MORPHOLOGICAL CHARACTERIZATION OF PULPS TO CONTROL PAPER PROPERTIES

ANA MORAL, M. CONCEPCIÓN MONTE, ELENA CABEZA and ANGELES BLANCO

Chemical Engineering Department, Complutense University of Madrid, Avda. Complutense, s/n. 28040 Madrid, Spain

Received November 8, 2009

Recovered paper quality is a key issue for papermakers that intend to maintain the quality of their final products at a high and constant level, for meeting the consumer demands. However, since paper recovery is increasing, the quality of recovered paper is decreasing and, what is even worse, it varies along the time.
The physical properties of recycled pulp fibres have a strong influence on most of the paper properties and, therefore, they play an important role in the establishment of the optimal papermaking conditions. These properties are directly related to the morphology of fibres and to pulp composition. In recent years, several new fibre and pulp morphological analyzers have been developed and released on the market. The use of on-line optical fibre analyzers allows papermakers to know the variations in pulp quality, enabling them to adjust the process and to maintain constant the quality of the paper produced. However, most of these devices are optimized for virgin fibres, their application for recovered paper being still limited.
The present paper describes the modifications carried out in the programs of a fibre and pulp morphological analyzer (Morfi V7.9.13.E) to optimize its performance for the characterization of recycled pulps. The three programs (VESSELS, FIBRES and SHIVES) that the device includes by default have been considered and validated in a paper mill, producing different grades of newsprint and light-weight coated papers. The results show that, with the modified program, the device appears as a very promising tool to control and improve the final quality of recycled paper.

Keywords: recovered fibres, morphological characterization, process optimization, paper properties, pulp quality

INTRODUCTION

Recovered paper collection is constantly increasing and the limits of paper recycling have been extended to papers of higher quality, although the quality of recovered paper as a raw material is decreasing.[1-3] Therefore, the control of the process to buffer the variability of the raw material is becoming more and more important each day.

The physical properties of recycled fibres have a strong influence on the papermaking potential of the corresponding pulps and on most of the end-use properties of the final paper products.[4-7] For example, fibre length greatly affects the strength of paper. Fibre width and wall thickness, on the other hand, affect the flexibility of the fibres and their tendency to collapse in the paper production process, which also affects the properties of the final paper. The fibres with thin walls collapse more readily and are, therefore, conformable. Conformable fibres bond better in a sheet structure and make denser, stronger and smoother sheets.[8]

Fines contribute to the optical and strength properties, to smoothness and to the structure of the sheet. The fines content is mainly determined by the type of cooking process applied to virgin fibre, by the fibre/filler relationship in recycled fibres and by the refining degree for both of them. In recycled pulp, the measurement of the fines content also accounts for the presence of fillers, pigments and sizing agents, employed in the recovered paper used as raw material. Fine elements can be related to the damages caused to the fibres by the use of chemicals and by the strong mechanical actions (stirring, beating, screening, dispersion, etc.) during the process. This parameter is very important in papermaking, since it limits strongly the drainage rate and the paper bulk properties; also, it is related to the optical properties.[1,5] Therefore, during the papermaking process, fibre length and width may decrease, and a higher amount of fines can be produced from fibre breaking. The association of structures with low length and width involves the creation of a matrix with a relatively low number of hollows, favouring the formation of more homogeneous paper sheets and, at the same time, improving the optical properties of paper, such as brightness and opacity. Depending on the final product, these properties will be different. For example, a light-weight coated (LWC) paper will require higher brightness and opacity than a newsprint paper (NP).

Another influent parameter is the amount of vessels present in the fibres. They occur as channels that carry sap from roots to leaves, mainly present in deciduous trees reaching 300 µm in diameter. Their properties impede their easy separation and make their size reduction difficult. It has been demonstrated that there is a relationship between the final properties of the paper sheet and the amount of vessels in the fibres. Vessels can cause problems in the final uses of the paper, *e.g.* during printing, the vessels attach to the surface of the rollers in the printing machine, producing a series of points on the paper sheet, not covered by ink, as shown in Figure 1. Traditionally, in the paper mills, the characterization of vessels has been done by tedious techniques, such as the analysis by optical microscopy.

To maintain the quality of the final product and to ensure the competitiveness of the mill, it is important to understand the correlations between fibre, pulp and paper properties, which implies the need to characterize fibre morphology and pulp composition in an efficient and reliable manner.

The on-line morphological character-rization of pulp suspensions through image analysis systems is relatively new. In recent years, several new fibre length analyzers have been developed and marketed. The new instruments provide faster measurements and the possibility of both laboratory and on-line analysis. All devices provide similar values of length, coarseness, width and fines content measurements for a given sample. That is a guarantee for their use in the morphological characterization of fibres. In the present investigation, MorFi v7.9.13.E (Techpap, France) has been selected for the study of recycled pulps.

Although this device can be applied directly to recycled pulps with its default programs (FIBRES, SHIVES or VESSELS) for the full characterization of the pulp suspension, it is necessary to adapt the program, by integrating all available information into one of them.

The objective of this work is to define which of these three base programs is the best for the morphological analysis of recovered pulps and which parameters should be modified based on the size criterion, to improve the results obtained when characterizing recycling pulps.

This paper describes the development of the program adapted to characterize recycled pulps sampled from any point in the paper mill. Furthermore, the possibility of using this methodology to replace the detection of vessels by optical microscopy has been also investigated.

EXPERIMENTAL

Three different kinds of samples were analyzed for selecting the right measurement program:

1. To select the base program:

- Pulp samples were taken from a Spanish paper mill producing recycled graphic paper from 100% recovered paper. The samples were taken from different paper machines (PM1 and PM2) and from two sampling points (p1 and p2). PM1 produces different grades of paper (newsprint, high-quality newsprint and light-weight coated), while PM2 produces only newsprint paper. The two sampling points were the headbox (p1) and the storage tank from the deinking plant (p2). Sampling was carried out for each point in different weeks, during two months, selecting the days randomly, to assess whether each base programs is able to carry out the measurements in a reliable manner when the variability of the samples is significant.
- Three kinds of recycled papers produced in PM1 and PM2 were selected: newsprint (PM1-NP and PM2-NP), high-quality newsprint (PM1-HQNP) and light-weight coated (PM1-LWC). The paper sheets were disintegrated and the pulp obtained was characterized morphologically.

In all cases, pulp concentration was set to 0.1 g/600 mL, a total of 10 samples being taken for each analysis.

2. To select the measurement program:

• Pulp mixtures were sampled from recovered and virgin fibres (*Eucalyptus Globulus*, TCF pulp – “totally chlorine free”). The recovered paper pulp was sampled directly from the headbox of the paper machine, obtained from 100% recovered paper. Also, the concentration of pulp mixtures for the measurements was of 0.1 g dry fibres/600 mL. The trials were carried out on the suspensions obtained from mixtures of virgin fibre pulp and recovered paper pulp, in different proportions (Table 1).

In this case, the pulps were also analyzed by visual counting of the vessels on the images taken by the Morfi device.

The fibre morphology analyzer Morfi V7.9.13.E has been used for the characterization of all kinds of samples: pulps, paper sheets and mixtures. The equipment integrates a digital camera and a software package for image analysis, to automatically measure fibres in suspension. This equipment is designed to analyze very diluted pulp suspensions and to measure different parameters by specifying fibre dimensions (length and width), the content of microfibrils attached to the fibres, fine elements, vessels, etc.[9,10]

The equipment includes several components. The optical part contains a measuring cell, a camera, a set of lens and a lamp. Theoretical optical resolution is defined at 10 μm, the practical one reaching 4 μm, as due to the possibility of carrying out specific image analysis treatments in grey scale. Another section of the equipment encompasses a hydraulic system (Fig. 2), consisting of a tank, a pump and electro-valves controlled by an electric system.

Samples may be taken either on- or off-line, from an automatic carrousel containing 9 sample cells controlled by the PC built into the equipment.

The software does not have a general program to detect the parameters of fibres, fines, shives and vessels, altogether. The equipment has three kinds of programs (BASE PROGRAMS): FIBRES, SHIVES and VESSELS – FIBRES does not measure vessels, VESSELS does not measure shives (even if the size limits of vessels can be modified), while SHIVES measures all parameters (without allowing however the modification of the size limits for vessels).

Within each BASE PROGRAM, the intervals of length and width may be established, to define each of the detected parameters. These intervals should follow some directives, according to the general definitions of fibres, fines, vessels and shives. Therefore, the variations consist in adjusting the interval defined for each species (fibres, vessels, fines and shives) and each sample type, by narrowing it down to the actual dimension of the respective item, thus increasing the accuracy of the measurement. The programming model follows the scheme shown in Figure 3.

As shown graphically, for each program it is possible to define a number of families, up to a total of N. For each of them, the intervals are defined through manual programming, by adjusting them to the study case – depending on pulp properties.

These base programs consider:

• ***Fibres***: elements with the length (L) varying between 100 and 10000 μm and width (W) between 5 and 75 μm. The rigidity and mechanical resistance of the fibre will vary according to the process it undergoes. The ratio L/W is defined as the ratio between fibre length and fibre diameter. Larger ratios will give pulps with higher mechanical properties, such as strength and toughness.[11]

• ***Fines***: elements with the length (L) under 100 μm and width (W) under 5 μm.

• ***Vessels***: elements defined by the relationship L/W as amorphous, non-fibrous, because their length (L) in recycled fibres is around 100 or 200 μm, while their width (W) is higher than that of the fines. The L and W parameters of the vessels can be modified only in the VESSELS base program.

• ***Shives***: elements with the length (L) varying between 100 and 10000 μm and width (W) higher than 75 μm, occurring as bits of fibres not well disintegrated during the treatment of wood and therefore appearing as loads and blobs. Shives are mainly present in the virgin and not in the recycled fibre.

The morphological parameters for each kind of element are shown in Table 2.

In all cases (pulps, paper sheets and mixtures), the analysis was made in triplicate.

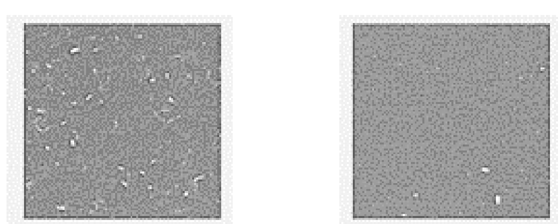

Figure 1: Printing defects generated by the presence of vessels (white points)

Table 1
Mixtures of pulps used

Content in recycled pulp (%)	Content in virgin pulp (%)
100	0
80	20
60	40
40	60
20	80
0	100

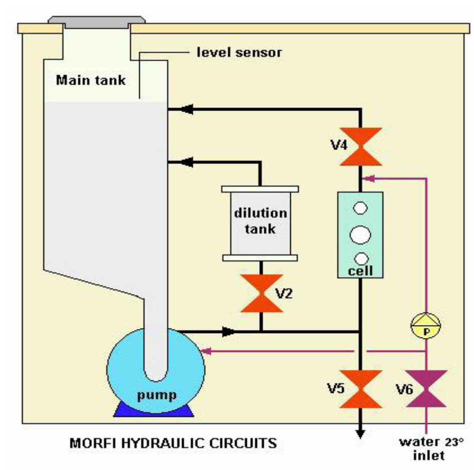

Figure 2: MorFi hydraulic circuits

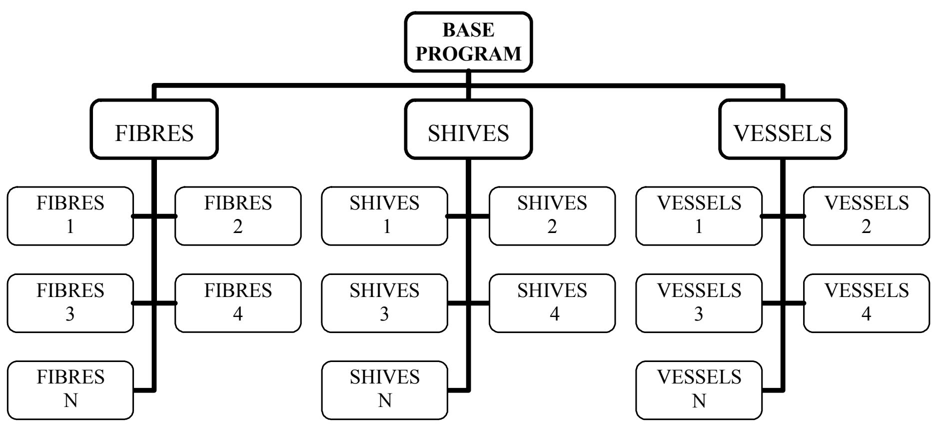

Figure 3: Programming model

Table 2
Morphological parameters for each kind of elements measured by MorFi

FIBRES	FINES
Number of fibres/gram	Number of fines/gram
Average length	Average length
Length distribution	Length distribution
Average width	Average surface

Width distribution	Surface distribution
Average curling	VESSELS
Curling distribution	Number of vessels/gram
Average curling grade	Average length
Percentage of kinked fibres	Average width
Kinked fibres distribution	SHIVES
Average kinked angle	Number of shives/gram
Average number of folds	Average length and width

RESULTS AND DISCUSSION

Selection of base program

To select the best base program, the same limit values for length, width and L/W ratio (Table 3) were used to characterize the different samples.

Tables 4 and 5 show the median results of the morphological characterization of 10 samples. The error of the measurements was below 5% for pulps, and below 3% for paper sheets, for all studied parameters.

For all cases, the results of the morphological characterization show that the number of shives was irrelevant. However, there was an appreciable quantity of vessels in the samples, which should be always taken into account (Table 4). In the case of paper sheets, the amount of vessels is practically zero (Table 5), which was expected, since the study was carried out on samples of a final product without defects. Figure 4 shows pulps obtained from mill pulps and from paper sheets. One may also observe that, in the case of paper sheets, the number of vessels is low, due to the high efficiency of the process in removing or degrading them.

As already mentioned, the FIBRES program does not measure the vessels; when using this program, vessels are detected as fines, which leads to an erroneous pulp characterization. The SHIVES program measures all parameters but, since no shives were present in the samples to be measured and since the limits of vessels could not be defined with this program, it was also ruled out. The VESSELS program was finally selected to characterize the pulp, given that it detects vessels with the highest precision, avoiding the possible alterations that might be induced by the presence of such elements during measurements.

Table 3
Limits of fibres set in MorFi for the three base programs

L_{FIBRES} min (μm)	L_{FIBRES} max (μm)	W_{FIBRES} min (μm)	W_{FIBRES} max (μm)	$(L/W)_{FIBRES}$
100	10000	5	75	1.5

Table 4
Results of different pulps obtained by each base program

			PM1 p1	PM1 p2	PM2 p1	PM2 p2
FIBRES	Fibres	l_l (μm)	996	954	963	925
	Fines	f_l (number)	71021	73994	76246	77962
	Vessels	v_l (μm)	0	0	0	0
	Shives	s_l (μm)	0	0	0	0
SHIVES	Fibres	l_l (μm)	999	943	958	911
	Fines	f_l (number)	67273	69569	64461	67350
	Vessels	v_l (μm)	773	752	761	749
	Shives	s_l (μm)	0	0	0	0
VESSELS	Fibres	l_l (μm)	991	960	962	925
	Fines	f_l (number)	68692	70144	66563	70291
	Vessels	v_l (μm)	775	711	752	704
	Shives	s_l (μm)	0	0	0	0

Table 5
Results of different paper sheets obtained by each base program

			PM1-NP	PM1-HQNP	PM1-LWC	PM2-NP
FIBRES	Fibres	l_l (μm)	912	894	886	919
	Fines	f_l (number)	79563	78521	80692	78947
	Vessels	v_l (μm)	0	0	0	0
	Shives	s_l (μm)	0	0	0	0
SHIVES	Fibres	l_l (μm)	906	901	894	913
	Fines	f_l (number)	79273	77569	81461	79350
	Vessels	v_l (μm)	10	4	7	16
	Shives	s_l (μm)	0	0	0	0
VESSELS	Fibres	l_l (μm)	991	960	962	926
	Fines	f_l (number)	78692	79644	77563	79551
	Vessels	v_l (μm)	11	9	10	15
	Shives	s_l (μm)	0	0	0	0

Table 6
Measurement programs and parameters of vessels

Measurement programs	Length max. (μm)	Width max. (μm)	L/W minimum
Vessels 1	100	75	1.2
Vessels 2	200	140	1.1
Vessels 3	100	75	1.5
Vessels 4	200	140	1.5
Vessels 5	200	140	1.2
Vessels 6	100	75	1.1

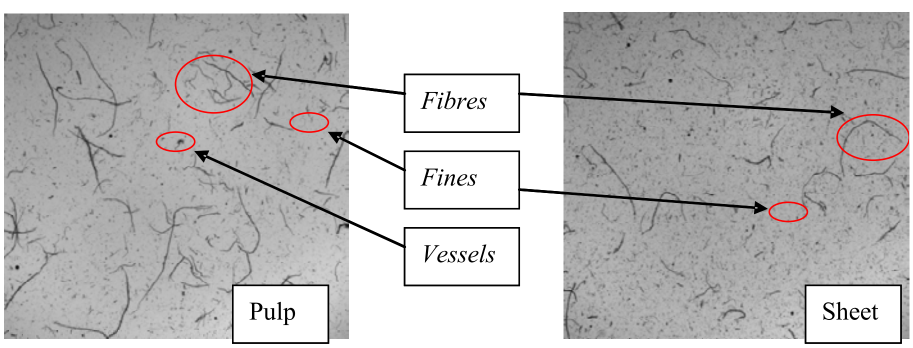

Figure 4: Images taken by MorFi from pulp and paper sheets of recovered paper

Selection of measurement program

To carry out a reliable characterization of recycled pulp fibres, it was also necessary to develop a measurement program that would take into account the ratio between fibre length and width. The next step in the study was to develop a specific analysis method to characterize pulps, including the vessels content.

Starting from previous results, a new program was developed from the base VESSELS, to check the characterization results obtained by Morfi. An optimization of the parameters that define the measuring program is complex in the case of a recovered paper sample, since the amount of vessels is too low for optical detection, presenting very slight variations. To create categories with a different number of vessels, a study with mixtures of recovered paper and virgin fibre was undertaken. Six programs were developed within the base VESSELS program. The limit values for the tested vessels are summarized in Table 6.

Ten experiments (e1 to e10) were carried out for each pulp mixture, by visual counting, to evaluate reproducibility and to analyze the increase in the number of vessels with the percentage of virgin fibres (Fig. 5). The same experiments were analyzed by the six programs defined (Fig. 6). The results were compared with those obtained by visual counting (Table 7), to select the best program.

The tendency observed for the number of vessels obtained by visual counting (Fig. 5) is similar to the one obtained with the image analysis program (Fig. 6) *i.e.*, as expected, there was an increase in the number of vessels with the percentage of virgin pulp in the mixture.

Table 8 shows the medium error of all experiments analyzed by the equipment *versus* the experimental results.

The *Vessels 3* program was selected for vessel characterization and for this kind of pulp, since it is the one giving the lowest error, around 5%. The summary of the limit values of elements for this program is shown in Table 8.

Table 7
Error of the program *versus* visual counting

Recycled pulp (%)	Vessels 1	Vessels 2	Vessels 3	Vessels 4	Vessels 5	Vessels 6
0	-239%	10%	5%	36%	-128%	17%
20	-249%	-13%	-8%	35%	-128%	17%
40	-246%	-24%	6%	37%	-137%	18%
60	-266%	-11%	-5%	36%	-146%	17%
80	-280%	18%	-4%	36%	-159%	15%
100	-272%	12%	-3%	35%	-160%	12%

Table 8
Limit values of different elements measured by *Vessels 3* program

	Length (µm)	Width (µm)	L/W
Fibres	100 < x < 10000	5 < x < 75	1.5
Fines	≤ 100	≤ 5	1.5
Vessels	0< x < 100	> 75	1.5

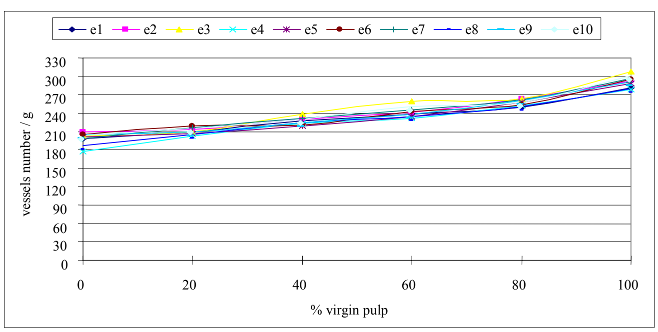

Figure 5: Number of vessels obtained by visual counting

CONCLUSIONS

The fibre morphology analyzer Morfi V7.9.13.E has been successfully adapted for the morphological characterization of different kinds of recycled pulps, by using the same program for pulp samples taken from different points of the paper mill, and for the analysis of the final paper product.

The base program selected was VESSELS, because it avoids counting vessels as fines. To optimize the measurements, the following parameters should be considered: fibre length values between 100 and 10000 µm, and width between 5 and 75 µm; fines length below 100 µm and width below 5 µm; vessel length between 0 and 100 µm, and width higher than 75 µm and, in all cases, an L/W ratio higher than 1.5.

This device with the adapted program is a promising tool for on-line pulp analysis, affording results that can be subsequently used to quantify vessels and to predict and control the final paper properties.

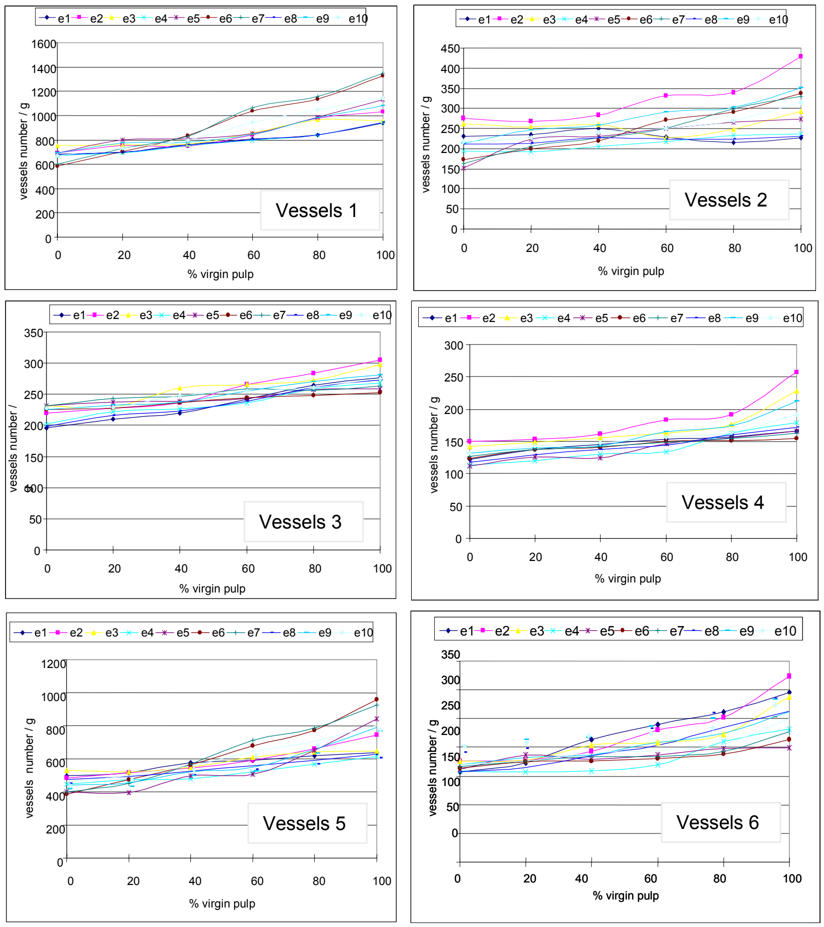

Figure 6: Number of vessels obtained by different measurement programs

ACKNOWLEDGEMENTS: The authors wish to acknowledge the financial support of the Community of Madrid to the Project PROLIPAPEL – CM (S-0505/AMB/0100), of the Ministry of Science and Innovation of Spain to the Project CTM2007-66793-C03-03, and of the European Union to the Project MODELPACK-CE (516322).

REFERENCES

[1] M. C. Silva, O. R. Lopez, J. L. Colodette, A. O. Porto, J. Rieumont, D. Chaussy, M. N. Belgacem and G. G. Silva, *Ind*. *Crop*. *Prod*., **27**, 288 (2008).
[2] M. Wollerdorfer and H. Bader, *Ind*. *Crop*. *Prod*., **8**, 105 (1998).
[3] A. Gandini and M. N. Belgacem, *J*. *Polym*. *Environ*., **10**, 3 (2002).
[4] R. S. Seth, *Procs*. *MRS Symposium*, Materials Research Society, Pittsburgh, PA, 2000, vol. 197, pp. 143-161.
[5] L. Paavilainen, *Pap*. *Puu*, **75**, 5 (1993).
[6] R. P. Kibblewhite, *Ninth Fundamental Research Symposium ‘Fundamentals of Papermaking’*, Cambridge, UK, 1989, pp. 121-152.
[7] D. C. McIntosh, *Tappi J*., **50**, 10 (1967).
[8] R. P. Kibblewhite, R. Evans and M. J. C. Riddell, in *Preprints*, *International Paper Physics Conference*, CPPA, Montreal, Canada, 1995, pp. P47-P48.
[9] P. Mutjé, M. A. Pèlach, J. C. García, S. Presta, F. Vilaseca and L. Jimenez, *Process Biochem*., **41**, 1602 (2006).
[10] R. Passas, C. Voillot, G. Tarrajat, G. Caucal, B. Khelifi and G. E. P. Tourtollet, *Récents Progrès en Génie des Procédés*, **15**, 78 (2001).
[11] B. Lauke and S. Fu, *Compos*. *Sci*. *Technol*., **59**, 5 (1999).