EFFECT OF DRYING AND CHEMICAL TREATMENT ON BAGASSE SODA PULP PROPERTIES DURING RECYCLING

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Effects of chemical treatment on the potential for recycling of bagasse pulp were evaluated. The pulps were recycled three times with water (without treatment), sodium hydroxide, and ethylamine separately. Changes in crystalline structure of the pulp during recycling were investigated by x-ray diffractometry. Water retention content was measured by centrifugation. Morphological changes were investigated by scanning electron microscopy. The breaking length, burst index, fold number, water retention value, and density decreased continuously after recycling. Chemical treatment didn't have a positive effect on the swelling of the recycled pulp. But the light scattering coefficient increased continuously. The crystallinity index and crystallite size increased to an extent, compared to virgin pulp. Some particles on recycled fibers with chemical treatment and treated handsheets had more curled fibers than untreated handsheets.

Keywords: Bagasse soda pulp; Recycling; Chemical treatment; WRV; Mechanical and optical properties; Scanning electron microscopy

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INTRODUCTION

Paper recycling is an important part of the paper industry, mainly motivated by economic profitability concerns. Paper recycling also has benefits to society, including the reduction of landfill loadings and less dependency on forest resources. Secondary fiber utilization is currently increasing at a rapid pace. In recent years, non-wood fibers have been increasingly considered as a potential source of papermaking raw material. The EPA estimates that, with current technology, the production of recycled paper uses 50% less water and reduces air pollution by 74%, compared to the production of new, virgin paper (Brancato 2008). Some studies have shown that the xylans of pulp increase the potential of recycling (Cao et al. 1998). Because many non-wood plant materials have high xylan content, it was expected that this fibers might show a good potential for recycling (Garg and Singh 2004).

Recovering the bonding potential of dried fibers can be done by beating and chemical treatment of recycled fibers (Howard and Bichard 1992). Garg and Singh (2004) studied recyclability of bleached chemical pulps from wheat straw and bagasse only with refining. Drying influences fiber strength, fiber swelling, and bonding potential, which are important for the strength of paper made from dried fibers (Brancato 2008; Howard and Bichard 1992; Hubbe et al. 2007; Jahan 2003). Wistara and Young (1999) observed that treatment of recycled bleached kraft pulps with aqueous and organic bases does not have a beneficial effect on the recovering the loss of bonding of recycled fibers.

The changes taking place in the fiber on recycling have been mainly attributed to hornification. According to Jayme (1958), hornification is a loss of wet fiber flexibility and bonding ability caused by self association of cellulose microfibrills during drying. Also, hornification affects the water retention value (WRV), which is defined as a ratio of water to dry fiber after centrifugation of a fiber pad under standard conditions and is used as a measure of fiber swelling. Fiber swelling is accompanied by an increase in fiber flexibility and fibrillation, which are of importance in developing fiber-fiber bonding in paper. In many articles, WRV is used as representative of the degree of fiber flexibility (Brancato 2008; Howard and Bichard 1992). Hornification causes the loss of water retention value and decreases the tensile strength of recycled pulp. At the most basic level, hornification is the permanent loss of swellability in cellulose fibers, leading to a loss of fiber flexibility. Fiber flexibility and swellability are the main contributors to the strength of interfiber bonding. Fibers that have been dried more closely resemble their dried state after rewetting than they do their never-dried state (Brancato 2008).

Gurnagul (1995) showed that adding sodium hydroxide to TMP pulp increased the rate of fiber swelling. In contrast, adding sodium hydroxide had no significant effect on swelling of low- yield, unbeaten bleached and unbleached kraft pulps.

Attala (1992) found that recycling increases pulp fiber crystallinity and decreases surface area. The increase of dried pulp crystallinity is thought to happen mainly because of the heating effects in the drying process. Swelling of the dried fibers would be decreased. This is indicated by the decrease of water retention value of dried fibers. The decreasing capability of the fibers to swell also decreases the tensile strength of the pulp. Cellulose is a homopolymer containing crystallites. The linear cellulose molecules are linked laterally by hydrogen bonds to form linear bundles, giving rise to a crystalline structure. The crystalline regions are interrupted every 60 nm with non-crystalline amorphous regions for all raw materials. Therefore, the crystalline structure of cellulose affects the physical and mechanical properties of cellulose fibres. The degree of crystallinity of cellulose is one of the most important of structural parameters of cellulose. Flexibility decreases with increasing ratio of crystalline to amorphous regions (Gumuskaya et al. 2003). Wistara et al. (1999) showed that recycling did not change the crystallinity of treated and untreated pulp, and they calculated the crystallinity by FTIR. Some investigators have suggested that alkali treatment influences the swelling capacity of the fibers, which is a very important factor to improve the bonding potential of the fibers (Howard and Bichard 1992). It is suspected that ethylamine treatment does not decrystallize cellulose but causes swelling of cellulose (Venkasteswaran and Van den Akker 1965). Also, the morphology of recycled fibers has been studied using microscopy techniqes, such as scanning electron microscopy under various magnifications (Howard and Bichard 1992).

While there is much information about different pulping conditions and characteristics of the recycled wood fibers, there is limited information on the behavior of non-wood fibers on recycling. In addition, most research has focused on mechanical treatment of recycled pulp. In this research, effects of chemical treatment and drying on the properties of baggase pulp during the cycles of recycling have been investigated.

EXPERIMENTAL

Bleached soda bagasse pulp was prepared by Pars Paper Co., which is located in Haft Tapeh, Khuzestan, Iran. Paper sheets were prepared in accordance with TAPPI standard T205 sp-95, with a modification: Instead of drying the sheets under a constant humidity condition, the wet hand sheets inside drying rings were placed in an oven at 90°C for 1 hour in order to simulate the effect of drying on a paper machine. The handsheets were repulped (recycled) with and without chemical treatment. Sodium hydroxide and ethylamine was used for chemical treatment in each step. The chemical treatment was utilized for swelling recycled pulp. These treatments were selected based on previous results of many investigators regarding the effects of aqueous and organic liquids for the recycled pulp fibers (Freeland Hrutfiord 1994; Gurnagul 1995; Wistara and Young 1999). Chemical treatment of recycled pulp was carried out overnight at a concentration of 5% (based on oven dried pulp) at a pulp consistency of 9% and at room temperature. The application of 5% concentration of chemicals and room temperature were selected based on the work of Wistara and Young (1999). Then the pulps were washed carefully with water and the moisture content of pulp was reduced to approximately 80%. The handsheets were then recycled in 3 steps.

The strength properties of handsheets were tested according with TAPPI method om-99. Breaking length, burst strength, tear resistance, and fold number were determined using TAPPI test methods T494, T403, T414, and T220, respectively. Brightness, opacity, and light scattering coefficient were determined using ISO 2469. Data presented in this paper are average numbers of three to five repeats.

Water Retention Value

The water retention value (WRV) was measured according to the following procedure: one gram of pulp of known water content was disintegrated, put into a 200 cm^3 Erlenmeyer flask and suspended in $100cm^3$ of distilled water. The suspension was shaken for 1h at 20 °C, and then transferred to a G_3 sintered-glass funnel to remove excess water under reduced pressure. The sintered-glass funnel was then transferred to a centrifuge tube and centrifuged at 2000G for 15 minutes. Then, WRV was calculated according to Formula 1 (Rom et al. 2007; Xiao and Meshitsuka 2001),

$$WRV = \frac{m_2 - m_1}{m_1} \times 100 \tag{1}$$

where m_1 equals the mass of dry sample, and m_2 equals the mass of moist sample. Four WRV measuring were conducted in each experimental series to calculate the average value.

Scanning Electron Microscopy

A Philips scanning electron microscope (XL30 model) was used for studying the morphology of the fibers after recycling. All images were taken at 17 kV power. The surface was coated with gold prior to analysis, which was done with a SCD005 sputter coater.

X- Ray Diffraction Method*

X-ray diffraction was performed with a Rigaku 3D/Max series diffractometer. The radiation was Ni-filtered CuKa of wavelength 0.1542 nm. The X-ray unit operated at 40 kV and 30 mA. Angular scanning was conducted from 3° to 50° at 1°/min, and data were collected using a 2-step scan mode with angular intervals of 0.05.

The crystallinity of cellulose in pulp samples was calculated from diffraction intensity data. The first approach was the empirical method proposed by Segal et al. (1959) for native cellulose,

$$CrI = \frac{I002 - Iamorph}{I002} \times 100 \tag{3}$$

where *CrI* is the degree of crystallinity, *I002* is the maximum intensity of the (002) lattice diffraction, and *Iamorph* is the intensity diffraction at 2θ= 18°. The average size of a crystallite was calculated from Scherer equation (Gumuskaya et al. 2003),

$$D_{(hkl)} = \frac{k\lambda}{B_{(hkl)} Cos\ \theta} \tag{4}$$

where $D_{(hkl)}$ is the size of crystallite, k is the Scherer constant (0.84), λ is the x-ray wavelength, $B_{(hkl)}$ is the FWHM (Full Width Half Maximum) of the measured *hkl* reflection, and 2θ is the corresponding Bragg angle (Gumuskaya et al. 2003).

RESULTS AND DISCUSSION

SEM Images

The morphology of recycled fibers was studied by scanning electron microscopy (SEM) under various magnifications. The SEM micrographs of handsheets from recycled pulps treated with chemical treatment showed more curled fibers (Figs. 1 though 3).

*This experiment was done at the Dept. of Pulp and Paper, Kardeniz Technical University, Turkey by Prof. Esat Gumuskaya.

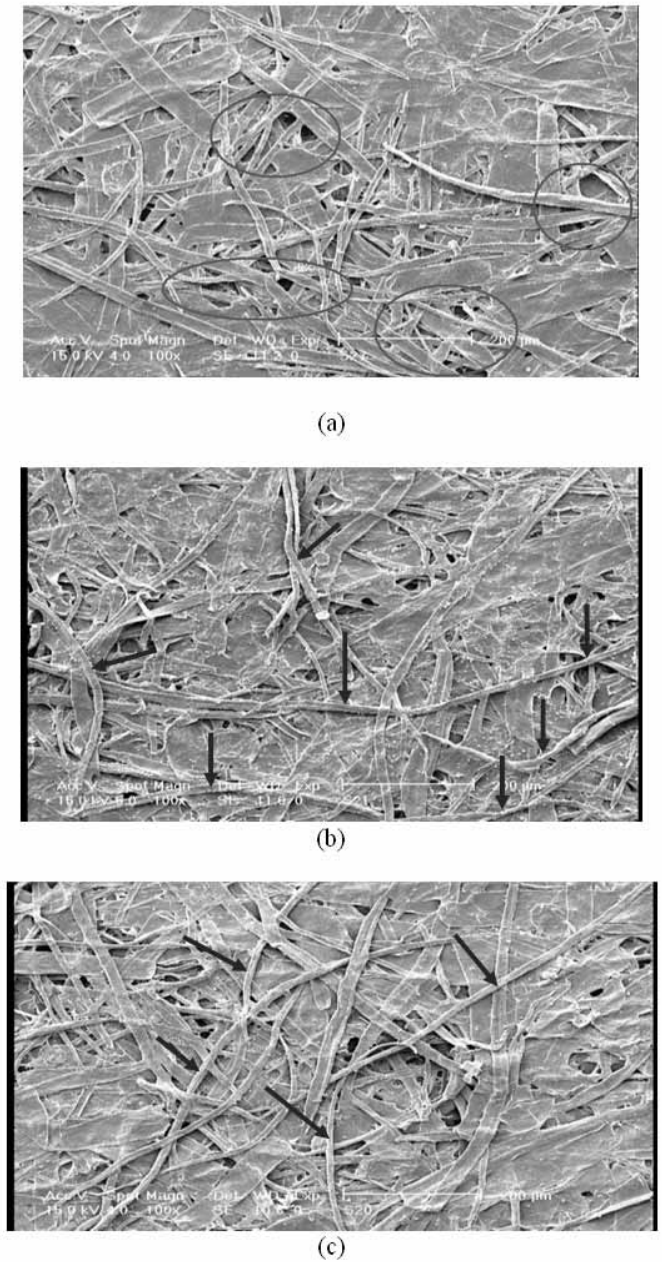


Figure 1. SEM images (100X) of first recycled hand sheets, untreated (a), NaOH-treated (b), and ethylamine treated (c)

Curling of fibers in the handsheet network could play a significant role to decrease the strength of the recycled pulps. Also, traces of solids that are left in the handsheets can function as filler in the handsheets and inhibit bonding (Wistara and Young 1999). The untreated recycled fibers appeared thinner than virgin fibers (Fig. 4). Such alteration in the appearance of recycled fibers also was found by Nanko et al. (1991) with electron microscopy.

Traces of solids could be observed on virgin fibers. The amounts of these solids were higher when the recycled pulps were treated, while there were no solids on untreated recycled fibers (Figs. 1 through 4). These solids were analyzed by an EDX[1] system and shown to include mostly the Ca. and Si elements. But there other elements such as Cr., Mg, and Fe were detectable (Fig. 5).

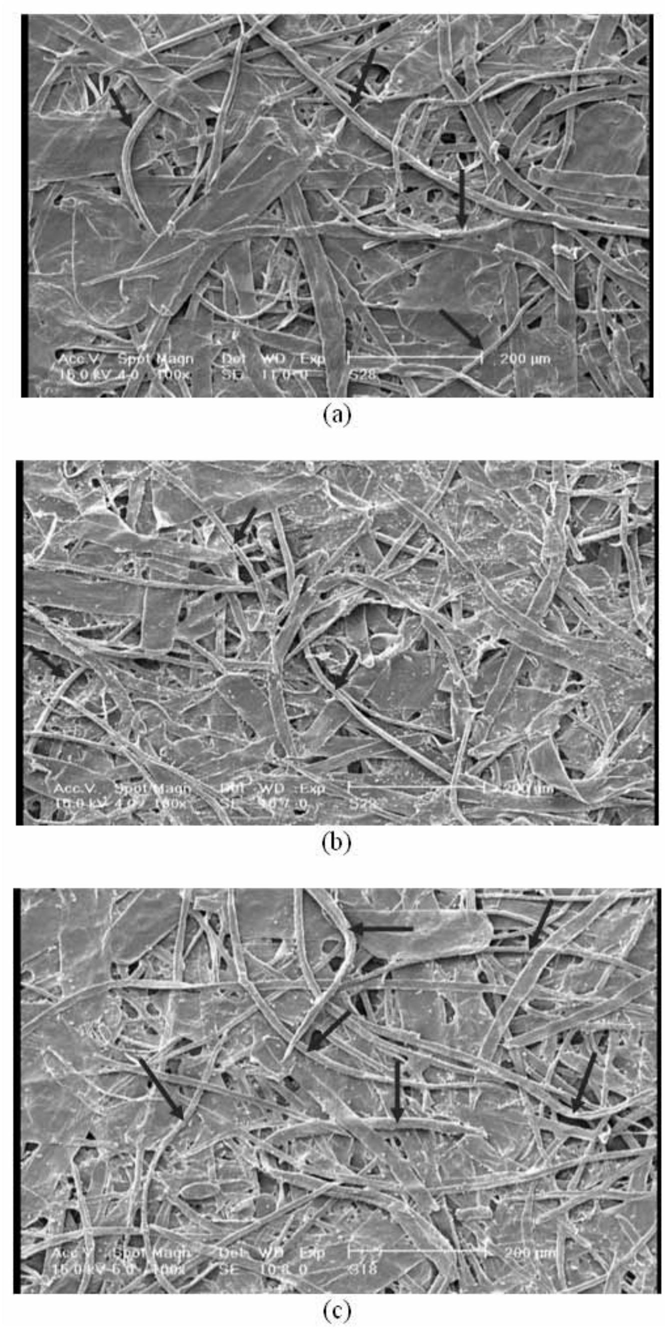
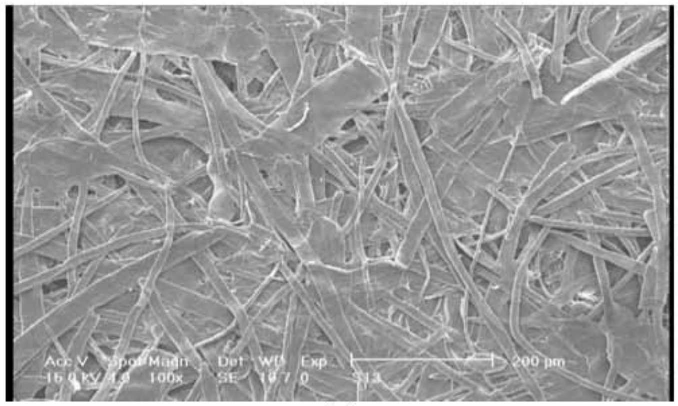


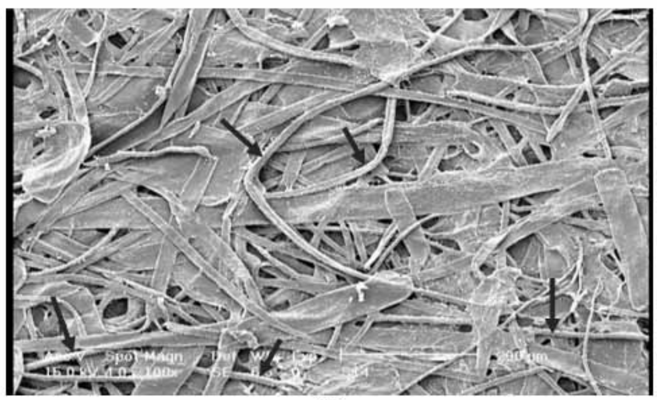
Figure 2. SEM images (100X) of second recycled hand sheets, untreated (a), NaOH-treated (b), and ethylamine-treated (c)

[1] SEM was equipped with an EDX system which can recognize the elements in the solids.

Villavicencio (1980) states that bagasse fibers typically contain about 0.5 wt. percent of silica within their structure and up to about 3 wt. percent of occluded sand and dirt. This sand and dirt contaminates the fibers at the time of harvesting of the cane and gets worked into the fiber bundles primarily during the production of sugar syrup (Villavicencio 1980).



(a)



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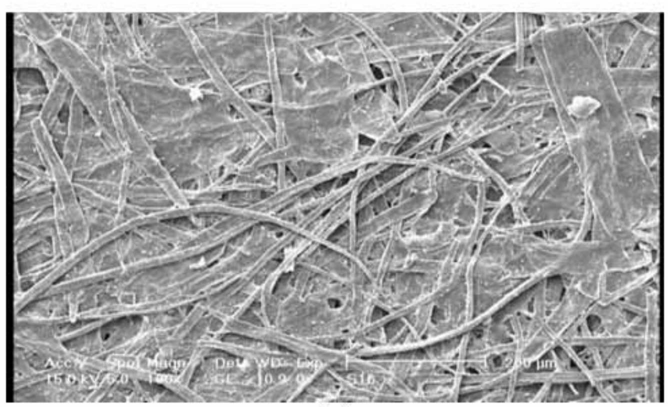
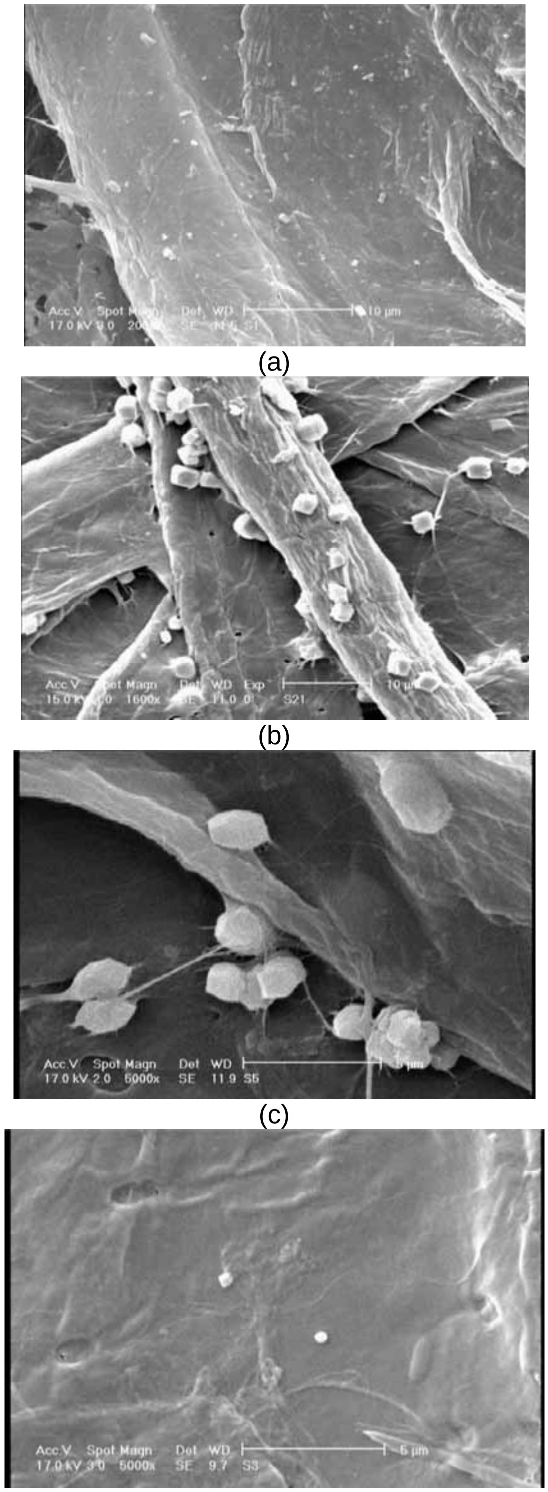


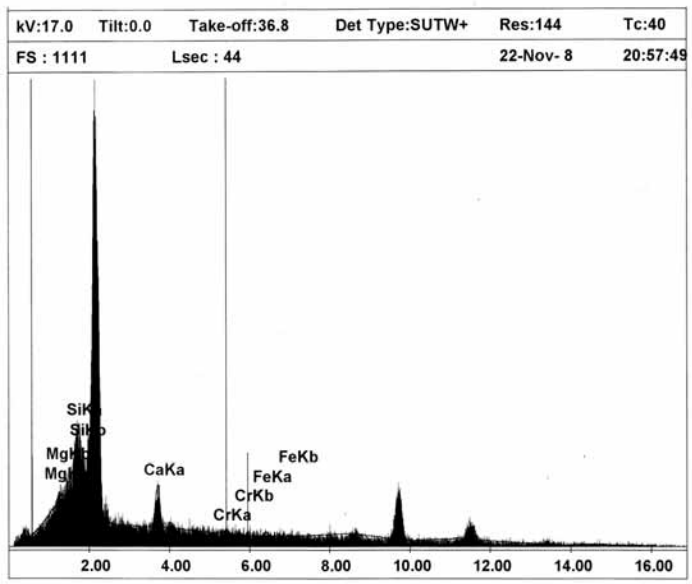
(c)

Figure 3. SEM images (100X) of third recycled hand sheets, untreated (a), NaOH-treated (b), and ethylamine-treated (c)



(a) (b) (c) (d)

Figure 4. SEM images under different magnifications, (a) unrecycled fibers (2000X), (b) Treated recycled fibers with ethylamine (1600 X), (c) treated recycled fibers with NaOH (5000 X), and (d) untreated recycled fibers (5000X)



EDAX ZAF Quantification (Standardless)
Element Normalized

Element	Wt %	At %
MgK	1.95	2.84
SiK	39.72	50.22
CaK	37.77	33.47
CrK	8.40	5.74
FeK	12.15	7.72
Total	100.00	100.00

Element	Net Inte.	Bkgd Inte.	Inte. Error	P/B
MgK	1.44	26.98	55.44	0.05
SiK	37.61	24.88	3.16	1.51
CaK	23.81	13.43	3.85	1.77
CrK	2.80	10.27	19.41	0.27
FeK	2.87	9.03	18.08	0.32

Figure 5. A sample of compositions the solids on recycled fibers

Crystallinity Properties

Bleached bagasse pulp initially had a crystallinity of 51.2%. During recycling the crystallinity increased up to 57.9% when the handsheets were recycled 3 times without any treatment. But the crystallinity increased to 52.2 and 54.9% when the handsheets were recycled with sodium hydroxide and ethylamine, respectively. It seems that chemical treatment prevented the increasing of crystallinity a little. For unrecycled pulp, the crystallite size of cellulose at 002 lattice plane was determined as 4.1 nm, and it increased after 3 times recycling to 4.9 nm. The crystallinity was increase during three-fold recycling without any treatment. The effect of ethylamine was more than that of sodium hydroxide. The increase in crystallinity was possibly originated from a slight increase in crystalline to amorphous ratio of fibers during recycling. The decrease in the amount of the amorphous region, affected the water absorbability of fibers, was indirectly detected as a loss in WRV.

Crystallinity of cellulose in bagasse pulp has been reported as 58.8% (Ibrahem et al. 1989). Bugajer (1976) observed that crystallinity increased after five recycles. Yamagishi and Oye (1987) reported a 1.1 to 1.2% linear increase in crystallinity index of softwood and hardwood kraft pulp throughout five recycles.

Although recycled pulps treated with sodium hydroxide and ethylamine had lower crystallinity, the strength of these pulps was not higher than that of untreated recycled pulp. This unusual behavior could be brought about by other phenomena, such as the curling of the fibers and the deposition of solids onto the fibers.

Table 1: Crystallinity Properties of Unrecycled and Different Recycled Pulps

Sample	Crystallinity (%)	Crystallite size (nm)
Unrecycled pulp	51.2	4.1
First recycled pulp	-	-
Second recycled pulp	-	-
Third recycled pulp	57.9	4.9
First recycled with NaOH	52.7	4.3
Second recycled with NaOH	52.9	4.1
Third recycled with NaOH	52.2	4.9
First recycled with Ethylamine	53.7	3.9
Second recycled with Ethylamine	54.1	4.1
Third recycled with Ethylamine	54.9	4.5

Sheet Density

The recycled handsheets exhibited a decrease in density, as shown in Table 2. The most remarkable change in density occurred in step 1 of recycling the untreated and treated pulp with sodium hydroxide. There was no significant change during steps 2 and 3. But, the handsheets that were first recycled with ethylamine had a high density compared to other recycled handsheets, which then decreased significantly in subsequent steps. Wistara and Young (1999) showed that the apparent density of recycled kraft pulps (softwoods and hardwoods) treated with aqueous bases is significantly lower than that of the never dried pulp.

Effect on WRV

When the handsheets of bleached soda pulp were recycled in water with and without chemical treatment, significant losses in WRV occurred (Table 2). In comparison with untreated recycled pulp, treated recycled pulp with sodium hydroxide had lower WRV. It seems that swelling caused by sodium hydroxide can increase the possibility of internal bonding within the fiber cell wall during each cycle of recycling. The recycled pulps with ethylamine had greater WRV compared to treated pulps with sodium hydroxide. There was no significant difference relative to untreated recycled pulp. Similar results have been found by other researchers, to the effect that dried chemical pulp has a lower WRV in comparison with the never-dried chemical pulp (Gurnagul 1995; Wistara and Young 1999). A correlation between the WRV and fiber flexibility has been reported for chemical pulp (Brancato 2008). Gurnagul (1995) found that alkali treatment (repulping in the presence of NaOH) had only a small effect on the swelling of the bleached kraft pulp. The WRV of the treated pulps with organic solvent was significantly lower than that of never dried untreated pulps (Gurnagul 1995).

Table 2: Strength Properties of Unrecycled and Different Recycled Handsheets

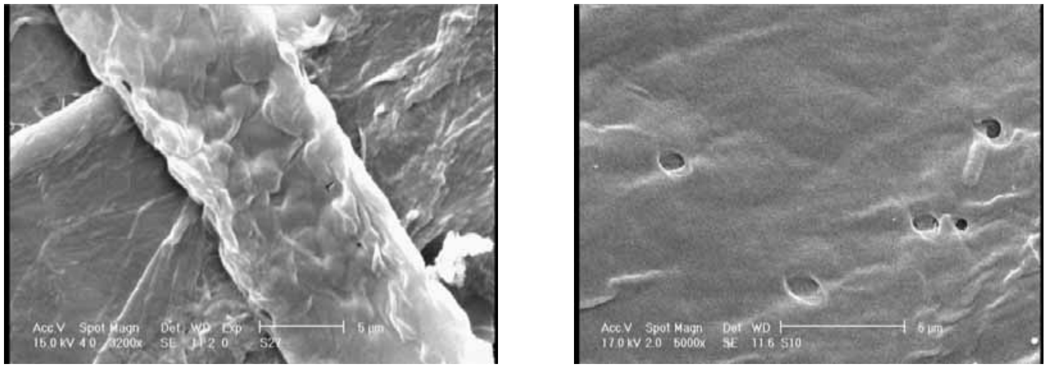
Sample	WRV (%)	Apparent density (g/cm^3)	Breaking length (km)	Burst index ($kPa.m^2/g$)	Tear index (mNm^2/g)	Number of double folds
Unrecycled (Virgin) pulp	242.7	0.632	6.84	2.754	4.567	361
Recycled pulp (Step 1)	197.7	0.593	5.02	1.93	4.36	65
Recycled pulp (Step 2)	161.3	0.595	4.2	1.43	4.58	31
Recycled pulp (Step 3)	144.4	0.573	3.65	1.2	4.36	13
Recycled pulp with NaOH (step 1)	187	0.6	4.92	1.85	4.93	55
Recycled pulp with NaOH (step 2)	154	0.59	4.33	1.52	4.43	17
Recycled pulp with NaOH (step 3)	142	0.59	3.57	1.28	4.71	28
Recycled pulp with Ethylamine (step 1)	207	0.61	5.29	1.87	4.89	71
Recycled pulp with Ethylamine (step 2)	172	0.57	3.6	1.27	4.87	16
Recycled pulp with Ethylamine (step 3)	150	0.52	2.23	0.63	3.59	4

Effect on Strength Properties

Table 2 reveals that most of the recycled pulps didn't have significant change in tear index during three steps. However, the tear index of handsheets treated with ethylamine significantly decreased during step 3. Wistara and Young (1999) showed that tear strength of treated pulps was lower than that of untreated twice recycled pulps. The influence of recycling on the breaking length and burst index is listed in Table 2. Both of them decreased significantly in every step, and the rate of decreasing in recycling with ethylamine was more at step 2 and 3.

The most generally recognized property losses in paper recycling are those associated with inter-fiber bonding, such as breaking length and burst index. Inter-fiber bonding consists primarily of hydrogen bonds between the surface carbohydrate macromolecules of neighboring fibers (Brancato 2008). Loss of inter-fiber bonding is observed in fibers that have been thoroughly dried. The loss is attributed to collapse of polysaccharide macromolecules onto each other as a result of dehydration. This general process has been termed hornification. As a result, handsheet strength decreases after drying. It can be observed that fibers pores had closed and collapsed during drying (Fig. 6). The main consequence of this change is a reduced bonding ability between the fibers.

Reducing bonding has been described as irreversible hornification (Brancato 2008; Hubbe et al. 2007).



(a) (b)

Figure 6. SEM image under different magnifications (a) collapsed fiber 3200X (b) closed pores 5000X

Lignin and hemicellulose have been removed in low yield pulps, namely those in which the final yields are about 40-55% (Cao et al. 1998). In this research, bagasse pulp yield was 45%. This relatively low yield implies that hemicellulose content was low. Hubbe et al. (2008) noted that the recycled sheets formed from fibers having low hemicellulose contents tend to be much reduced in strength. Changes in chemical composition, especially hemicellulose, of dried pulp might lead to the loss of its strength. Since it is well known that hemicellulose is beneficial to the strength development of paper, the loss of hemicellulose should be responsible for the loss of inter-fiber bonding after recycling (Cao 1998). For example, numerous studies showed a decrease in breaking length when low-yield chemical pulp was recycled (Gurnagul 1995; Jahan 203; Wistara and Young 1999). Jahan (2003) observed a loss of breaking length of about 50% after repetitively recycling five times. The strength loss of bagasse pulp is similar to that of softwood and hardwood pulps (Garg and Singh 2004). Wistara and Young (1999) concluded that treatment of recycled bleached kraft pulps with the aqueous bases does not have a beneficial effect on the tensile strength. The fold number also decreased sharply at the step 1, and the results did not differ significantly from the control. The decrease at step 2 was significant again (Table 2). The results showed that the flexibility of the handsheets decreased. Jahan (2003) observed that the double fold numbers for jute and cotton stalk pulps declined from 1481 to 156 and 503 to 70, respectively, during five successive recycling steps.

The chemical treatments were selected to maximize the swelling effects on cellulose to improve bonding ability. The fact that treatment did not alter and even reduced the strength properties, such as breaking length and burst index, seems to indicate that other factors are important to the bonding of recycled fibers. There is a possibility that traces of solids are left in the pulps, which then function as filler in the handsheets, inhibit bonding, and decrease the strength properties (Wistara and Young 1999).

Effect on Optical Properties

Table 3 shows the effect of recycling on the handsheet optical properties. In the case of untreated sheets the light scattering coefficient and opacity increased upon the first recycling and didn't change during the second and third recycling. Brightness didn't change upon recycling of the untreated sheets. All of the optical properties increased in recycling with chemical treatment, and the greatest gains were observed after 3 times recycling with ethylamine. Decreasing interfiber bonding was indicated by the increased light scattering coefficient and opacity (Gurnagul 1995; Talaeipoor and Imani 2008). The scattering coefficient is related to the area of bonding between the fibers, and it is thus generally a measure of the degree of bonding. The light scattering coefficient varies inversely with all the individual mechanical properties (Gurnagul 1995). Decreasing in strength properties can be attributed to loss of inter-fiber bonding, as indicated by the increasing light-scattering coefficient. With decrease of density and breaking length, light scattering coefficient increased, as shown in Tables 2 and 3.

Table 3. Optical Properties of the Unrecycled and Different Recycled Pulps

Sample	Brightness (%)	Opacity (%)	Light Scattering coefficient (m^2/k)
Unrecycled (virgin) pulp	69.16	77.5	29.8
Recycled pulp (Step 1)	69.24	81.3	34.35
Recycled pulp (Step 2)	68.89	81.55	33.51
Recycled pulp (Step 3)	69.02	81.3	34.33
Recycled pulp with NaOH (step 1)	69.1	85.02	39.06
Recycled pulp with NaOH (step 2)	72.09	85.58	43.41
Recycled pulp with NaOH (step 3)	71.48	83.95	41.56
Recycled pulp with Ethylamine (step 1)	68.33	84.07	36.27
Recycled pulp with Ethylamine (step 2)	69.9	84.15	40.67
Recycled pulp with Ethylamine (step 3)	72.32	88.65	51.32

CONCLUSIONS

1. SEM images indicated that the surfaces pulp fibers that were recycled with sodium hydroxide or ethylamine became covered with particles. Also, curled fibers could be observed in SEM images.

2. The crystallinity of bagasse pulp was affected by recycling. The crystallinity of untreated pulp was more than that of the chemically treated recycled pulps.
3. In comparison with untreated recycled pulp, chemical treatment with sodium hydroxide decreased the WRV after recycling. The effect of ethylamine in decreasing the WRV of chemically treated recycled pulp was less than that of sodium hydroxide.
4. The tear index of the handsheets didn't change during recycling, while it decreased significantly in recycling with ethylamine at step 3.
5. Recycled paper properties such as breaking length, burst index, and fold number, as well as apparent density decreased, and light scattering coefficient and opacity increased. But, in the case of fibers treated with ethylamine, there was greater decrease observed at steps 2 and 3.
6. The brightness increased significantly with chemical treatment of recycled pulp.

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