MULTIVARIATE STATISTICAL EVALUATION OF PHYSICAL PROPERTIES OF PULPS REFINED IN A PFI MILL

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ABSTRACT

Aim of the present work is to evaluate the physical properties of cellulosic pulps refined in a PFI mill through analysis of interlaboratorial data. These data were collected from physical tests on laboratory handsheets prepared with refined pulps supplied by 10 different laboratories. Although a high dispersion in the univariate graphics due to the singular calibration of each of the PFI mills, the several multivariate PCA analysis for 20, 30, 40, 50 °SR degrees (Schopper-Riegler) clearly show the variations in thickness, tensile index, tear strength, burst strength, air permeance associated with the different refining level. Additional properties included in the experiments are elongation and opacity. This is the first work, at least for eucalyptus bleached fiber, analyzing 10 different PFI mills, and it is a companion paper for a second one which compares the PFI mill action with an industrial disk refiner for same class of fibers and same °SR, and also for a third work analyzing the collaborative testing laboratory control for PFI.

INTRODUCTION

Multivariate statistical analysis has an important role in data analysis. Multivariate data consist of several different attributes or variables that are assigned to every observation. If there are p variables in a given data set, each variable represents a different dimension in a p-dimensional space. It is difficult to visualize a p-dimensional space; therefore, the goal of multivariate statistical analysis - especially the principal component analysis (PCA) - is to reduce this dimension by grouping similar observations within two or three dimensions. The principal component analysis seeks to reduce this size by extracting the smallest number of components responsible for most part of the variation in the original multivariate data.

In PCA the uncorrelated principal components are obtained by linear transformation of the original variables, so that the first principal component contains most of the variation of the original data. The other main components correspond to minor variations, and are numbered in descending order, i.e., the first principal component corresponds to the greater variation, and the last to the smallest. To reproduce the total variability of the original p variables system, all principal components are needed.

However, if, for instance, the first two components account for more than 80% of the variability and considering that it adequately explains the variance of the system, then the dimension reduction has already been reached and there is no need to proceed in analyzing the others components.

Since the first principal component accounts for the covariance of all attributes, i.e., the first component is already a linear combination of the original variables, the principal component can be a better estimation than a simple weighted average of the original data. Therefore, the PCA can be useful when there is a high degree of correlation between the various attributes.

In this work, data of pulp testing will be introduced. In these tests, two samples of bleached eucalyptus pulp - named a and b - were sent to a number of laboratories. These laboratories carried out the PFI mill refining procedure, determined the drainage resistance and produced laboratory handmade sheets. These sheets were then analyzed by a single manager laboratory, where the physical tests were performed.

The PFI mill

The evaluation of the refining process in a laboratory is considered, by convention, done by refiners whose specific characteristics are well established and tested in their

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respective laboratory. While many laboratories can maintain the reproducibility of their refiners at a satisfactory level, it is a general experience of being difficult to achieve consensus among different laboratories.

Major and Lawford (1962) report that in 1947 Erik Stephansen - who worked for Papirindustriens ForskningInstitutt from Oslo -, in an attempt to overcome some disadvantages in the use of refiners developed a refining device that became known as the PFI mill. Thanks to Stephansen (1948), a complete description of this refiner and the study of some variables already appeared in the literature in 1948.

The PFI mill basically consists of a rotor, a refining housing with lid and a device to apply pressure during the refining. The rotor and the housing rotate on a vertical axis. The rotor has 33 bars, and each bar is 50 mm long and 5 mm wide. The bars are arranged radially, parallel to the axis of the rotor. The rotor diameter is 200 mm, measured through the bars, and the depth of the cavities between bars is 30 mm. The rotor is driven by a motor of approximately 1 kW and the rotational frequency is (24.3 ± 0.5) s^{-1}, when no pressure is applied. The number of rotor revolutions is indicated by a counter. The refining housing, with an internal diameter of 250 mm, is powered by a 400 W motor. The speed of the housing is to be adjusted so that a difference in peripheral speed (6.0 ± 0.2) m/s between the refining elements is obtained when under zero charge and a rotor rotational frequency of (24.3 ± 0.5) s^{-1}.

The refining pressure is achieved through load applied by a lever, which presses the rotor against the housing wall. The refiner has a screw, with which the gap between rotor and housing is adjusted when the bars have been sharpened or conditioned. The refining elements are manufactured in stainless steel.

The Physical and Chemical Standards Committee (1971) presents the following diagrams for the PFI mill:
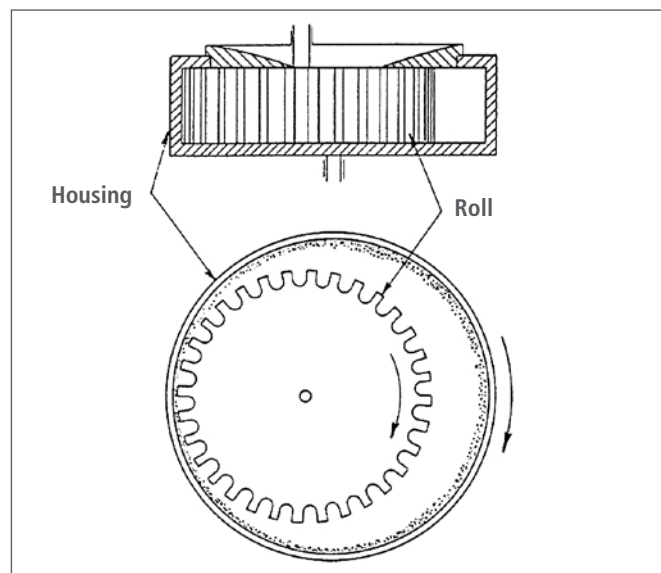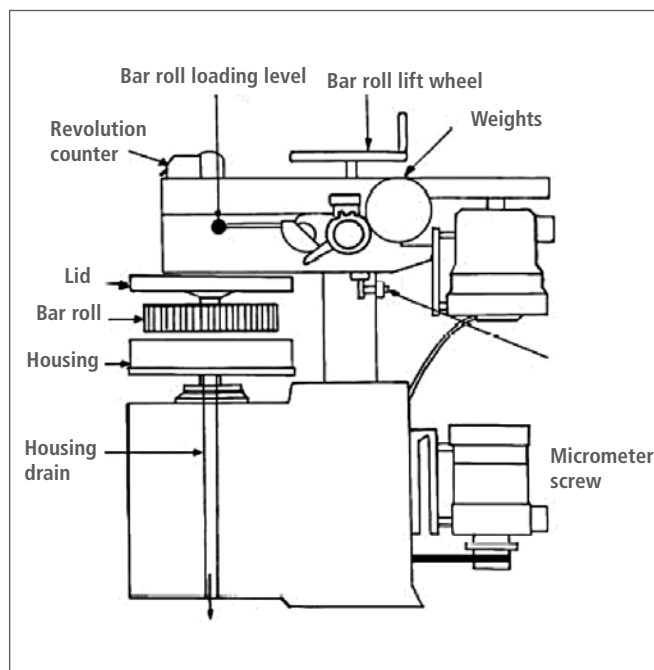

Figure 1. Main element of a PFI mill

Figure 2. Diagram of the PFI mill cross-section

Figure 3. A PFI mill

In Brazil, the procedure for the PFI mill laboratory pulp refining is described in the ABNT NBR 14345 standard. For each refining run in the PFI mill the procedure makes use of (30.0 ± 0.5) g of oven-dried pulp at 10% consistency.

The PFI mill is the most widely used laboratory scale refiner. Many are the studies based on this type of refiner. Examples are: Leatham and Myers (1990) and Formento *et al.* (2001).

At first, the development of the PFI mill was considered a great improvement over the previous existing laboratory scale refiners, especially when compared to the Valley beater. The Valley beater is a reproduction of the "Hollander" beater, both used in former times, but with a very important contribution. It consists of a recirculating open vessel with a horizontally fixed rotor separated from the stator. Details and pictures can be seen in Ekstam (1966).

Ekstam (1966) states that there were many criticisms on the Valley beater. One is that results obtained with the Valley do not correspond to results obtained in an industrial refiner and, therefore, it would not be able to classify fibers quality in the same sort as being done by an industry refiner. Another criticism is that the Valley beater does not maintain a constant condition with continued use and, consequently, does not produce reproducible results. Also according to Ekstam (1966), if an effort is made to achieve a given characteristic on the paper to be produced and refining in laboratory seen as a method for gathering information on industrial scale refining, then it would be necessary to use special refiners that can achieve the desired characteristics. However, to test the strength of a pulp and for the continuous production quality control, the simple and easy methods are of real value.

Despite these criticisms on the Valley beater, Ekstam (1966) states that that beater – properly conditioned and regularly checked –, can provide reproducible results over several years. Similarly, many refiners can be adjusted to provide the same values.

Keays, McDowell and Hatton (1977) assert that some of the advantages of the PFI mill are: the smaller amount of sample required (30 g oven-dry to 450 g oven-dry for the Valley beater)

and shorter refining time necessary to achieve the same drainability.

Hughes (1970) compares the performances of the PFI mill and the Valley beater. According to him, the refiners have shown to produce similar effects on the pulp and the results did not show statistically significant differences between the refiners. However, due to the advantages of the PFI mill already mentioned, between the PFI mill and the Valley beater, the PFI mill is preferred.

The present work is the first one - at least for eucalyptus bleached fiber – to analyze 10 different PFI mills, and is also a companion paper to a second work, where the PFI mill action is compared to that of an industrial disk refiner for same class of fibers and same °SR, published in Yasumura *et al.* (2008), and also companion to a third work analyzing the collaborative PFI laboratory testing control in Yasumura (2004).

Handsheets tests after its pulp refining in a PFI mill

For testing handsheets formed after the PFI mill refining, pulp samples were sent to ten laboratories that carried out the refining and the handsheets manufacturing in their own equipment. Two samples were sent - sample a and sample b - to each of the participating laboratory. Without prior knowledge of the participants, samples a and b are from the same production batch and, therefore, should offer the same physical and chemical characteristics. The parameter for controlling the refining was the Schopper-Riegler drainability, which provides values in °SR.

Each laboratory carried out the refining with aim to four distinct refining points: point 1: around 30 °SR, point 2: around 40 °SR and point 3: around 50 °SR. Handsheets of each of these points, and also of point 0 (without refining), were sent to a single laboratory, where the proposed physical tests were performed. **Table 1** shows the chosen tests for analysis.

Table 1. Tests performed on handsheets made with pulp refined in PFI mill

Properties	Standard	Results reported as	Unit
Thickness	ABNT NM-ISO 534:2000	Thickness	mm
Tearing resistance	ABNT NBR NM-ISO 1974:2001	Tear index	mN.m^2/g
Tensile Strength	ABNT NBR NM-ISO 1924-1:2001	Tensile index	Nm/g
Elongation	ABNT NBR NM-ISO 1924-1:2001	Elongation	%
Air permeance, Gurley	ABNT NBR NM-ISO 5636:2001	Air permeance, Gurley	s/100mL
Opacity	ABNT NBR NM-ISO 2471:2001	Opacity	%

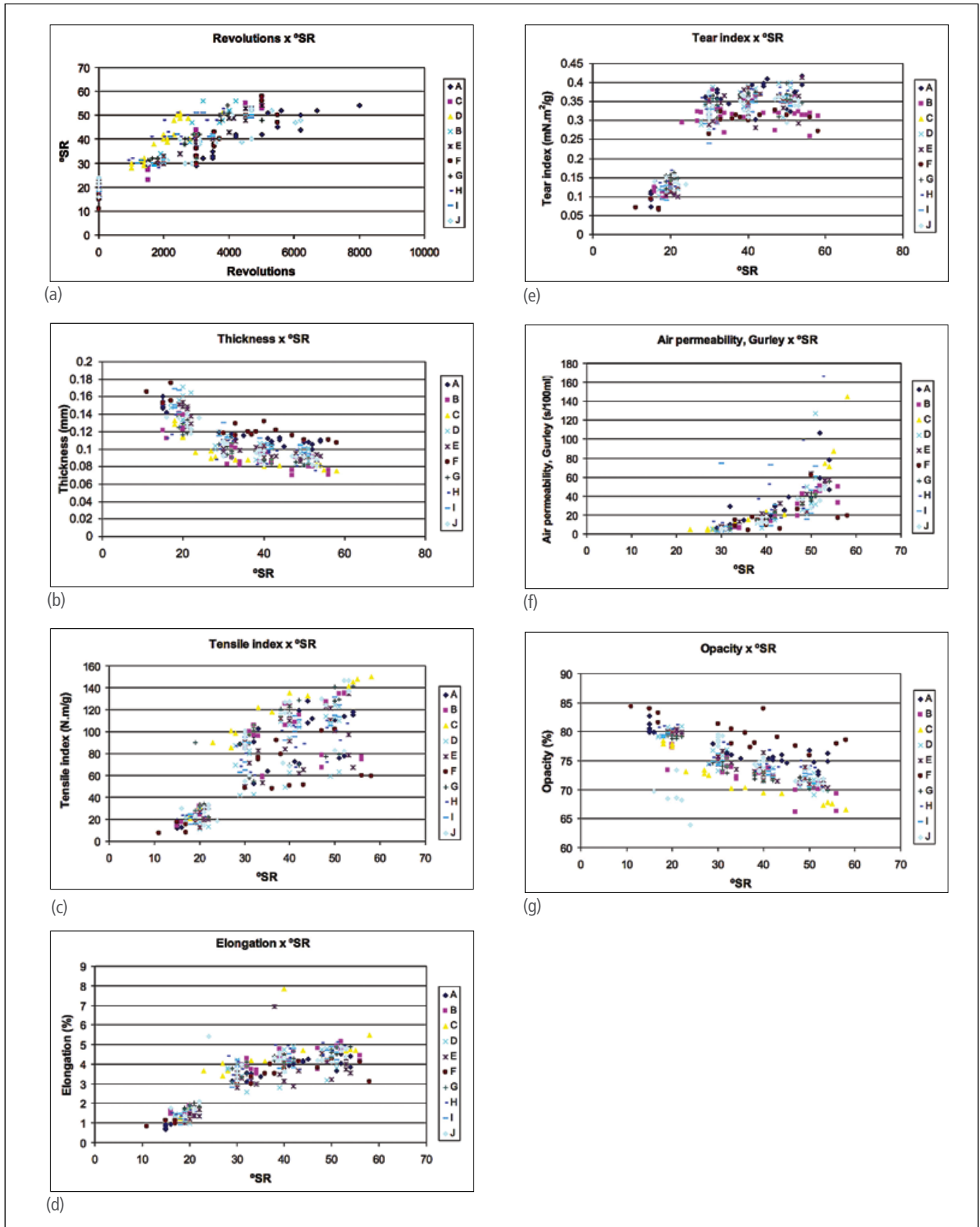Every laboratory also reported the number of revolutions and degree of refining related to each of the points.

(a)

(b)

(c)

(d)

(e)

(f)

(g)

Figure 4. Graphs illustrating variations in results of the PFI mills of the 10 different laboratories (letters indicate the laboratories codes)
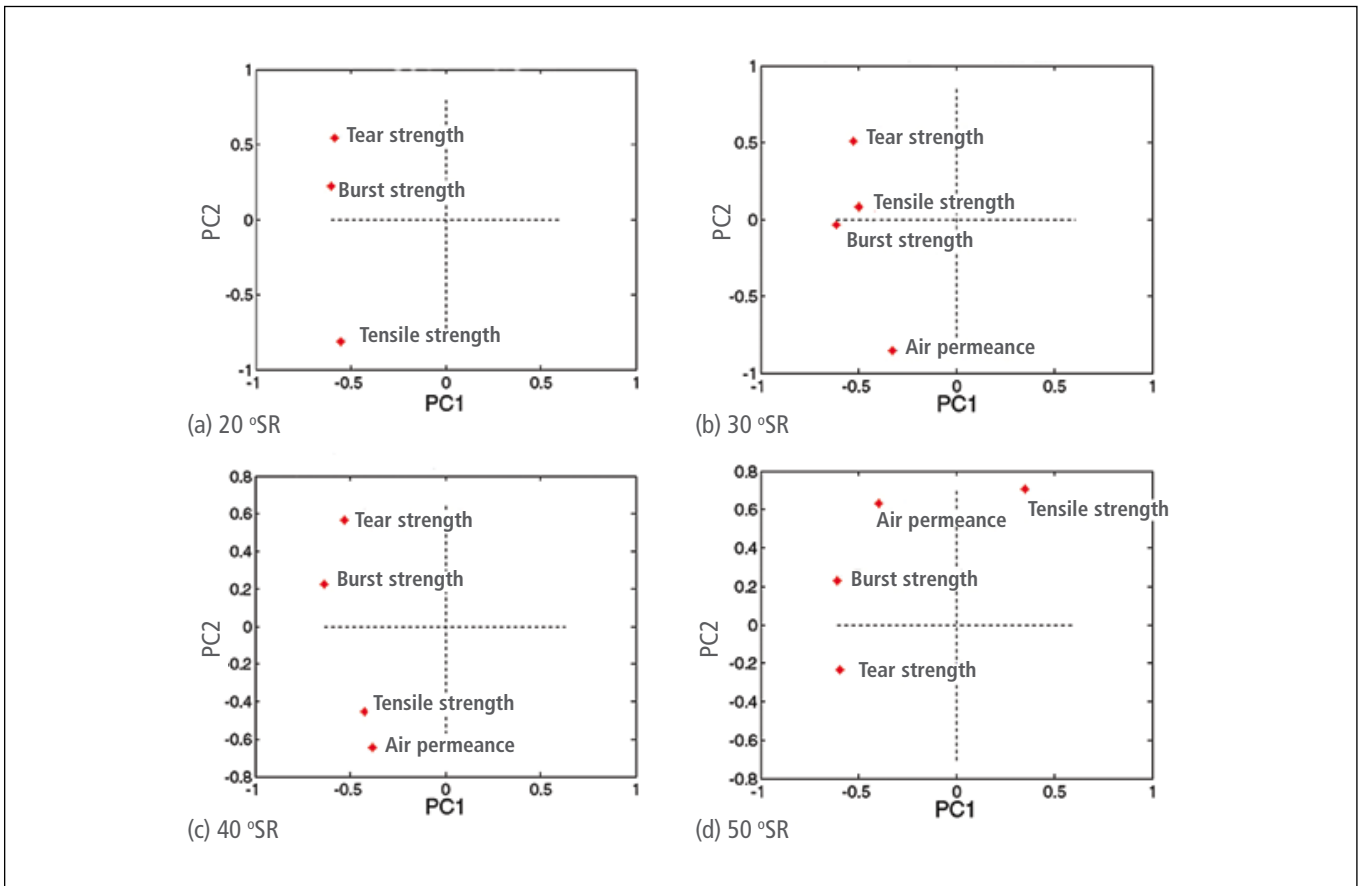
Figure 5. Loading plots for each °SR group

Multivariate analysis for extraction of information using principal components is not that simple when considering the refining in a PFI mill. To investigate this complexity, the interlaboratorial data were analyzed. For this analysis, data were grouped into four groups. For each of the set of points, named groups 0, 1, 2 and 3, the PCA analysis was performed. Group 0 denotes the set of values around 20 °SR, group 1 indicates 30 °SR, group 2 indicates 40 °SR and, finally, group 3 indicates 50 °SR.

The graphs show the relationship of each physical property with the principal components (in this case, the first and second components). Thus, it is expected that properties with similar behaviors are to be placed in the same quadrant.

For group 0 there are no values for the Gurley air permeance, since for unrefined pulp the equipment scale is not sensible enough to allow any air resistance measurement. The treatment for three variables: tear, tensile and burst strengths can be seen in **Figure 5**.

Figure 5a shows the loadings, i.e., the positions of tear, tensile and burst strength variables. Here, these properties remain on the same side of the first component axis, but for the second component tensile and burst are on opposite sides.

In Figure 5b it is to note that with the 30 °SR refining, tensile and bursting strength are close, and apart from tear strength, when compared on the axis of the second component.

If Figure 5b is compared with Figure 5c, tensile strength is still closer to the Gurley air permeance when compared to the burst index.

In Figure 5d the tensile strength shifts to the opposite side of the axis for the first principal component. There is not an immediate explanation, but it is to remember that 50 °SR is an extreme refining condition for hardwood pulp.

RESULTS AND DISCUSSION

To understand the behavior of physical properties in relation to principal components, the refining curves must be analyzed. This analysis is done with **Figure 4**.

In Figure 4a, as expected, as the number of revolutions increases, the higher are the values of the achieved drainability (°SR). However, it is to note that there is a great variation in number of revolutions required to achieve a given °SR for each laboratory. This variation tends to increase with the increasing of the °SR. This may indicate that the PFI mills of the different laboratories have different characteristics, calibration or bars sharpening.

Formento *et al.* (2003) show, with the obtained results of drainability and refining time, that the drainability is strongly influenced by the generation of fines and, to a lesser extent, by the flexibility and external fibrillation of the fiber.

At this point it should be noted that in this study, as per instructions sent to the participating laboratories on the performing of the refining, no specific guidance was proposed about the use of some standard water. Differences between °SR degrees may arise from

differences in the amount of ions and fines generated whereas the refining was performed with deionized or, otherwise, with tap water.

It should also be noticed that the observed variability can come not only from the determination of the °SR, but also from the handsheet formation and from the determination of the physical property as such.

For the thickness (Figure 4b), despite the scattering points on the graph, four distinct groups of points can be identified, around 20 °SR, 30 °SR, 40 °SR and 50 °SR. The general trend of the curve follows the expected evolution of thickness along with the increasing degree of refining. The PFI mill refining causes changes in fibers structure that leads to its better compliance because of the fibers internal fibrillation and collapse, therefore reducing thickness.

The increase in tensile index shows that there is a higher degree of connection between the fibers, either by external or internal fibrillation.

The tensile index graph (Figure 4c) is the one with the greatest results variability. This variability may arise from the handsheet production or in the tensile strength determination itself. The others properties do not show such a large points scattering, which suggests that the variability obtained comes from the test itself. Possible causes can be: changes in procedures between trials or the equipment in use. The elongation (Figure 4d) follows the same tensile index trend, increasing with the increasing of the refining level.

The tear index (Figure 4e) undergoes a large increase in the first point of refining, but then stabilizes. If refining time were increased, the tear index curve would probably suffer a fall due to damage to the fiber structure, which turns out very weak.

The Gurley air permeance (Figure 4f) tends to increase along with the refining degree due to fines formation and better conformity between fibers, reducing spaces in between and increasing the resistance to the air flow. The largest source of variability for the permeance is the handsheet production. Depending on this procedure, the sheet may present itself more or less "closed".

Some laboratories have air permeance values well above others. These higher values are supposed to originate from the handsheet formation, where the produced sheet may have a higher degree of the surface "closing", making it smoother and brighter, and less permeable to air.

According to Scott (1995), the refining has two distinct effects on the opacity. Refining increases the total surface area of the fiber where the light scattering occurs, and because of this the opacity should increase. On the other hand, refining increases the contact area between fibers increasing the bonding area, and this is cause of opacity decrease. For most types of pulps reducing light scattering by increasing bonding area between fibers has greater influence and, therefore, opacity should decrease for most of the pulps.

Figure 4 clearly show the influence of each laboratory handsheet. The thickness values are spreading on a greater range of values. The method the handsheets are produced is of great importance in physical properties development. This graph also allows observing how difficult analyzing data of different laboratories is. Factors such

as the procedure used, the analyst's skills, environmental conditions, equipment, etc., must be carefully weighted when analyzing physical property of pulp handsheets submitted to the refining process and obtained by many different laboratories. The same considerations must be made when trying to compare physical property data originated by different laboratories in different times and conditions.

Formento *et al*. (2003) show results of various properties of paper made with long fiber kraft pulp. Among the results, the tensile index *versus* density graph shows that as the refining progresses a linear relationship is set up between the two properties. This is to indicate that tensile strength is a direct function of the fiber flexibility, which, in turn, is linearly related to the internal fibrillation. The fines contribution to increase bonding between fibers and paper density can also be observed.

As in Retulainen (1996b), Formento *et al*. (2003) observed an increasing tensile index due to the increased fines contribution to the bonding degree between fibers.

Analyzing Figure 5, which compares the behavior of physical properties in relation to principal components, along with the comments made to Figure 4, the behavior of the properties with refining can be better understood.

The behavior of tensile strength with refining in group 3 may be due to a condition of extreme refining, which can be confirmed by the degree of fibers cutting, i.e., the fines generation.

More absorbing than the behavior of the tensile strength is the behavior of the tear strength. As pointed out by Cowan (1995), tear strength is a far more complex property than tensile strength. It is a measure of the energy required to propagate the tear line in a plane of paper at a predetermined distance. Low tear strength occurs in pulps without refining, and there is an increase even through a short refining time in a PFI mill indicated by the °SR degree, i.e., the number of revolutions. With the increase of refining, this resistance to tearing notably decreases. Therefore, it is expected, in Figure 5, a change in the quadrant for the tensile index as the degree of refining increases with each °SR.

The tensile strength and tear strength have different influences. According to Retulainen (1996a), for tensile strength parameters such as fiber length, fiber width, specific bond strength between fibers and coarseness influence the observed values. For the tear strength, only the length and the fiber strength have some influence at a given value of tensile strength.

The tensile strength is related to the amount of fines produced in the refining. Thus, for a drainability close to 50 °SR there is a large production of fines, fact leading to a rapid increase in tensile strength, present in a quadrant opposite to the first principal component for group 3 (around 50 °SR) in Figure 5d.

The influence of fines on the tensile strength can be seen in Retulainen (1996b), who observed, through the addition of fines obtained by a two hours refining in a Valley beater, an increase in tensile strength with the increase of the amount of fines added to the pulp. At the same time, there was a decrease in light scattering or

opacity. This shows that the tensile strength is directly related to the amount of fines in the pulp, and the opacity is inversely related to it.

The fibers cutting have influence on the Gurley air permeance that also undergoes a change of quadrant in Figure 5d. Fibers cutting leads to the closure of the paper's surface increasing air permeance. This same cutting, however, causes the tear strength, which depends on fiber length, to decrease.

CONCLUSIONS

The behavior of the refining curves shown in Figure 4 is consistent with the expected refining effect given by Hiltunen (2000).

By means of the principal component analysis it can be observed, in a visually simple way, the influence of the physical properties on the results obtained for pulp refining. These data, seeming at first scattered and somewhat confusing, were analyzed with principal component analysis in a simpler form. However, to segregate the influence of properties on the results it is necessary to know the principles of each property, how they are achieved and what factors may affect them.

Observing the graphs obtained in this work, it can be seen that there is a number of factors affecting results. Unfortunately, it is not possible to isolate all sources of error, especially in interlaboratory comparisons.

The objective of this study was not to compare the interlaboratorial data of pulp in order to get a prediction of physical properties that the paper may present. This has been the subject of many papers related to the Valley beater. It is known that all factors that influence the paper properties determination limit the use of the data for this purpose. The goal was to evaluate the pulp quality and its potential use by means of data analysis.

The limitations of the evaluation of paper properties in the laboratory can be seen in Mohlin (2002), which shows that there are significant differences in properties of papers formed on a paper machine and in laboratory and, therefore, when analyzing the refining under an industry's perspective, the laboratory tests have limitations. It was not the objective of this study to achieve a higher correlation between the properties for use in industrial scale.

Skowronski and Elaahi (2003) state that the laboratory handsheets are inadequate and do not offer a simulation of the paper machine. This is one of the reasons why refining is not yet fully understood, and there is not a universal equation to predict and optimize the quality of the paper. Therefore, the prediction of industrial paper properties by laboratory evaluation has limitations.

The difficulties can also be found in the PFI mill itself. There are many works related to the use of PFI mill as a tool for the analysis of pulp physical properties, which could predict results in industry refiners. However, it is known that the refining action of a PFI mill is very different from those refiners (Yasumura, 2008) and, therefore, a direct comparison of data cannot be performed. There is need of finding a basis for comparison.

Although high dispersion in the univariate graphics from Figure 4, due to the very different calibration of each PFI mill, the several multivariate PCA analysis for 20, 30, 40, 50 °SR show clearly the changes of thickness, tensile index, tear strength, burst strength, air permeance along with the different level of refining. ∎

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