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CHEMICAL TREATMENT OF RECYCLED PULP FIBERS FOR PROPERTY DEVELOPMENT: PART 2. EFFECTS ON OLD NEWSPAPERS

The recycling of paper is an important issue and requires a systematic and investigative approach. However, it is necessary to scrutinize alternative methods on the strength of the development of recycled fibres to explore the impact of recycling on properties of recycled fibres. In this study, it was found that repeated treatments of recovered fibres with all three chemicals usually enhance their ability to re-swell and improve bonding potential in fibre networks. This is evidenced by higher water retention values (WRV) and improved strength properties. The highest WRV value of 2.65 g/g was observed with a 10.0% ethyl acetate treatment in the third recycling stage, which indicates approximately 660% higher WRV compared with control samples (0.34 g/g). Measurements also showed that the optical properties of lightness and the brightness properties of the sheets show variations. However, the highest brightness improvement of 3.8% was observed at 5.0% ethyl acetate treatment at the second recycling stage. This level of treatment has also given the highest lightness value of 73.97, which indicates only 0.54% improvement. The maximum tensile strength value of 23.89 N·m/g was observed at the third recycling stage with 10.0% formamide charge. However, this level of treatment also gave the highest burst at 1.50 kPa·m^2/g and tear value of 2.88 N·m^2/g, which indicates a 128.2% higher burst and 176.9% higher tear strengths, compared with control samples, respectively.

Keywords: newspaper, recycling, chemical treatment, ethyl acetate, formamide, paper strength, tensile, water retention value, optical properties

Introduction

Paper-based products such as news, business, industrial, and packaging papers are among the paper products tied to a country's political and economic nature. However, the level of paper consumption has become an indication of cultural and civilization development. In recent years, environmental pressure has contributed considerably to worldwide recycling awareness, and it has driven lawmakers to make recycling mandatory in many countries. The United States is one of the leading countries in paper recycling; it is not only a consumer of

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recycled fibres but also an exporter to many European countries that lack virgin fibre sources [Cathie and Guest 1991; Biermann 1993; Göttsching and Pakarinen 2000].

In typical pulp drying, cellulose and hemicelluloses (polysaccharides) can be changed from a gel state into a hard and crusty material that will not swell in water. However, hydrogen bonds or cross-links could be formed between adjacent microfibrils in dried pulps; this specific condition is usually called hornification in the literature [Carlson and Lindstrom 1984; Ellis and Sedlachek 1993; Hubbe et al. 2007].

Minor [1994] found that recycled fibres are thinner than those of the never-dried fibres. Wistara and Young [1999] proved that dried fibres with higher initial swelling capacity have a lower capability to re-swell. It has already been reported that water molecules can hardly penetrate into microfibrils to swell them. Upon severe drying, fibres lose free and bound water, which causes them to shrink [Clark 1978; Nanko et al. 1991; Brancato 2008]. However, the reduction of swelling was not only due to a change in the fibre polymer structure. Instead, the closure of larger pores is the most likely origin of the reduction of the re-swelling ability of dried fibres. Hence, the pores do not re-open upon re-wetting due to a combination of the effects of high surface tension forces and a subsequent strain-hardening mechanism that welded the pores [Clark 1978; Wistara and Young 1999; Brancato 2008].

Many studies have explored the increased suitability of recycled fibres for papermaking. These include various techniques of beating and chemical treatment during recycling [Clark 1978; Katz et al. 1981; Wistara and Young 1999; Wistara et al. 1999; Hubbe et al. 2007; Bajpai 2014].

Chemical treatment of pulp is a means to increase and regain the bonding potential of pulp. A number of chemicals have been evaluated for recycled pulps to improve certain properties. However, the most common chemical used for improving the bonding potential of pulp is alkaline-based [Clark 1978; Katz et al. 1981; Wistara and Young 1999; Hubbe et al. 2007; Brancato 2008; Sheikhi et al. 2010; Bajpai 2014;].

Howard and Bichard [1992] found that bleached pulps (i.e., Kraft) undergo cross-linking by hydrogen bond formation upon drying, which bring about loss of swelling ability (hornification), while mechanical pulps (CTMP) show flattening and flexibilizing and unbeaten bleached pulp undergoes curling. However, it has been known that high-yield pulps (i.e., mechanical) shrink less than chemical pulp does upon drying. This is because of the presence of lignin as an incrusting material between cellulose microfibrils, which prevents direct contact among cellulose microfibrils when bound water is removed. Thus, lignin also prevents re-swelling [Bajpai 2014].

Bhat et al. [1991] proposed that alkali treatment affects higher strength properties than those of just refining; in some cases, the strength properties were comparable with those of the virgin pulp. It has been well explained that sodium hydroxide treatment improves the swelling capacity of dried mechanical and chemical pulps [Clark 1978; Katz et al. 1981; Wistara and Young 1999].

A number of researchers have proposed that recycling produces different effects on different types of pulps. Hence, chemical and mechanical pulps show different recycling abilities [Cao et al. 1998; Wistara and Young 1999; Jahan 2003; Garg and Singh 2004; Bajpai 2014]. Howard and Bichard [1992] suggested that, unlike chemical pulps, the walls of mechanical pulp fibres are not extensively delaminated in the wet state. Hornification during drying is therefore limited and may have little if any, effect on interfiber bonding. It was proposed that TMP and CTMP pulps showed some increase in sheet density and tensile strength upon recycling, but CMP exhibited a decrease in density and in tensile strength [Hubbe et al. 2007; Bajpai 2014], which indicate that different pulps show different responses during recycling.

It appears that the recycling of lignocellulosic fibres is a complex phenomenon and requires further investigation. It must be taken into consideration that the investigations on the methods of strength development of recycled fibres need to be carried out, along with a systematic approach to measure the impact of recycling on the strength reduction of recycled fibres. However, few recycling studies have been conducted on the selected chemicals and special papers (i.e., newspapers). Thereby, systematic approaches have been carried out with various types of chemicals on different waste paper substrates to determine the clear effects on recycling approaches and the chosen methods. The first part of this study, "Chemical Treatment of Recycled Pulp Fibres for Property Development: Part 1. Effects on Bleached Kraft Pulps" has already been published in Drewno [Sutcu and Sahin 2017]. In the second part of this study, we seek to provide a more fundamental understanding of the properties and strength development of recycled fibres from newspapers.

Materials and methods

Daily newspapers were supplied from a store and disintegrated in a laboratory--type blender as per standard procedure. The pulps were refined to a PFI mill at the freeness of 400-450 ml (CSF). The refined pulps were made into hand sheets following TAPPI standard procedures. The technical properties of newspaper used in the recycling studies are supplied by a newspaper company, as shown in table 1.

Chemical treatments were utilized after the disintegration of pulps (recycling) and were designed to mainly increase the pulp's swelling capacity. Ethyl acetate, formamide, and sodium hydroxide were selected for the chemical treatment of recycled fibres from newspapers to study the effects on swelling and bonding (strength) of fibres during recycling. All chemicals, with a purity of 99.9% (unless otherwise noted), were purchased from a chemical company. The chemical treatments were selected to maximize the swelling effects on cellulose to improve bonding ability. The detailed selection criteria of the chemicals, hand sheet preparation, and experimental testing approaches have been given in the first part of this study.

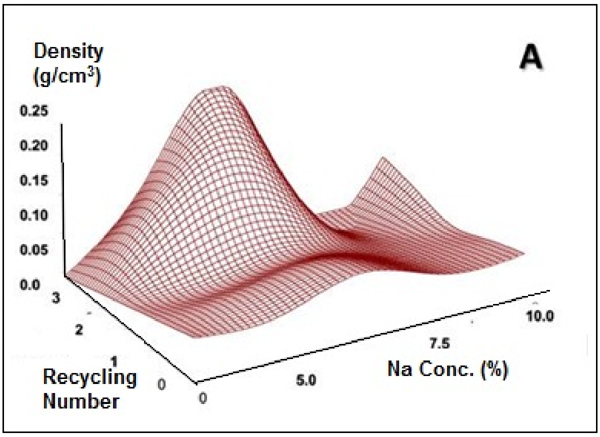
Table 1. Technical properties of newspaper

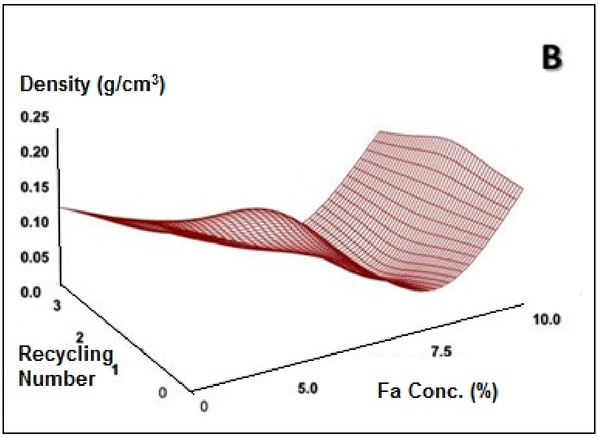
Property	Value
Grammage	45 g/m^2 (±2)
Bulk	1.55-1.60 cm^3/g
Density	0.65 g/cm^2
Air permeability	300 ml/min (min.)
Brightness (ISO)	60% (min.)
Opacity	92% (min.)
Surface properties (Bendsten)	70-200 ml/min
CIE, L*	83.7
CIE, a*	-0.45
CIE, b*	5.1

Results and discussion

Figures 1 and 2 show the comparative sheet density and water retention values (WRV) against chemical concentration and recycling numbers. It can be seen that sodium hydroxide has an increasing effect at the first recycling stage and low chemical charge (fig. 1A). Nevertheless, formamide treatments usually lower the density of sheets up to 7.5% chemical charge and then increase sheet density (fig. 1B). However, no noticeable correlation was found with ethyl acetate treatment (fig. 1C). The highest sheet density of 0.32 g/cm^3 was observed at the third recycle stage with 5.0% NaOH treated sheets (5Na_3), which indicates a 28% higher sheet density at similar recycling conditions of untreated sheets (N_3: 0.25 g/cm^3). It was speculated that the reason for the increase in density of recycled mechanical pulps is probably due to the progressive flattening and flexibilizing of the stiff, uncollapsed fibres during the recycling stages. However, the more flexible fibres bond better and give a thinner, denser sheet due to the recycled fibres containing lignin binding the cell wall matrix.

However, it was also proposed that the fibre walls of mechanical pulps do not delaminate extensively, even if mildly refined at low consistency, so recycling would not be expected to cause a significant reduction in swelling [Howard and Bichard 1992; Bajpai 2014]. In contrast, we found that repeated recycling effects considerably lower WRV values of fibres. At first, recycled fibres WRV value of 0.88 g/g was found. However, at the second and third





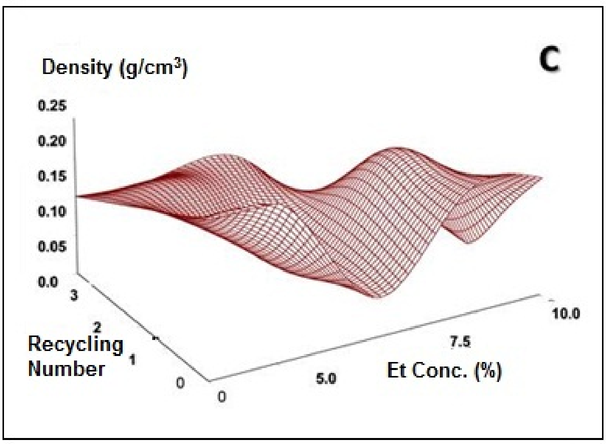


Fig. 1. Comparative sheet density against chemical concentration and recycling number

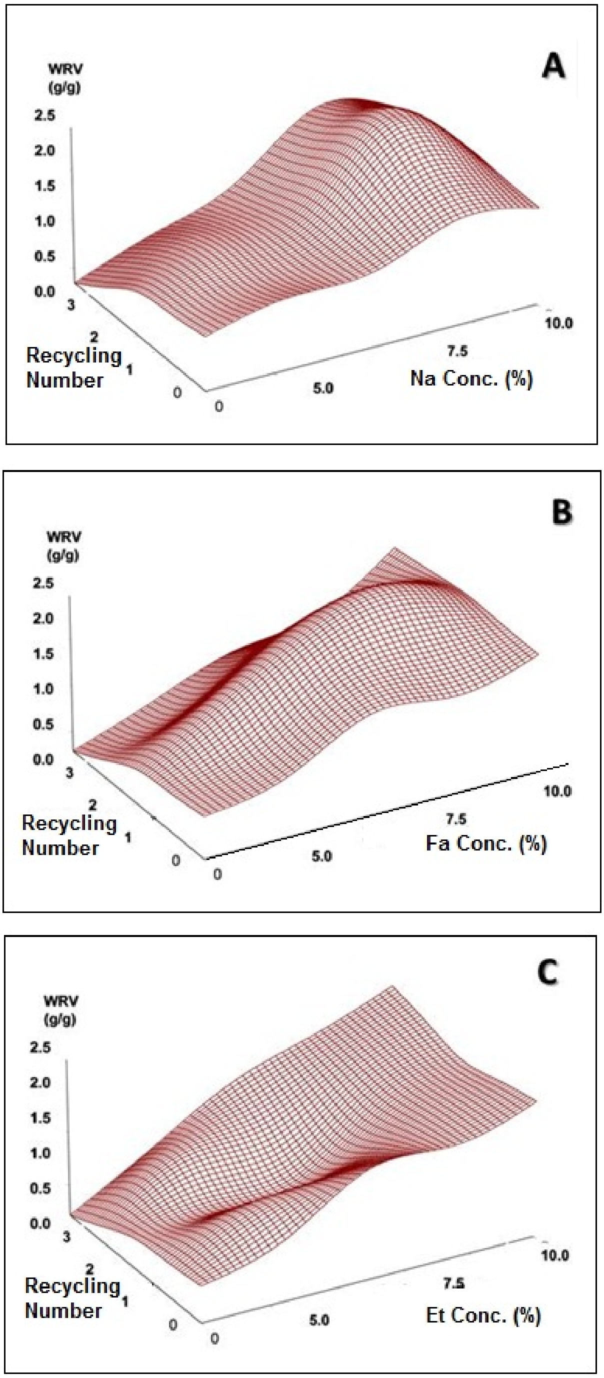


Fig. 2. Water retention values (WRV) against chemical concentration and recycling number

recycling stages, 0.78 and 0.34 g/g values indicate 11.3% and 61.3% reduction of WRV according to the first recycled fibres, respectively.

It was also observed that all three chemical treatments usually positively affect WRV values at a similar recycling stage compared with control samples. However, formamide and ethyl acetate have a tendency to improve effects on all three-recycling stages (figs. 2B and C), whereas sodium hydroxide has improved trends up to the second recycling stage and a 7.5% chemical charge level; then, further chemical charges and recycling affects some level lowering (fig. 2A).

The highest WRV value of 2.65 g/g was observed with a 10.0% ethyl acetate treatment at the third recycling stage ($10Et_3$), which indicates 660% higher WRV compared with control samples (N_3: 0.34 g/g). A number of researchers have predicted that cellulose fibres having chemical treatments could influence cellulose-water interactions at some level [Clark 1978; Hubbe et al. 2007; Bajpai 2014]. However, mechanical pulp is considerably different from chemical pulp, so the recycling effect on those fibres is also different. When chemical fibres undergo repeated drying and rewetting, they are hardened (hornified) and can significantly lose their high bonding potential. As previously mentioned, the recycling of untreated pulp significantly reduced water retention value. The loss of WRV could be due to increased hornification of the cellulose fibres and/or loss of hemicelluloses with recycling. The treatment of recycled pulps with solvents increases the WRVs. It is assumed that the chemical treatment might influence the cellulose re-swelling properties to some degree in addition to affecting the physical characteristics (density) of the sheets. It can be hypothesized that short-chain carbohydrates (hemicelluloses) could be soluble at the high alkali concentration (10%) that typically occurred (i.e., a "peeling" reaction) [Clark 1978; Smook 1994; Scott and Abbott 1995]. The results found in our study with sodium hydroxide and other chemical treatment support this information.

Table 2 lists the comparative air permeability and optical properties of paper samples produced from recycled newspaper fibres. However, it appears that the chemical treatments usually have an effect on lowering lightness and brightness properties of the sheets, except in a few conditions. The optical properties of lightness and brightness of sheets presented a variation in the recycled sheets. The highest brightness improvement was 3.8% according to control (N_2), which was observed at 5.0% ethyl acetate treatment at the second recycling stage ($5Et_2$). This level of treatment has also given the highest lightness value of 73.97, which indicates only a 0.54% improvement. However, marginally similar 1-3% reducing lightness values were observed with all three chemical treatments.

It can be seen that there is no direct correlation between chemical treatment and recycling stages for the air permeability properties of sheets. The highest air permeability value of 38.7 (m^2/sec) was found at the third recycling stage of 5.0% formamide treated samples ($5Fa_3$). Moreover, no direct correlation was observed for air permeability and optical properties of the sheets either. This is expected, considering the complicated phenomena that occur during the recycling of lignocellulosic fibres.

Table 2. Physical and optical properties of chemically treated recycled pulps

Trt.	Air Perm. (m^2/sec)	Change accor. to (N_{1-3}, %)	Lightness (%)	Change accor. to (N_{1-3}, %)	Brightness (%)	Change accor. to (N_{1-3}, %)
N_1	21.1	0	73.90	0	42.49	0
N_2	22.1	0	73.57	0	41.22	0
N_3	22.4	0	75.25	0	42.71	0
$5Na_1$	15.2	-27.9	71.30	-3.5	37.97	-10.6
$5Na_2$	15.2	-31.2	72.36	-1.6	38.37	-6.9
$5Na_3$	17.5	-21.8	74.48	-1.0	41.21	-3.5
$7.5Na_1$	8.49	-59.8	72.45	-1.9	38.74	-8.8
$7.5Na_2$	14.6	-33.9	73.79	0.3	39.94	-3.1
$7.5Na_3$	18.2	-18.8	75.87	0.8	41.23	-3.5
$10Na_1$	10.7	-49.3	71.42	-3.4	37.13	-12.6
$10Na_2$	13.4	-39.3	73.67	0.14	38.94	-5.5
$10Na_3$	15.7	-29.9	74.82	-0.57	40.03	-6.3
$5Fa_1$	14.9	-29.4	72.36	-2.1	40.49	-4.7
$5Fa_2$	28.4	28.5	73.80	0.31	41.52	0.73
$5Fa_3$	38.7	72.8	75.25	0.0	42.93	0.52
$7.5Fa_1$	21.2	-0.5	73.75	-0.2	42.42	-0.19
$7.5Fa_2$	22.2	-0.5	74.19	0.84	41.85	1.5
$7.5Fa_3$	20.4	-8.9	75.84	0.78	43.43	1.7
$10Fa_1$	16.9	-19.9	73.13	-1.04	41.39	-2.6
$10Fa_2$	14.6	-33.9	73.30	-0.37	41.17	-0.12
$10Fa_3$	19.4	-13.4	75.17	-0.11	42.94	0.53
$5Et_1$	18.8	-10.9	71.65	-3.04	38.75	-8.8
$5Et_2$	27.9	26.2	73.97	0.54	42.78	3.8
$5Et_3$	11.3	-49.6	73.82	-1.90	41.49	-2.9
$7.5Et_1$	17.6	-16.6	74.17	0.37	43.53	2.4
$7.5Et_2$	14.3	-35.3	73.87	0.41	41.96	1.8
$7.5Et_3$	17.3	-22.8	74.55	-0.93	41.09	-3.8
$10Et_1$	13.9	-34.1	71.37	-3.42	38.96	-8.3
$10Et_2$	13.9	-37.1	72.94	-0.86	40.60	-1.5
$10Et_3$	22.3	-0.4	73.96	-1.71	41.53	-2.8

N: control, 1-3: recycling stage; Na: Sodium hydroxide treatment; Fa: Formamide treatment; Et: Ethyl acetate treatment; 5, 7.5, 10: % chemical charge (based on oven dry pulp).

The comparative mechanical strength properties of sheets manufactured from recycled fibres with statistical results are given in table 3. They show that all three strength properties of the sheets decreased at some level as the recycling stage increased. It is likely that the cell wall structure undergoes modification in the recycling stages and negatively affects the bonding potential of the fibres. These results are in agreement with the results reported on strength properties for various types of paper by Bajpai [2014] and Hubbe et al. [2007]. However, the chosen chemical treatments have typically improved the strength of the paper rather than control samples at a similar recycling stage. In general, all three chemicals were affected by stopping strength, which decreased at some levels.

Table 3. Strength properties of chemically treated recycled pulps

Trt.	Tensile Index (N·m/g)	Change accor. to (N_{1-3}, %)	Burst Index (kPa·m^2/g)	Change accor. to (N_{1-3}, %)	Tear Index (N·m^2/g)	Change accor. to (N_{1-3}, %)
N_1	19.79	0	1.31	0	1.98	0
N_2	13.99	0	1.09	0	1.33	0
N_3	10.47	0	0.92	0	1.04	0
$5Na_1$	13.91	-45.5	0.90	-31.3	1.32	-33.3
$5Na_2$	15.93	13.9	0.93	-14.7	1.79	34.6
$5Na_3$	14.84	41.7	0.80	-13.1	1.41	35.6
$7.5Na_1$	19.14	-3.2	1.08	-17.6	1.48	-25.3
$7.5Na_2$	16.84	20.4	0.92	-15.6	1.50	12.8
$7.5Na_3$	19.08	93.3	0.92	0	1.71	64.4
$10Na_1$	15.44	-21.9	1.08	-17.6	1.68	-15.1
$10Na_2$	14.36	2,6	0.93	-14.7	1.42	6.8
$10Na_3$	14.08	34.5	0.89	-3.2	1.35	29.8
$5Fa_1$	12.66	-36.0	0.95	-27.5	1.36	-31.3
$5Fa_2$	13.04	-6.8	0.97	-11.1	1.52	14.3
$5Fa_3$	15.07	50.9	1.03	11.9	2.06	98.1
$7.5Fa_1$	14.34	-27.6	1.01	-22.9	1.65	-16.6
$7.5Fa_2$	13.44	-3.9	1.03	-5.6	2.24	68.4
$7.5Fa_3$	22.32	113.2	1.44	56.5	2.66	155.8
$10Fa_1$	16.56	-16.3	0.98	-25.2	2.69	35.9
$10Fa_2$	16.42	17.4	1.07	-1.8	2.38	78.9
$10Fa_3$	23.89	128.2	1.50	63.1	2.88	176.9
$5Et_1$	15.69	-21.3	0.98	-25.2	1.53	-22.7
$5Et_2$	19.06	36.2	0.98	-10.1	1.84	38.3
$5Et_3$	15.75	50.4	1.05	14.1	1.56	50.0
$7.5Et_1$	18.69	-5.6	1.06	-19.1	1.29	-34.8
$7.5Et_2$	19.05	36.2	1.11	1.8	1.46	9.8
$7.5Et_3$	20.60	96.8	1.15	25.0	1.33	27.9
$10Et_1$	14.63	-26.0	0.93	-29.0	1.55	-21.7
$10Et_2$	14.53	3.9	1.03	-5.6	1.12	-15.8
$10Et_3$	16.25	55.2	1.00	10.0	0.91	-12.5

N: control, 1-3: recycling stage; Na: Sodium hydroxide treatment; Fa: Formamide treatment; Et: Ethyl acetate treatment; 5, 7.5, 10: % chemical charge (based on oven dry pulp).

For the sodium hydroxide treatment, the maximum tensile and tear strengths values of 19.8 N·m/g and 1.71 N·m^2/g were found at 7.5% chemical charge in the third recycling stage, which indicates a 93.3% higher tensile and 64.4% higher tear strengths according to control samples. However, this level also gave a burst strength value of 0.92 kPa·m^2/g, which is similar to that of the control samples (stopping burst decrease). In all other conditions, the burst strength was decreased at 3.2% to 31.3%. But this is not expected because tensile and burst strengths have close relations rather than tear strengths. However, tensile and burst strengths of paper are closely related to single fibre strength, but primarily on the degree of bonding amongst the fibres network. Although a number of researchers have already reported a definite relationship between tensile and burst strengths of paper [Fernandez and Young 1996; Wistara and Young 1999; Wistara et al. 1999; Bajpai 2014], this information may be partly true only for other types of paper as realized in this study.

For formamide treatment, it was observed that, except for the second recycling stage of 10.0% ($10Fa_2$), 17.4% higher tensile strength, all other first and second recycling stages had no improved effects on the tensile strengths of the paper. However, it is also realized that the third recycling stage showed 50.4-128.2% improvement for all formamide treatments. The highest tensile strength value of 23.89 N·m/g was observed at the third recycling stage of 10.0% chemical charge ($10Fa_3$), which indicated a 128.2% improvement according to the control (N_3). However, this level of treatment also gave the highest burst at 1.50 kPa·m^2/g and a tear value of 2.88 Nm^2/g, which indicated a 128.2% higher burst and 176.9% higher tear strengths according to control samples. The results clearly indicate that all the strength properties of sheets are positively affected by formamide treatment at certain conditions.

For ethyl acetate treatment, similar results were found for tensile and burst strengths of sheets. The highest tensile strength of 20.60 N·m/g (96.8% improvement) and burst strength value of 25.0 kPa·m^2/g (25.0% improvement) were found at the third recycling stage of 7.5% chemical charge, while the highest tear strength improvement (50.0%) was found at the third recycling stage, but with 5.0% chemical charge conditions.

It appears that repeated treatments of fibres with ethyl acetate enhance their ability to re-swell and improve bonding potential in the fibre network. This is also evidenced by the higher WRV of fibres after ethyl acetate treatments (fig. 2C).

The dependence of the three important strength properties as a function of the chemical charge (%) and recycling number are shown in figure 3 (tensile strength), figure 4 (burst strength), and figure 5 (tear strength), respectively.

For tensile strength, some level of improvement was observed at the third recycling stage and up to 5.0% concentration with sodium hydroxide treatment (fig. 3A). However, in most cases, the trend increased tensile strength after the

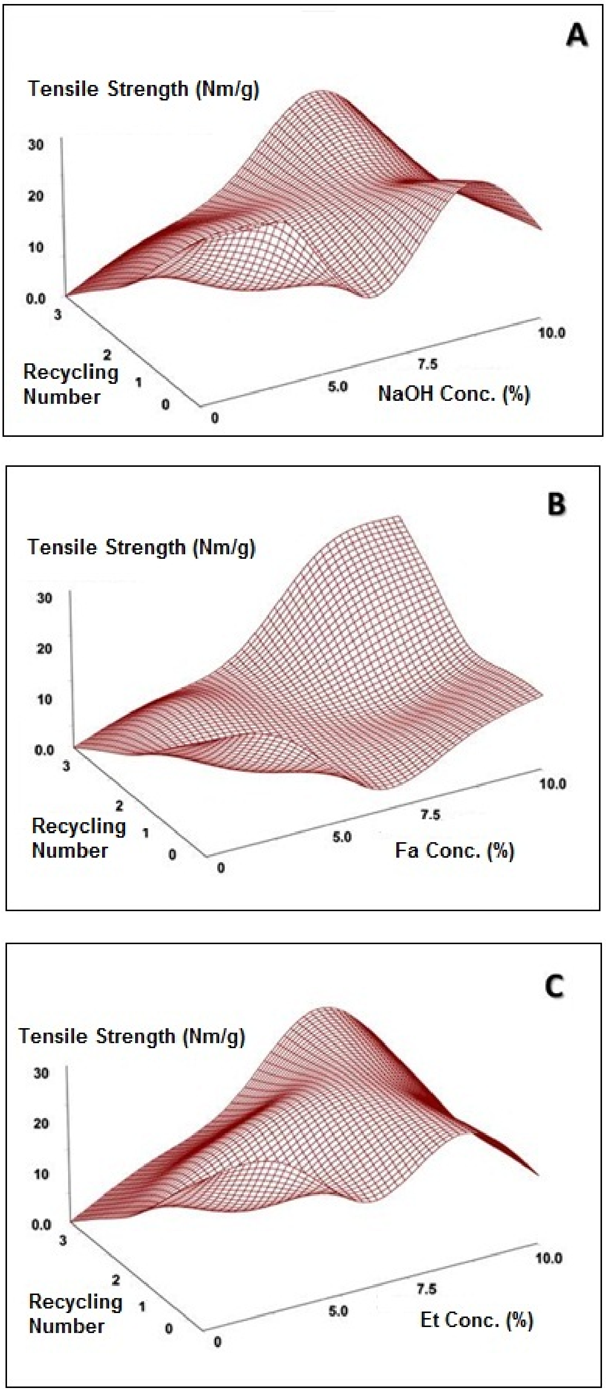


Fig. 3. Tensile strength of recycled papers

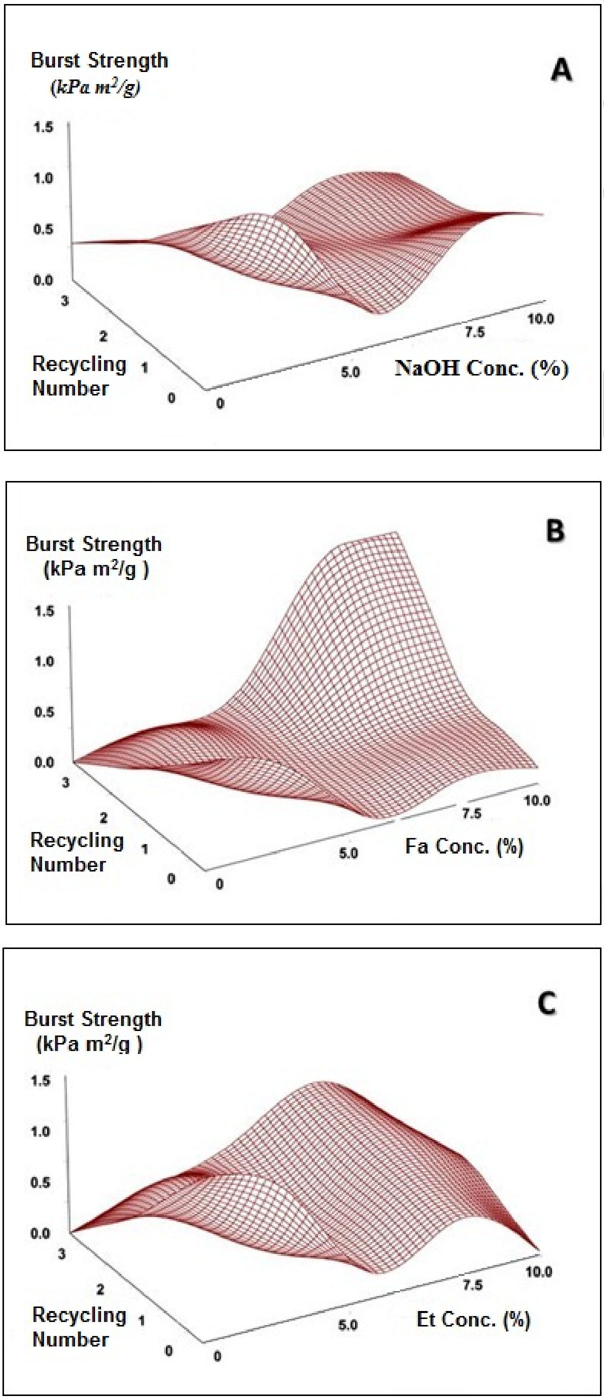


Fig. 4. Burst strength of recycled papers

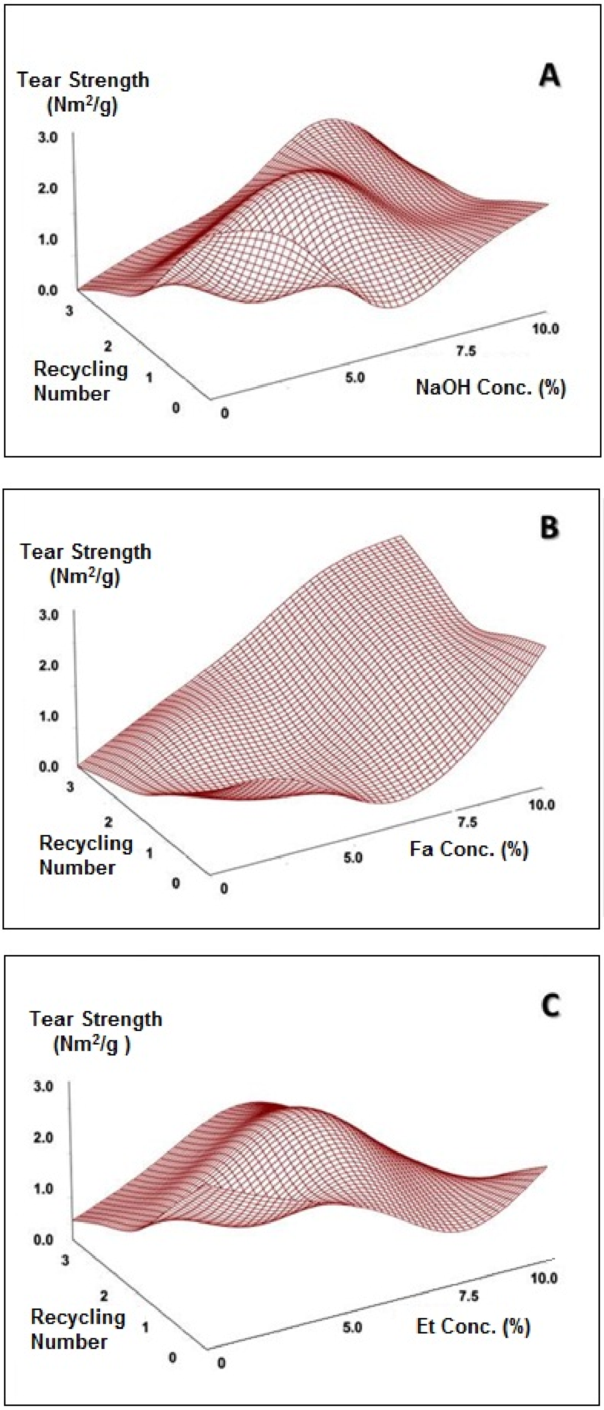


Fig. 5. Tear strengths of recycled papers

second recycling at 10% formamide charge (fig. 3B). Moreover, an improvement was observed up to 7.5% ethyl acetate concentration with an increasing recycling number, but further ethyl acetate concentration (10%) usually lowered effects on tensile strength (fig. 3C). These findings clearly suggest that tensile strength improvement is possible at optimum treatment conditions. Hence, a different mechanism should control the increased or decreased tensile strengths. It can be attributed to the re-orientation of microfibrils and a better alignment of fibres in a sheet network structure that promote additional fibre bonding sites at certain treatment conditions.

For burst strengths, more or less similar figure shapes (trends) observed that some level of improvement of burst strength is possible for all three chemicals, but at specific treatment conditions (fig. 4A-C). Numerous studies have reported that tensile strength is, in general, linearly correlated with burst strength. Comparing the burst and tensile strength, it can be seen that all chemicals demonstrate a similar trend for recovered fibres. The fact that the treatments were altered and even increased the strength properties, such as burst and tensile, seems to indicate that other factors are important to the bonding of recycled fibres, while increases in tear strength are usually reported.

However, considerably different results were observed for tear strengths of the paper (fig. 5A-C) compared with figures 3 and 4. It can be seen that a sodium hydroxide concentration of up to 7.5% concentration at first and second recycling stages have primarily positive effects on improving tear strengths of paper (fig. 5A). A similar trend was observed for ethyl acetate treatment conditions (fig. 5C). However, in all formamide treatment conditions, tear strengths of sheets improved at some level. It is clear that formamide shows a marked effect on improving the tear strength of sheets. A number of studies have shown that tensile strength usually behaves contrary to tear strength. This behaviour is also shown by the treatment of recycled fibres with sodium hydroxide and ethyl acetate treatment in this study. Fernandez and Young [1996] proposed that tear strength has a complicated effect in terms of fibre properties.

Conclusions

The recycling of paper-based products is becoming an important issue for a sustainable environment and economic conditions. However, secondary pulp (recycled) has also become an important raw material for many papermaking grades around the world. Many newsprint and tissue grades commonly contain various levels of secondary pulp. Intensive scientific research has already been conducted to reveal the benefits of recycled paper.

It has been well explained that some irreversible changes occur during the recycling of cellulosic fibres. However, in understanding these changes, it is possible to better utilize recycled fibres in the papermaking industry. The results of this study indicate that all the liquids used in this study have improved effects on re-swelling properties, i.e., the bonding potential of fibres. The addition of organic solvents (formamide and ethyl acetate) during recycling has been found to offer advantages that are important for strength improvement compared to untreated samples. The addition of those chemicals resulted in increased water retention value (re-swelling) and strength properties.

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