additional information about the content of fines and their role on the drainability of pulps, and the length and the curliness of the beaten and unbeaten fibres is also provided.

Experimental

Sample preparation

The unbleached kraft pulp used in this study was produced in the laboratory by cooking Eucalyptus globulus wood chips from a 10 year old specified clone in a MK circulation digester. The cooking liquor alkaline charge and sulphidity were 15% and 20%, respectively, and the pulp kappa number 15 (similar to that obtained in the Portuguese pulp and paper mills which process identical raw material).

Beaten pulps were obtained in a PFI laboratory mill, as specified in the ISO 5264/2. A moderate beating level of 2000 revolutions was applied since preceding studies had revealed that this was adequate to assess morphological changes in fibre structure and the corresponding formation of fines, maintaining however the final paper properties within an acceptable range. The beating degree was quantified by comparing the drainage characteristics of the unbeaten and the beaten pulps conforming to the Schopper-Riegler (SR) freeness method (ISO 5267/1). The effect of fines on the drainability of the pulps has also been evaluated by measuring the SR number before and after fines removal.

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Fines separation

Pulp fines are frequently considered to be the fraction of material passing through a 100 -mesh /5,11,12/ or through a 200-mesh screen /2-4,7-9/ of a classifier. The latter definition, which is the most common, was adopted in this work. In order to obtain sufficient quantities of fines-free pulp (decrilled pulp) necessary for Schopper-Riegler measurements, for making handsheets and for fibres characterisation, the Bauer-McNett classifier was used according to Tappi T 233 cm-82 method. This procedure also enabled the calculation by difference of the weight percentage of fines in each pulp.

As the fines fractions from the Bauer-McNett are extremely diluted, the separation of fines for their subsequent size analysis was carried out in the Dynamic Drainage Jar (DDJ), as specified in the Tappi T 261 cm-94 method. To keep the fines concentration at a reasonable level, only the 500 ml of filtrate corresponding to the first screening was retained – preliminary tests have indicated that most of the fines which are ultimately separated are present in this first filtrate.

Primary fines were directly separated from the unbeaten pulp and characterised in terms of size and shape. Part of the original pulp was refined and the mixture of primary and secondary fines was subsequently separated for particle sizing. For the unbeaten as well as for the beaten pulps standard handsheets were prepared with both the decrilled and the whole pulp fractions. Finally, a sample of decrilled unbeaten pulp was further beaten in order to obtain a portion of fine material exclusively constituted by secondary fines essential for particle characterisation. Like before, handsheets of this pulp were made with and without secondary fines.

Fibre measurements

Among the several techniques and instruments available for measuring fibres (classifiers, optical or electrical devices /5,13,14/), it was decided to employ the Kajaani FS-200 /14/ and also a computer-controlled microscopic image analysis system. The former was chosen due to its extensive use in the pulp and paper industry, while the latter was utilised as reference since it is an absolute method. Additionally, the microscope enables the direct visualisation of fibres and therefore, the assessment of other morphological parameters such as fibre width and curvature. Both analysers have already been compared in a previous study /15/.

For each pulp tested with the Kajaani, stock suspensions of approximately 0.010%

consistency were prepared and further diluted so that an average of 20 000 fibres was counted in each 500 ml. Triplicate measurements of two independent suspensions were performed and the average of the results so obtained was considered.

The microscope measurements were only conducted with decrilled unbeaten and beaten pulps. In fact, the presence of fines highly disturbs the image acquisition and is irrelevant for quantifying fibre shortening. Microscopic slides containing dyed and air dried fibres were placed on the stage of an Olympus microscope (BH-2) coupled to a CCD camera, and the analyses were carried out in the manual mode with a magnification corresponding to 1.67 µm per pixel and a camera factor of 0.68. The number of counted fibres was selected in such a way that a deviation of 5% relative to the arithmetic average specific length at a 95% confidence level was reached. This counting corresponded to two hundred fibres on average and involved different slides of each pulp.

Fines measurements

Although image analysis has been used in some works to measure fines /3,7,9/, it presents some major drawbacks. Indeed, the physical and chemical heterogeneity of the fines particles (with flakelike material mixed together with brighter fibrilar fines) renders the accurate object detection, the threshold definition and the subsequent segmentation rather difficult. The main consequence is that some fine material may be lost. Even when using elaborate algorithms to improve thresholding, such as the relative entropy and joint entropy algorithm /3/, the results are still strongly dependent on the sample preparation, on the criteria utilised by the operator and on the number of analysed particles. This number must be large enough in order to gain statistical information about the image content, which makes this analysis very tedious and time consuming. For all these reasons, it was decided to use the microscope not as a measuring device but mainly to provide a random two-dimensional visualisation of the fines and to anticipate some information about shape and size differences between primary and secondary fines.

A comparative study between techniques for the measurement of fines /16/ has revealed that the electrical sensing zone technique /17/ (Coulter-Counter method) seems to be one of the most promising. In fact, this technique besides being fast and in principal not affected by the particle shape and optical properties, is operator independent, gives accurate numerical as well as

volume distributions, and is quite reproducible even for low solids concentration suspensions (as it is the case of the secondary fines suspensions). However, as fines are not spherical but rather irregular (most having an elongated or flakelike shape), the equivalent volume diameters may deviate significantly from the real particle dimensions. In addition, since there is no visual control of fines, fibres eventually passing the 200 mesh screen openings which in image analysis are eliminated, will also be included in this analysis /2/.

In the present work, the fines separated in the Dynamic Drainage Jar were sized with the Coulter-Multisizer II unit, using a saline isotonic solution as suspending medium (Isoton, supplied by Coulter Electronics). A 100 µm aperture tube, covering the 2 μm to 63 μm range of equivalent volume diameters (d_j), was found adequate for all samples (smaller apertures tend to obstruct with such irregular fines). Experiments were performed in the siphon mode (2 ml), with coincidence correction and background subtraction. For the various filtrates (containing primary, primary plus secondary, and secondary fines), at least two independent suspensions were prepared and for each one, five measurements were performed. Average values were obtained for the size distributions as well as for the corresponding mean equivalent diameters.

Finally, laboratory sheets of the unbeaten and the beaten pulps, including and excluding fines, were prepared and tested against several mechanical and optical ISO standards.

Results and discussion

Amount of fines and pulp drainability

The amount of fines and the Schopper-Riegler number of the various pulps are presented in Table 1, where P1 represents the unbeaten pulp, P2 is the pulp P1 after beating and P3 is the pulp P1 beaten after primary fines removal.

As can be seen and as expected, the mass percentages of fines of these pulps are much smaller than those of mechanical pulps (25–50%) /3,11/ and larger than those of softwood kraft pulps /5,18/. The amount of primary fines corresponds to about 8% and the increase of 2% in the beaten pulp results most certainly from the generation of secondary fines which, as mentioned, consist of material detached from the fibres. The mass percentage of fines present in pulp P3 (exclusively secondary fines) was not directly evaluated because no sufficient sample was available. However, regarding the above

Table 1. Amount of fines and Schopper-Riegler number of the unbeaten and beaten pulps.

	Fines content	Schopper Riegler number (SR)		
	(w/w, %)	Whole pulp	Decrilled pulp	
Unbeaten pulp (P1) (0 rev.)	8.1 ±0.3	29	15	
Pulp beaten with primary fines (P2) (2000 rev.)	10.1 ±0.2	42	18	
Pulp beaten after primary fines removal (P3) (2000 rev.)		22		

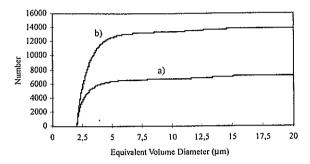


Fig. 1. Cumulative undersize number distributions of fines suspensions, measured with the Coulter Multisizer: a) primary fines; b) primary and secondary fines.

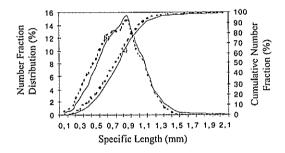


Fig. 2. Decrilled pulp fibre length distributions measured with the Kajaani, before (—, a)) and after beating (---, b)).

results, it is expected to be close to 2%.

It must be stressed that fines are small particles and so their mass percentage may not adequately denote their effect on properties like drainability or paper opacity, for instance. A comparison between the number of fines in pulps P1 and P2 performed with the Coulter Multisizer II for suspensions prepared under identical conditions depicted in Fig. 1, show that the beaten pulp contains a number of fines which is approximately double of that of the unbeaten one. As this increment only corresponds to 2% in mass, it can be anticipated that the fines generated by beating must be smaller in size.

With regard to the SR number, it is evident from Table 1 that this parameter is strongly influenced by the presence of fines. As a matter of fact, when these are removed, the SR number decreases by approximately 50% for both the unbeaten and beaten pulps. This confirms that fines play a major

role in water retention for they fill voids between fibres and they swell better than fibres (namely due to their large specific surface area) /4,5,8/. Furthermore, the SR number of the whole pulp P2 is remarkably larger than that of the whole pulp P1 as a result of the additional presence of the secondary fines and fibrillation. However, if the comparison is made between the decrilled pulps, the increase in the SR number is not substantial. These findings are in agreement with other authors who claim that at least for moderate beating levels, the freeness values are only dependent on the fines content and when fines are eliminated, remain nearly constant during beating /1,5,19/.

An attempt was made to isolate the effect of the secondary fines on the pulp drainability by measuring the SR number of pulp P3. Since pulps P2 and P3 submitted to the same degree of beating present analogous handsheet properties after fines re-

moval (Table 4), it can be concluded that the fibres from these two pulps are equally fibrillated. Hence, the difference between the SR number of the decrilled pulp P2 (18) and that of the whole pulp P3 (22) is exclusively due to the influence of the secondary fines. On the other hand, if the SR number of the whole pulps P2 and P3 are compared, a large difference is found (42 versus 22) which can only be explained by the absence of primary fines in pulp P3. These results suggest that the primary fines fraction retain more water than secondary fines one, most probably due to the distinct quantities, shapes and nature of both kind of fines. Furthermore, the SR number of the whole pulp P2 reveals that the combined impact of primary fines, secondary fines and external fibrillation far exceeds the isolated effect of each one.

Fibre measurements

As for fibre measurements, only decrilled pulps were considered in order to eliminate the impact of fines which greatly affects the length distributions and the average lengths, as proved by the authors in previous studies /15,16/. The length frequency distributions of the decrilled pulps P1 and P2, obtained with the Kajaani and shown in Fig. 2 confirm that during beating, fibres are shortened. However, as the beaten pulp curve is only slightly shifted to the left, it may be concluded that the cutting action is not relevant and occurs mainly at fibre ends.

Table 2 shows the length-weighted averages, L₁ (selected because they correlate better than the other length averages with paper properties /5,13/), and the numerical average fibre widths and curl indexes of the unbeaten and beaten pulps, P1 and P2 respectively. The parameters measured by image analysis for each fibre were the specific length (L) and the specific width (W), defined as the length and the width respectively, of a rectangle whose area and perimeter match those of the fibre image and also the end-to-end fibre distance (L'). The latter provides the calculation of the curl index defined as /20–22/:

$$I_{curl} = L/L' - 1$$
 [1]

This index was used to assess the fibre bending induced by beating which significantly affects the papermaking potential of pulp.

As can be seen from Table 2, the mean fibre lengths given by the Kajaani are validated by those of the microscope since they are practically coincident and as expected, corroborate the above conclusions regarding

fibre shortening. As for fibre width, a modest reduction with beating is found, which is somehow expected due to the tightening of the cell wall structure /23,24/.

Regarding the arithmetic mean curls presented in Table 2, they indicate that beaten fibres exhibit a larger deviation towards the original length directional shape, reflecting a reduction in fibre bending stiffness, probably as a consequence of internal fibrillation and fibre wall deformation. Identical conclusions were withdrawn by using the FQA (Fiber Quality Analyzer) /13/ (unpublished data). This trend is also referred to by Clark /1/ and is in accordance with the results reported elsewhere /20/. On the other hand, preliminary studies carried out with 10% consistency pulps involving increasingly higher beating degrees (up to 6000 revolutions at the PFI mill) also confirm that refining at these conditions is sufficient to induce curls and kinks. Opposite results have been published and explained in terms of the capability of fibres to straighten due to an increase of the swelling pressure at the damaged points /21,22/.

Fines measurements

The discussion of fines dimensions will be mainly based on the equivalent volume diameter distributions, measured with the Coulter-Multisizer II. Fig. 3 presents these curves in number percentages, from which two zones can be distinguished: one between 2.5 and 10 µm and another above 10 µm. (These values may seem quite small at first sight. However, it should be pointed out that for instance, a short fibre with an average length and width of 300 µm and 14 µm respectively, has an equivalent volume diameter of approximately 45 µm, assuming cylindrical shape. Thus, it is reasonable to expect much smaller values for the majority of fines as observed). In addition, average diameters in the range 10-20 µm have already been listed elsewhere /4/.

The high number percentages detected near the left hand side of the distributions suggest the existence of very fine fines (below 2.5 µm). However, these may be regarded as impurities originally present in the unbeaten pulp suspension and not as primary or secondary fines. Additionally, part of these values may be due to excessive electronic noise resulting in countable pulses.

Fig. 3 also shows that the relative amount of particles with equivalent diameters between 10 and 20 µm is significant for the primary fines, decreases with beating and is irrelevant for the secondary fines fraction. These particles – the brick-like parenchyma cells or tube-like structures (mainly

Table 2. Fibre dimensions of the unbeaten and the beaten decrilled pulps given by the Kajaani and by microscopy.

	Unbeaten pulp (P1)			Beaten pulp (P2)		
Instrument	Length, L (µm)	Width (µm)	Curl index	Length, L _; (µm)	Width (µm)	Curl
Kajaani	885			830	(1011)	ındex
Microscope	880	14.0	0.086	825	13.5	0.111

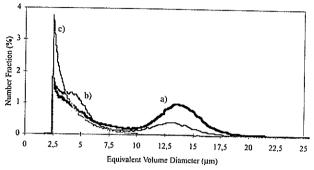


Fig. 3. Number fraction distributions of fines, measured with the Coulter Multisizer: a) primary fines; b) primary and secondary fines; c) secondary fines.

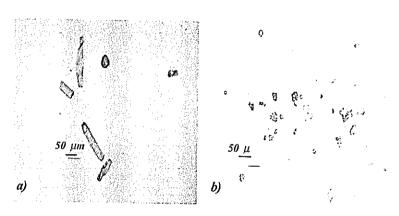


Fig. 4. Microscopic images of unbeaten pulp primary fines (a)) and beaten pulp secondary fines (b)).

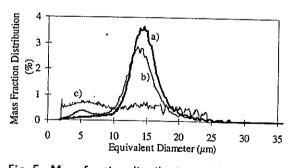


Fig. 5. Mass fraction distributions of fines, measured with the Coulter Multisizer: a) primary fines; b) primary and secondary fines; c) secondary fines.

fibre fragments) – correspond to the largest primary fines depicted in the microscopic images as that of Fig. 4-a). Nonetheless, the primary fines suspensions also contain material below 7.5 µm, as visible in Fig. 4-a).

The subsequent formation of fines with beating increases the amount of material in the range of 2.5–7.5 µm, resulting in the emergence of a peak in this branch of the

distribution curve. Furthermore, from Fig. 3 it can also be concluded that in the secondary fines suspensions a negligible number of particles larger than 10 µm exist, in agreement with the visual observations as that of Fig. 4-b).

In order to minimise the influence of the very fine fines, and since most studies concerning fines involve mass determinations (e.g., Table 1), it was decided to additionally analyse the correspondent volume (or mass) fraction distributions, presented in Fig. 5. This figure clearly shows: a unimodal distribution for the primary fines (centred around 15 µm); two peaks for the mixture of primary and secondary fines; and an unexpected broad distribution for the secondary fines alone. The latter can be due to either primary fines which were not efficiently removed in the original screening, or large fibre wall fragments and fibrils bundles formed during beating or eventually broken and very small fibres which have passed through the last screening. However, it should be emphasised that these large particles although just a few, have a significant effect on the volume percentages, being responsible for the long tail towards the right.

Table 3 summarises the mean diameters of the above distributions, number and volume weighted. In both cases, the secondary fines are much smaller than the primary fines.e 3

Paper properties

As mentioned earlier, the ultimate goal of this work is to study the role of fines on the papermaking potential of pulp. For this, standard tests have been performed on laboratory handsheets prepared with the aforementioned unbeaten and beaten pulps before and after fines removal. The corresponding results are given in Table 4.

The differences found between pulps P1 with and without fines are related to the presence of primary fines while those between the whole pulp P3 and the decrilled one reflect the influence of the secondary fines. In both cases, the presence of fines modestly decreases the light scattering coefficient by improving the bonded area and thus reducing air-solid interfaces, and slightly increases sheet density by promoting the network compactability /5,18/. However, as expected, the increment in this property due to the secondary fines is considerably small, for they correspond to a minimal volume and consequently play a minor role in bulk. Fines also greatly increase the Gurley air resistance, but the influence of the secondary fines is again not so substantial. As for the strength properties, both tensile and burst indices are greatly reduced when primary fines are removed and remain nearly unchanged whether secondary fines are present or not. This means that these fines barely contribute to the improvement of stress distribution and fibre bonding ability. For the unbeaten pulp, fines enlarge the tear index, while for the beaten pulp the tendency is the opposite as

Table 3. Fines dimensions given by the Coulter-Multisizer II.

	Mean diameter (µm)			
	Number weighted	Volume weighted		
	$d_{1,0} = \frac{\sum n_i, d_i}{\sum n_i}$	$d_{4,3} = \frac{\sum (n_i, d_i^3) . d_i}{\sum n_i . d_i^3}$		
Primary Fines	6.1	14.3		
Primary+Secondary Fines	5.0	12.5		
Secondary Fines	3.8	9.6		

Table 4. Paper sheet properties of the unbeaten and the beaten pulps, before and after fines removal.

Paper Properties		Pulp with fines			Pulp without fines		
	Pulp P1	Pulp P2	Pulp P3	Pulp P1	Pulp P2	Pulp P3	
Light scattering (m2/kg)	34.8	25.5	26.5	35.3	28.0	27	
Density (g/cm³)	0.668	0.767	0.748	0.600	0,708	0.714	
Air resistance (s/100 ml)	7.8	54.5	5.7	1.2	2.6	2.4	
Tensile index (N.m/g)	70.9	99.4	82.5	48.0	81.7	79.9	
Burst index (kPa.m2/g)	3.8	6.9	5.0	2.1	5.0	5.1	
Tear index (mN.m²/g)	8.8	9.5	9.9	7.0	10.2	10.2	

also reported elsewhere /5/. This may indicate that due to their size the intrinsic strength of the primary fines and their contribution to fibre bonding must be relevant for the overall tear resistance. On the other hand, the secondary fines interposed between fibres, scarcely reduce the out-of-plane resistance, presumably because being in general smaller, their intrinsic strength must be negligible and so they create fragile points in the tearing pathway.

When fines of pulp P2 are eliminated, much more pronounced variations are found for the above properties than those obtained for pulps P1 and P3, denoting that the combined contribution of primary and secondary fines is not the mere addition of the isolated effects of each one.

Handsheet properties of decrilled pulps P2 and P3 are coincident, indicating that the extent of fibrillation, fibre flexibility and collapsibility and fibre bonding is the same and originates identical three-dimensional structures. These pulps were submitted to the same degree of beating, the unique difference being that primary fines were absent in the beating process of pulp P3. This suggests that this operation is not affected by the fines originally present in the pulp, at least for the above quantities (8% in mass).

Since fibre shortening with beating is found negligible (Table 2), the differences detected between the handsheet properties of decrilled unbeaten pulp P1 and decrilled beaten pulps P2 and P3 are mostly due to fibre cross-sectional changes associated with the external and internal fibrillation. As fibrillation increases the fibre bonded area, it is understandable that better paper tensile, burst and tear resistances are achieved in the sheets made with beaten fibres. The observed increment of density and air resist-

ance as well as the diminution of the light scattering coefficients are also a result of the higher packing tendency of the fibrillated fibres (the more consolidated handsheet structure is related to the increase in fibre flexibility and collapsibility which improve the conformability of fibres in the network /25/).

The alterations of the sheet properties of the whole pulp with beating are fully justified by both the aforementioned basic phenomena – fines formation and fibrillation. Nonetheless, considering the previous discussion regarding the role of secondary fines, it is expected that the differences between the whole pulps P1 and P2 be more affected by fibrillation than by the production of these fines.

Finally, the role of primary fines on the handsheet properties of beaten pulps may be assessed by comparing the whole pulps P2 and P3, since, as discussed above, the characteristics of the fibres themselves seem equal and, assuming that primary fines do not degenerate into secondary fines, they have the same amount of secondary fines. When primary fines are not present (pulp P3), light scattering increases, while density, air resistance, tensile and burst strengths diminish, as also found for the unbeaten pulp P1. Concerning the tear strength, it barely increases confirming the cited effect of the fines taking off on beaten pulps.

Conclusions

E. globulus kraft pulps, cooked in a laboratory MK digester and beaten in a PFI mill at 2000 revolutions, exhibit an amount of fines about 10% of their total weight, the secondary fines, although numerically abundant, corresponding to only 2%.

Size measurements reveal that both primary and secondary fines have a broad distribution of equivalent volume diameters, predominantly smaller than 20 µm. However, the former, besides including very fine fines, comprise a significant amount of fines with diameters between 10 and 20 um which presumably consist of fibre fragments and other cells like those from the parenchyma. On the contrary, the secondary fines despite the presence of some few large particles, are generally very small, apparently flat particles, with equivalent diameters smaller than 7.5 µm. The electrical sensing zone technique proved to be adequate for routine analyses of these particles since it is fast, reproducible, not affected by the transparency of the fines, suitable for low concentrations and operator independent.

The proximity of the fibre lengthweighted averages of the decrilled unbeaten and beaten pulps, as well as the closeness of the respective fibre length distributions, show that for the applied beating level (2000 revolutions), fibre shortening is not relevant and occurs mainly at fibre ends.

With regard to the influence of fines, it is evident that both primary and secondary fines affect pulp drainability, the former being predominant, thus exhibiting a more pronounced swelling effect. The results also suggest that the morphological fibre changes due to moderate beating degrees scarcely affect the SR number. However, the repercussion of these changes and fines formation altogether is important and exceeds the separate influence of each one.

Concerning the role of fines in the handsheet properties, the results show that this is not determinant for light scattering and sheet density, and that primary fines also have a more relevant impact than that of the secondary fines, which only play a minor role on some sheet properties (namely those dependent on the fibre network arrangement in the Z-direction). Nonetheless, once again, the behaviour of the secondary and the primary fines together goes beyond the summation of the influence of each one separately, suggesting that the sheet formation is different whether primary fines alone, secondary fines alone or primary and secondary fines together are present.

Finally, the presence of primary fines during beating (at 10% consistency) seems not to affect the fibrillation process since decrilled handsheet properties are identical regardless whether the pulps are beaten with or without primary fines.

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