OPTIMIZING ALKALINE SIZING IN SUGAR CANE BAGASSE PAPER RECYCLING

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The objective of this work was the variation of additives dosage in the sizing treatment of sugar cane bagasse paper for liners and flutings, on taking into account the cycles of use, for reaching the same sizing degree in each cycle. The degradation of the physical properties under different conditions of relative humidity and temperature for each stage of the papermaking cycle was also studied.
The sizing agent utilized was the alkyl ketene dimer (AKD). Cationic starch and a retention agent (a modified high molecular weight polyethylenimine) were also added. The experimental design applied was a Central Composite Design (CCD). Once the optimum dosage of additives found, the sheets were submitted to different conditions of humidity and temperature, and their physical properties were tested. Three papermaking cycles were carried out: a papermaking cycle, followed by two recycles.
It was observed that a lower amount of AKD was required to reach the water absorption objective (measured by the $Cobb_{120}$ test), as it progresses during the papermaking cycles, and also that good properties in liner and fluting paper could be attained in spite of the recycles, although, at 75% relative humidity (RH), the liner and fluting properties drastically decreased.

Keywords: recycling, alkaline sizing, AKD, bagasse

INTRODUCTION

The use of recycled paper as a raw material is nowadays a fundamental economic factor, although it is generally accepted that the quality of secondary fibers is lower than that of virgin pulp.[1-5] Mechanical and chemical treatments contribute to the reduction of this difference, yet without wholly recovering the total quality of virgin pulp.[6-8]

Whatever the origin of the raw material, a sizing treatment has to be carried out in a stock preparation stage for liner and fluting papermaking, to limit the penetration rate of aqueous liquids into the paper sheet (in this case, especially water absorption). This is a fundamental condition for corrugated cardbox, since it should maintain its resistance with respect to variation in humidity. To this end, a sizing agent is added, besides other additives, such as cationic starch and a retention agent, to improve its performance. The sizing mechanism of AKD has been thoroughly studied.[9-21] The AKD forms covalent ester bonds with the cellulose hydroxyl groups, although it does not cover the complete fiber surface and does not interfere with interfiber bonding.[22,25] Nevertheless, some investigators question the existence of covalent unions between AKD and fibers.[26] It is also known that the sizing effect is lost when sized fibers are re-slushed. The influence of the previous treatment on the new sizing has been studied by Sjöström and Ödberg,[27] in a bleached softwood kraft pulp. These authors found out a residual effect of the previous sizing treatment, as the fibers were hydrophobized to some extent.

Sugar cane bagasse fibers are more and more frequently found in recycled pulp used for liner and fluting paper, in national productions. This work is focused on the behavior of bagasse pulp subjected to sizing treatments during recycling. No minerals were added to each papermaking cycle, for minimizing their interference in the sizing treatment, although an industrially recycled pulp could be obtained.

Even when some studies suggest that the use of large quantities of secondary fibers requires higher amounts of additives, some mills that use great amounts of recycled fibers have noticed no changes in the consumption of sizing and retention agents.[28]

The influence of relative humidity on the mechanical strength of sized paper was described by different authors, using other kinds of fibrous materials.[29-31]

EXPERIMENTAL

A sample of sugar cane bagasse semichemical pulp was used as raw material. This pulp was screened on a Weverk screen equipment (0.15 mm slots). In the first stage, the screened pulp was characterized as to fiber length, refining degree and percentage of fines passing through a 200-mesh, after which the pulp was mechanically treated in a PFI refiner, recording the energy consumption, in accordance with the refining degree industrially applied for this raw material (35 ºSR).

For the sizing treatment, an aqueous dispersion of a fatty alkyl ketene dimer with a medium cationic charge (Basoplast 4118 MC) was used. To achieve a better performance, the sizing agent was accompanied by cationic starch and a retention agent (high molecular weight polyethylenimine, Polymin SK).

A solution of 1% cationic starch was prepared after cooking it at 90-95 ºC for 15 min. According to the manufacturer's recommendations, the aqueous solutions of the sizing and retention agent were formulated during the addition to the furnish.

The additives were added to the pulp at 1% consistency, under stirring for 30 s at high speed (TAPPI disintegrator), to ensure a correct mixing. The addition was carried out observing the following order: 1) cationic starch, 2) AKD, and 3) retention agent.

The pH of the medium for the chemical treatment was fixed at 7.5 with H_2SO_4 1N, according to manufacturer's specifications, in relation to the optimum pH range of the sizing treatment, and also to the performance of the retention agent.

The optimization of the chemical treatment was carried out with a three-factor experimental design called Central Composite Design (CCD) involving: (I) a two-level full factorial or fractional factorial design; (II) a star design in which the experimental points occur at an α distance from its center; and (III) an experimental point in the center.[33] The center runs provide information on the existence of a curvature in the system. By these means, the design displays properties, such as rotability or orthogonality, for fitting the quadratic polynomials. CCD allows the estimation of the main effects, as well as of the interactions and quadratic effects. Usually, CCD consists of a 2k factorial runs with 2k axial runs and C_0 center point runs.

The concentration range studied for each additive was taken from the literature in the case of cationic starch: 0.25 to 0.50% on oven dry pulp base (odp), and from the specifications sheet in the case of the sizing agent (1 to 2% odp) and of the retention agent (0.1 to 0.25% odp), the last two being expressed as percentages of commercial products. The levels studied are shown in Table 1.

Particle charge was determined through the Total Cationic Demand (TCD), for checking a -0.5 to -0.05 meq/L medium charge in each point of the experiment. This range was recommended by the sizing and retention agent supplier, to ensure the good performance of the additives.

Testing sheets of 120 g/m^2 were formed in a Rapid Köethen from the furnish, as indicated by each point of the experimental design. The sheet-former equipment has a drying system operating under vacuum, at a temperature of 120 ºC, for 5 min, for AKD curing.

Water absorption was measured on the formed sheets by the $Cobb_{120}$ method. The optimum condition was defined as a $Cobb_{120} = 35 \pm 1$ g H_2O/m^2.

Once the optimal dosage to achieve the water absorption objective was established, sufficient sheets were formed to obtain the raw material for the following papermaking cycles. The use of the material was simulated by subjecting the sheets to different conditions of relative room humidity and temperature, for 24 h at each environmental stage (Table 2).

Ring Crush Test, Concora Medium Test, Tensile Index and Burst Index tests were carried out on air-conditioned paper in sequences of 50, 75 and 90% relative humidity (RH), in the water adsorption stage, and at 75 and 50% RH in the desorption stage. These values were selected as the most representative ones for the environmental conditions to which a corrugated cardboard package can be subjected.

Following the humidity adsorption and desorption stages, the sheets were disintegrated in water, at room temperature, in a 15 L pulper. The characterization of the recycled pulp involved the measuring of fiber length, percentage of fines passing 200 mesh, pulp refining degree (ºSR) and water absorption ($Cobb_{120}$ method), on 120 g/m^2 sheets.

Further on, the pulp was refined in a PFI mill, to the initial refining degree (35 ºSR), and the energy necessary for achieving it was measured. The optimization study of the additive dosages (Table 1) was repeated on the refined pulp, to obtain the same quality of paper ($Cobb_{120}$ = 35 ± 1 g H_2O/m^2), and the sheets were formed again under the same conditions. Two recycling stages were simulated, called from now on second (first recycling) and third papermaking cycle (second recycling).

Table 1
Treatment combinations (t_c) in the CCD experimental design (as real variables)

Nº	A	B	C	Sizing agent (% odp)	Retention agent (% odp)	Cationic starch (% odp)
1	-1	-1	-1	1.0	0.10	0.25
2	1	-1	-1	2.0	0.10	0.25
3	-1	1	-1	1.0	0.25	0.25
4	-1	+1	-1	2.0	0.25	0.25
5	-1	-1	+1	1.0	0.10	0.50
6	+1	-1	+1	2.0	0.10	0.50
7	-1	+1	+1	1.0	0.25	0.50
8	+1	+1	+1	2.0	0.25	0.50
9	No additives	No additives	No additives	0	0	0
10	- α	0	0	0.66	0.175	0.375
11	+ α	0	0	2.3	0.175	0.375
12	0	- α	0	1.5	0.05	0.375
13	0	+ α	0	1.5	0.3	0.375
14	0	0	- α	1.5	0.175	0.165
15	0		+ α	1.5	0.175	0.585
16	0	0	0	1.5	0.175	0.375

Table 2
Environment parameters in acclimatization stages

Stage	RH (%)	T (ºC)
1	50	23
2	75	27
3	90	30
4	75	27
5	90	30

RESULTS AND DISCUSSION

Pulp characterization and preparation

Pulp characterization prior to mechanical and sizing treatments and the refining energy for each papermaking cycle are shown in Table 3. The pulp used in the first papermaking cycle shows the quality of the virgin pulp. The pulp was refined in each papermaking cycle for opening the fibrous structure to generate new binding points, by subjecting the fiber to the same treatment that it would receive in a mill.

The energy supplied per unit of refining degree gained in the recycled pulp was lower than that supplied for the virgin pulp. In the first papermaking cycle, fiber fibrillation is the major cause of the increase in the refining degree while, in the subsequent uses, cutting is believed to be the most important effect of refining, caused by fiber hornification.

In the second papermaking cycle, the initial degree of refining was lower than the objective, and fibers were refined to 35 ºSR while, in the third papermaking cycle, the initial degree of refining was higher than the objective. As expected, the additions in previous cycles had altered the Schöpper value, so that the recycled pulp was refined by increasing the Schöpper value to 5 points.

The first part of this work demonstrates that, during recycling, fiber length presents a mean decrease of 30% in the second papermaking cycle *versus* the first one, as due to fiber cutting. The third papermaking cycle presented no significant differences in relation to the second one. Fines in the bagasse pulp, in the first and second papermaking cycles, occur in the range of 15%, and for the third one – in the range of 20% (Table 3).

Optimization of sizing treatment (application of CCD design)

The statistical treatment of the CCD design is based on the Analysis of Variances (ANOVA). The experimental results were fitted to a second-order polynomial model including only the significant variables at a 95% level of significance ($P < 0.05$). Thus, the best equations obtained for water absorption in different cycles were as follows:

1) For the first papermaking cycle:

$$\text{Water absorption } (\text{Cobb}_{120}) = 40.8 - 5.8 \times \text{Sizing agent} - 41.1 \times \text{Starch} + 3.3 \times \text{Sizing agent}^2 - 35.6 \times \text{Sizing agent} \times \text{Starch}$$

$R^2 = 71.5\%$

2) For the second papermaking cycle:

$$\text{Water absorption } (\text{Cobb}_{120}) = 42.7 - 11.3 \times \text{Sizing agent} - 133.5 \times \text{Retention agent} + 2.6 \times \text{Sizing agent}^2 + 263.5 \times \text{Retention agent}^2 + 64.7 \times \text{Starch}^2 + 144.8 \times \text{Retention agent} \times \text{Starch}$$

$R^2 = 64.5\%$

3) For the third papermaking cycle:

$$\text{Water absorption } (\text{Cobb}_{120}) = 36.0 - 2.0 \times \text{Sizing agent} + 4.9 \times \text{Retention agent} + 4.8 \times \text{Starch}$$

$R^2 = 60.5\%$

Table 3
Pulp characterization before and after mechanical treatment

Pulp	Fiber length µm	Initial refining degree °SR	Final refining degree °SR	Fines %	Energy J/°SR
1st cycle	1360 ± 458	18	35	17.3	2742
2nd cycle	980 ± 489	26	35	16.2	807
3rd cycle	893 ± 361	39	44	20.2	1841

Note: Fiber length expressed as average ± standard deviation

The optimum (minimum) dosage of additives obtained by the equations above and the cost of each treatment (combination of additives) are presented in Table 4. The theoretical and experimental results of the optimum additive dosage, and the effects of the additives for different uses are shown in Table 4 and Figures 1 to 5, respectively. At the beginning of recycling, the effect of the sizing treatment was lost. It is believed that this effect appears during the tap water reslushing stage, when the chemicals had been partially desorbed from the fiber surface.[27] The analysis of the overall results, within the same additives dosage limits, for the three papermaking cycles shows that water absorption (Cobb's method) decreases with recycling, from

46-42 in the first to 36-35 in the third papermaking cycle (Figs. 1 to 5). The bagasse recycled pulp did not seem to require so many additives as the virgin pulp to achieve the same sized quality. Otherwise said, each new recycling will require fewer additives for the sizing treatment, which might be due to fiber hornification (and its consequent hydrophobicity), in accordance with Sjöstrom *et al.*,[27] rather than to a sizing residual effect, since the secondary fiber sheets formed without sizing treatment had no capacity of water absorption. Sjöstrom *et al*. suggested that the recycled fibers obtained from sized sheets are hydrophobized to some extent for subsequent uses and that, even if major desorption occurs at high ionic strength of the solution, the sizing treatment in a recycled pulp evidences no differences between an alkaline and an aqueous slushed medium.

In the first papermaking cycle, the sizing agent, the starch and their interaction greatly influence the response (Figs. 1 and 2). The retention agent (high molecular weight polyethylenimine) has no significant influence over the working range proposed by the manufacturer, its activity being primarily based on its ionic strength. It is also assumed that the ionic nature of the fibrous mass is sufficiently anionic to promote the reaction between fiber, sizing agent and cationic starch. In this case, the mean value of the cationic demand for each combination of the experimental design was -0.11 meq/L for virgin pulp and -0.05 meq/L for recycled pulp, respectively.

The effects of the variables and the interactions between them upon the second-cycle pulp are illustrated in Figures 3 and 4, while the effects of the variables upon the third-cycle pulp are presented in Figure 5. The sizing and retention agents are the most influential factors in the response to the second papermaking cycle. The linear effect of starch is not significant, although it presents a second-grade polynomial relationship in the response. The sizing agent and, secondarily, the starch, are the factors that influenced the most the response to the third papermaking cycle (Fig. 5).

The effect of the sizing agent (AKD) on water absorption presented a similar behavior in all three papermaking cycles (Figs. 1, 3 and 5), which agrees with the result of the $Cobb_{120}$ test carried out at the beginning of each cycle, as the paper lost all the sizing in the repulping process. It seems that the ageing of the handsheets under high humidity conditions caused the hydrolysis of the unreacted AKD, which contributes to sizing.[34] In the first two papermaking cycles, the effect of AKD is represented by a parabola, which confirms the optimal range of work recommended by the suppliers. It is not possible to assure the same response in the third papermaking cycle, not even beyond the studied range. The effect of both starch and retention agent on water absorption has moved from being directly proportional (first cycle) to indirectly proportional (third cycle), presenting a minimum in the second cycle. This demonstrates the gradual saturation of the system in relation to these additives. Sjöstrom and Ödberg[27] demonstrated that the adsorption capacity of the cationic polymers diminishes with an increasing number of papermaking cycles. However, it cannot be neglected that the presence of retention agents and cationic starch from previous uses contributes to the formation of microflocs which, in turn, diminish the exposed surface.[35]

Table 4
Optimum dosage of additives and sizing treatment cost for a stock close to neutral charge

Pulp	Sizing agent (% odp)	Starch (% odp)	Retention agent (% odp)	TCD (meq/L)	Cost (US $/ton pulp)
1st cycle	2.45	0.10	0.27	-0.17	6.4
2nd cycle	2.15	0.42	0.175	-0.06	23.9
3rd cycle	1.60	0.17	0.35	-0.15	21.1

Note: DCT (Total Cationic Demand)

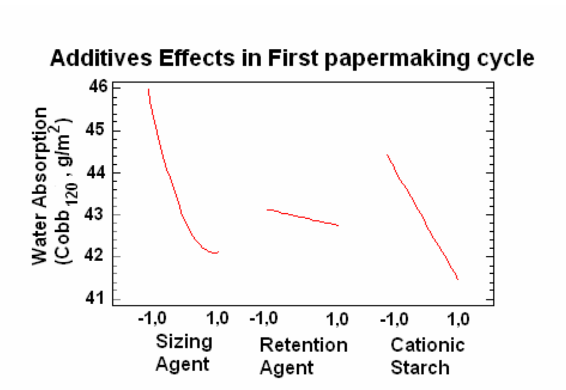

Figure 1: Effects of sizing agent, cationic starch and retention agent on water absorption in the first papermaking cycle

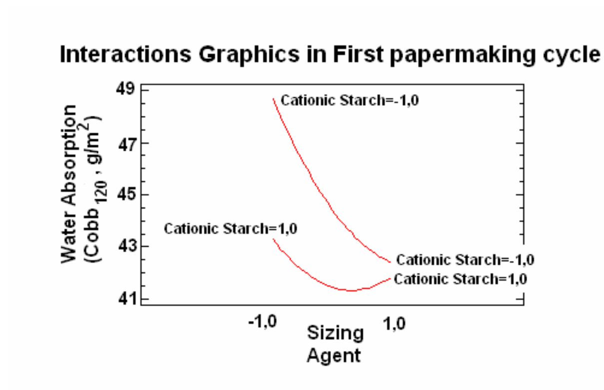

Figure 2: Interaction between cationic starch and sizing agent in the first papermaking cycle

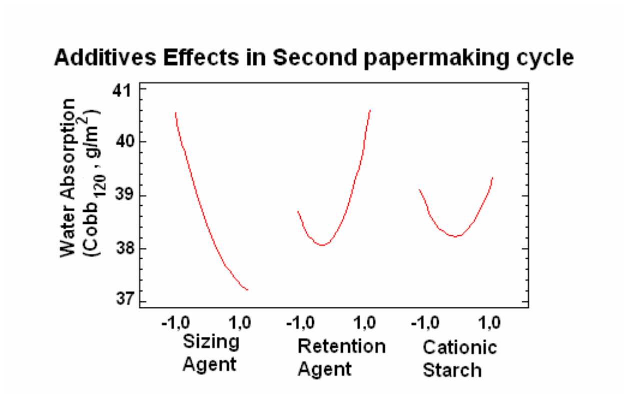

Figure 3: Effects of sizing agent, cationic starch and retention agent on water absorption in the second papermaking cycle or first recycling

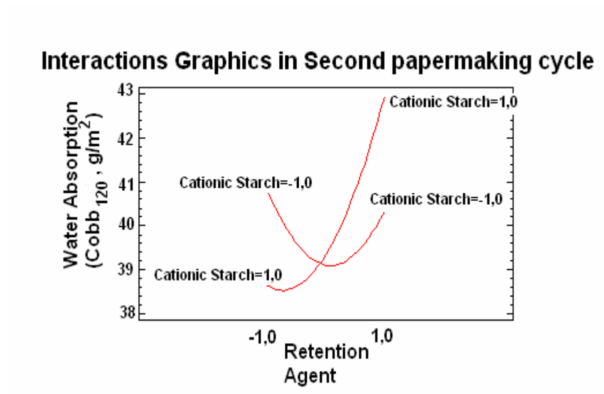

Figure 4: Interaction between cationic starch and retention agent in the second papermaking cycle or first recycling

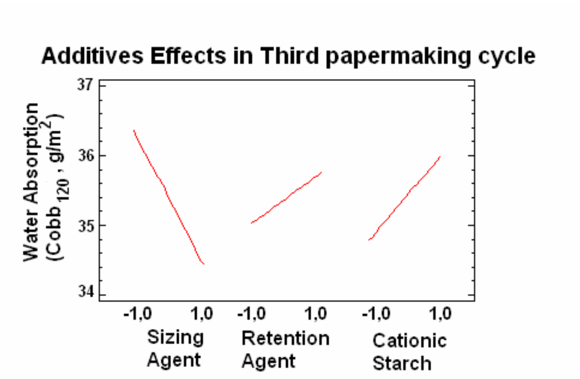

Figure 5: Effects of sizing agent, starch and retention agent on water absorption in the third papermaking cycle or second recycling

It should also be taken into account that, due to the methodology used, our system is free of the fines and anionic trash occurring in industrially recycled pulps. The large specific surface of these pulps would be the main responsible for the higher consumption of additives in the mills. In future studies, the addition of additives will be avoided, to observe whether the development of sizing is possible using only the residual and auxiliary agents from previous papermaking cycles.

The behavior of cationic starch with respect to water absorption varies with the papermaking cycles. However, it always influences each cycle, in an either linear or quadratic form. It also interacted with the other additives, presenting a synergetic effect with the sizing agent from the virgin pulp. It did not interact with the retention agent in the second papermaking cycle, because of the interference produced by the retention agent, which has cationic characteristics.[1]

In a neutral medium, the ionic nature of the retention agent plays an important role.[35] Unlike other retention agents, polyethylenimine presents a short molecular chain, its action being based on the formation of microflocs. On the other hand, it favors the decrease of water viscosity, which improves drainage in the formation stage.

Physical properties

The physical properties of sized papers in the adsorption–desorption cycles, as a function of relative humidity (RH), are presented in Figures 6 to 9. The properties of the sheets obtained from bagasse pulp recommend them for their use as liner and fluting material, in comparison with those obtained from industrial paper, which can be partly due to the use of a PFI refiner, known as producing fewer fiber cuts and higher internal and external fibrillation than industrial refiners.

The tensile and burst indices are significantly better in virgin pulp than after the subsequent papermaking cycles (Figs. 8 and 9). According to the variance analysis, after the adsorption and desorption of humidity, the paper properties from virgin pulp (first papermaking cycle) generally returned to the values of the standard test conditions (50% RH).

The burst index classifies liners into three different qualities: Kraftliner, Testliner and Biclass.[36] According to this classification, the virgin bagasse pulp sheets (first papermaking cycles) presented the quality of a Kraftliner, while the secondary fiber paper (second and third papermaking cycles) evidenced the quality of a Testliner.

Generally, pulps behave within the same range of test conditions (50% RH), even after having undergone the adsorption–desorption cycle. Among the properties assayed for cardboards, paper mechanical compression in the first papermaking cycle was highly diminished, between 75-80% RH and, in spite of some recovery, cardboards did not return to the same quality. The three pulps present the same behavior as class-B fluting.[36] In all cases, the quality of the paper formed from bagasse pulps recommends it for the manufacture of liner and fluting.

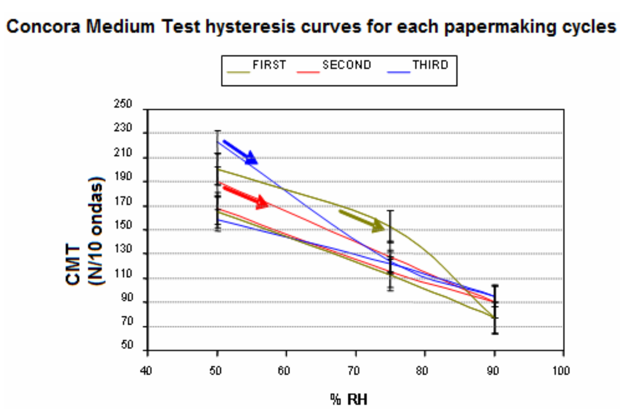

Figure 6: CMT (Tappi 809 om – 99) variation with RH for each papermaking cycle

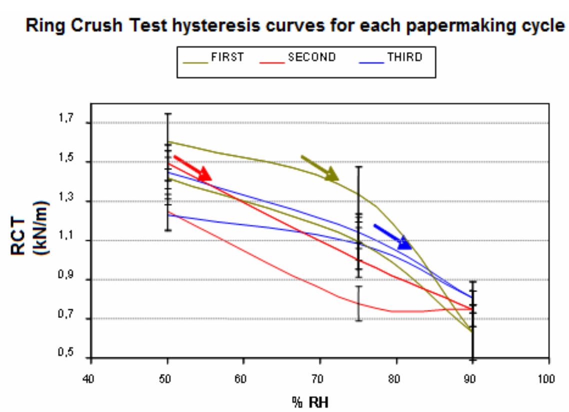

Figure 7: RCT (Tappi 822 om – 93) variation with RH for each papermaking cycle

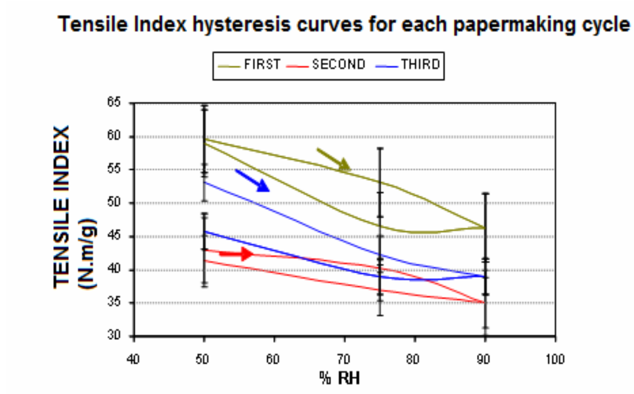

Figure 8: Tensile index (Tappi 220 sp – 96) variation with RH for each papermaking cycle

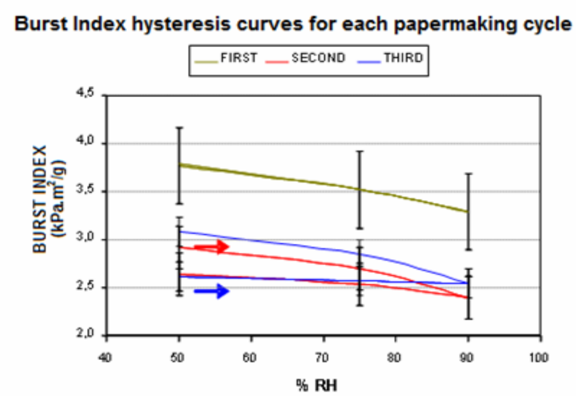

Figure 9: Burst index (TAPPI 220 sp – 96) variation with RH for each papermaking cycle

CONCLUSIONS

The effect of the sizing treatment is lost during repulping with tap water. However, it is noticed that the sizing effect in papermaking cycles is more easily attained when the sludge has no mineral charges and the level of anionic trash is low.

Both cationic starch and the retention agent (high molecular weight polyethylenimine) continue to be used, in spite of all mechanical effects suffered by the fiber during pulp preparation.

For a given sizing, the paper produced from sugar cane bagasse semichemical pulp, destined for liner or fluting, did not modify its behavior with respect to the papermaking cycles, under standard test conditions. On the contrary, during the first papermaking cycle, its properties had drastically diminished at a relative humidity of around 75%. In relation to the other papermaking cycles, the deterioration is uniform with respect to the increase in humidity.

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