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H. Turgut Sahin, Abdullah Sutcu, Ahmet Tutus

CHEMICAL TREATMENT OF RECYCLED PULP FIBRES FOR PROPERTY DEVELOPMENT: PART 3. EFFECTS ON OCC PULPS

Chemical treatment with sodium hydroxide, formamide and ethyl acetate is shown to affect certain properties of paper made from recovered fibres. Sodium hydroxide treatment improves the brightness of sheets by approx. 0.12–17.2% under similar recycling conditions. All chemical treatments usually improve the tensile and burst strengths of sheets to some degree. The highest tensile and burst strength values of 42.09 Nm/g and 2.60 kPa m^2*/g were obtained at the first recycling stage with 10% ethyl acetate treatment (*$O10Et_1$*), and these represent approximately 142.2% and 100% improvement of tensile and burst strength respectively. In contrast, although the results revealed that certain chemical treatments markedly improved both tensile and burst strengths, there is some variation observed for tear strengths. The largest improvement in tear strength (72.9%) was found at the third recycling stage with 5.0% formamide treatment (*$O5Fa_3$*), followed by* $O10Et_3$ *(33.9%) and* $O10Na_3$ *(29.2%). It is important to note that the highest tear strength value of 9.09 N*m^2*/g was found at the second recycling stage of the control samples. The results clearly show that the tensile and burst strengths of sheets can be improved by certain chemical treatments, but there is no correlation with tear strengths.*

Keywords: Old Corrugated Container, recycling, chemical treatment, fibre, paper properties, tensile strength, burst strength

Introduction

The pulp and paper industry is one of the world's most energy-intensive industries. Depending on technology and end products, it requires approximately 5000 kW of electrical energy and 400 tonnes of water to produce each tonne of paper products. However, it also has huge effects on the environment. Among others, the use and processing of lignocellulosic materials have significant negative effects on ecology and the natural life cycle [Biermann 1993]. At the present time, worldwide paper and paperboard production totals around

H. Turgut Sahin✉ (*halilsahin@isparta.edu.tr*), Abdullah Sutcu (*abdullahsutcu@isparta.edu.tr*), Department. of Forest Products Engineering, Isparta University of Applied Sciences, Isparta, Turkey; Ahmet Tutus (*atutus@ksu.edu.tr*), Department of Forest Products Engineering, Kahramanmaraş Sütçü İmam University, Kahramanmaraş, Turkey

400 million tonnes, and is expected to reach 500 million tonnes by 2020 [Bajpai 2014].

One of the main benefits of recycling is the reduction of environmental impact, with economic advantages. It is already well known that paper recycling has many advantages in terms of protecting forestlands and reducing investment, operational and energy costs to the papermaking industry. Hence, the utilization of waste paper (recycling) has become a rapidly growing industry worldwide [Sahin 2014, 2016; Twede et al. 2015].

The hydrophilic nature of the fibre surface is very important for fibre–fibre bonding. However, fibre strength, fibre length, fibre swelling, and bonding potential are some of the important parameters for the strength properties of paper made from recycled fibres [Clark 1978; Howard and Bichard 1992; Hubbe et al. 2007]. Therefore, quantification of the effects of recycling on these fibre properties may open up possibilities for prevention of strength loss. Beating or refining, chemical treatment and blending with virgin fibres are reported to be some potential ways to recover the loss of bonding potential of recycled fibres [Biermann 1993; Brancato 2008; Bajpai 2014; Sahin 2014, 2016].

Although a number of researchers have reported that sheets made from recycled fibres can be improved by refining, the recovered fibres are usually more brittle and sensitive to beating, and misapplication may cause reduced fibre length, strength and bulk, with a detrimental effect on sheet strengths [Clark 1978; Howard and Bichard 1992; Ellis and Sedlachek 1993]. However, Laivin and Scallan [1996] reported that hornified fibres are brittle and very susceptible to fragmentation during beating, and once-dried fibres can be very difficult to re--swell by refining. Moreover, Katz et al. [1981] found that the treatment of waste paper with sodium hydroxide may improve the strength properties of recycled fibre, but the degree of improvement strongly depends upon the conditions of chemical treatment. It has been reported that alkalis (such as sodium hydroxide) usually promote fibre swelling, thereby increasing fibre flexibility and surface conformability [Clark 1978; Katz et al. 1981 ; Bajpai 2014]. Wistara [1997] and Bhatt et al. [1991] and reported that recycling under alkaline conditions could be used to improve the strength of secondary fibres. De Ruvo et al. [1986] studied this as a means of improving strength properties in OCC recycled pulp. Alkali treatment was found to improve bonding and strength characteristics, probably because of softening and improved re-swelling properties of the fibres. Freeland and Hrutfiord [1993] reported that soaking of old corrugated containers (OCC) in sodium hydroxide before the recycling process results in dramatically reduced re-pulping and refining energy for specified strength requirements. However, it is important to note that swelling of the cell wall continues until the osmotic pressure differential is balanced by the residual cohesive forces of the cell wall.

The use of recycled fibres in low or medium grades such as newsprint, packaging or OCC does not cause noticeable deterioration in product quality and performance. However, the increase in recovery rates of used paper products will require an understanding of the fundamental nature of recycled fibres and the differences from virgin fibres. It was expected that a study on the recycling of OCC papers with certain chemical treatments would lead to more desirable paper products with possible improved strength properties.

It is well known that the recycling of paper-based products is a very complex phenomenon. However, approaches to the quality development of secondary fibres need to be established, along with a systematic method to measure the impact of recycling and recovery on the quality of secondary (recycled) fibres. Some investigations have already been conducted on certain chemicals and special types of paper (e.g. newsprint). A systematic investigation has been carried out with selected types of chemicals on different waste paper and paperboard (OCC) substrates to determine the effects of recycling approaches and the chosen methods. The first and second parts of this study, "Chemical Treatment of Recycled Pulp Fibres for Property Development: Part 1. Effects on Bleached Kraft Pulps" and "Chemical Treatment of Recycled Pulp Fibres for Property Development: Part 2. Effects on Old Newspapers" were published in the journal *Drewno* in 2017 (Vol. 60, No. 200, pp. 95-109) and 2018 (Vol. 61, No. 201, pp. 177-192) respectively. In the third part of this study, we seek to demonstrate clear effects on the properties and strength of recycled fibres from OCC.

Materials and methods

Used old corrugated container (OCC) boxes were supplied by a store and disintegrated (re-pulped) in a laboratory-type blender as standard procedure. The pulps were refined with a PFI mill at a freeness of 400-450 ml (CSF). The refined pulps were made into hand sheets following TAPPI standard procedures [TAPPI T205 and T220]. Physical properties of the pulp such as burst, tear and tensile strengths, and the brightness and air permeability of the paper, were determined with TAPPI test methods [TAPPI T403; T414; T460; T494 and T525]. Water retention value (WRV) was measured over a range of centrifugation conditions, including the condition specified in the Scandinavian test method SCAN-C 62:00.

The chemical treatments were applied after disintegration of pulps (recycling) and were designed mainly to increase the swelling capacity of the pulp. Ethyl acetate, formamide and sodium hydroxide were selected for chemical treatment of recycled fibres from OCC to study effects on the swelling and bonding (strength) of fibres during recycling. The detailed selection criteria for chemicals, hand sheet preparation and experimental testing approaches are given in the first and second parts of this study.

Fibre fragmentation was conducted in a Bauer McNett fibre classifier. Six different screens were used to classify fibres (16, 30, 50, 100, 200, >200 mesh). The fibres were carefully collected on the screens and then dried in an oven at

105 (±5)°C. After drying the fibre fractions were weighed and each class of fibre determined. This procedure was repeated three times and an average fibre class was calculated. The fibre fragmentation of recovered OCC pulps is shown in Table 1.

Table 1. Fibre classification in Bauer McNett

Screen openings (mesh)	1st classif. (g)	2nd classif. (g)	3rd classif. (g)	Average (g)
16	6.105	6.304	6.501	6.303
30	1.465	1.590	1.340	1.465
50	1.002	1.025	1.095	1.041
100	0.163	0.181	0.155	0.166
200	0.542	0.432	0.554	0.509
< 200	0.268	0.249	0.241	0.253

Results and discussion

A comparison of physical properties of sheets made from recovered fibres is given in Table 2. For the control samples it was found that the densities of sheets increased as the recycling stage number increased. However, the chemical treatments had clear effects at the first and second recycling stages: the sheet densities remained similar (did not decrease or increased) to some degree. It is important to note that all three chemical treatments had lowering effects at the third recycling stage. This is probably because the outermost layers of the fibres (fines) could be removed during recycling. According to Scott and Abbott [1995], fines are very important for the properties in mechanical pulp, and to some extent in chemical pulp. Fines may assist inter-fibre bonding, thus increasing density to some degree. The results obtained for chemical treatment at the first and second stages clearly support this finding.

However, in contrast to the control samples, where Water Retention Values (WRV) decreased as recycling continued, the chemically treated recovered fibres exhibited increasing values in all chemical charge conditions and recycling stages. The highest WRV value of 3.02 g/g was observed at the third recycling stage under formamide treatment ($O7.5Fa_3$), this being approximately 102% higher than the value for the control (OC_3: 1.49 g/g). These findings are very significant in terms of the ability of certain chemicals to improve the re-swelling properties of secondary fibres.

As seen as Table 2, the brightness value is virtually unchanged for the control samples. Values of brightness are positively affected by sodium hydroxide treatment in all conditions, with improvements ranging from approximately 0.12% ($O5Na_1$) to 17.2% ($O7.5Na_3$) compared with the control.

However, in all formamide treatment conditions, usually similar or somewhat lower (by 0.1-2.7%) brightness values were observed. Lower brightness properties were observed for ethyl acetate treated samples. Hence it is clear that ethyl acetate usually negatively affects the brightness values of sheets.

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

Trt.	Density (g/ cm^3)	Change rel. to O_{1-3} (%)	WRV (g/g)	Change rel. to O_{1-3} (%)	Air Perm. (m^2/sn)	Change rel. to O_{1-3} (%)	Brightness (%)	Change rel. to O_{1-3} (%)
OC_1	0.37	0	1.91	0	4.94	0	25.49	0
OC_2	0.38	0	1.81	0	8.14	0	26.53	0
OC_3	0.48	0	1.49	0	4.67	0	25.98	0
$O5Na_1$	0.41	10.8	2.37	24.1	10.8	118.6	25.52	0.12
$O5Na_2$	0.39	2.6	2.26	24.9	3.32	-59.2	27.6	4.1
$O5Na_3$	0.39	-18.8	2.03	36.2	5.64	20.8	30.24	16.4
$O7.5Na_1$	0.45	21.6	1.99	4.2	8.2	65.9	26.24	2.9
$O7.5Na_2$	0.44	15.8	1.82	0.6	5.6	-31.2	27.85	4.9
$O7.5Na_3$	0.35	-27.1	1.74	16.8	21.4	358.2	30.42	17.1
$O10Na_1$	0.37	0	1.87	2.1	19.8	300.8	26.91	5.6
$O10Na_2$	0.38	0	1.92	6.1	10.5	28.9	28.60	7.8
$O10Na_3$	0.37	-22.9	1.72	15.4	24.1	416.1	29.64	14.1
$O5Fa_1$	0.38	2.7	1.96	2.6	12.4	151.1	25.98	1.9
$O5Fa_2$	0.43	13.6	1.94	7.2	5.5	32.4	25.95	-2.2
$O5Fa_3$	0.45	-6.3	2.17	45.6	6.24	33.6	26.80	3.2
$O7.5Fa_1$	0.38	2.7	2.39	23.6	11.8	138.7	24.91	-2.3
$O7.5Fa_2$	0.41	7.9	2.46	35.9	6.5	-20.1	26.14	-1.5
$O7.5Fa_3$	0.42	-12.5	3.02	102.7	14.5	210.5	26.83	3.3
$O10Fa_1$	0.43	16.2	2.50	30.9	9.93	101.1	25.31	-0.71
$O10Fa_2$	0.43	13.2	2.55	40.9	12.3	51.1	25.82	-2.7
$O10Fa_3$	0.39	-18.8	2.63	76.5	3.75	-21.8	26.41	1.7
$O5Et_1$	0.46	24.3	2.20	15.2	6.01	21.7	23.57	-7.6
$O5Et_2$	0.41	7.9	1.84	1.7	9.55	17.3	24.64	-7.1
$O5Et_3$	0.41	-14.6	2.12	42.3	9.87	111.3	24.52	-5.7
$O7.5Et_1$	0.46	24.3	2.92	52.9	3.6	-27.1	23.36	-8.4
$O7.5Et_2$	0.40	8.1	1.92	6.1	9.53	17.1	24.60	-7.3
$O7.5Et_3$	0.39	-18.8	1.96	31.5	11.1	137.7	24.85	-4.3
$O10Et_1$	0.46	24.3	1.82	-4.7	3.42	-30.8	23.51	-7.8
$O10Et_2$	0.42	10.5	2.59	43.1	6.55	-19.5	26.61	0.3
$O10Et_3$	0.41	-14.5	2.25	51.0	5.95	27.4	24.92	-4.1

OC: Control, **1-3:** Recycling stage, **Na:** Sodium hydroxide; **Fa:** Formamide, **Et:** Ethyl acetate, **5, 7.5, 10:** % chemical charge (based on oven dry pulp)

To determine correlations between sheet densities and recycling stage with chemical treatment, these variables were plotted for treatment with sodium hydroxide (Fig. 1A), formamide (Fig. 1B) and ethyl acetate (Fig. 1C). It can be seen that sodium hydroxide treatment positively affects sheet density (Fig. 1A) up to 7.5% chemical charge; it then decreases with increasing chemical charge and recycling stage. In contrast to sodium hydroxide treatments, all of the formamide treatment conditions (Fig. 1B) usually have positive effects as the chemical charge and recycling stage increase. However, ethyl acetate usually produces little change or a small improvement in sheet density (Fig. 1C).

Similarly, for determining correlation between water retention values (WRV) and recycling stage with chemical charge, these variables were plotted for sodium hydroxide (Fig. 2A), formamide (Fig. 2B) and ethyl acetate (Fig. 2C) treatment. Sodium hydroxide treatment (Fig. 2A) was found to have a positive effect with 5.0% chemical charge; the values then decreased as chemical charge and recycling stage increased. However, the formamide treatments (Fig. 2B) usually had positive effects as chemical charge and recycling stage number increased. Ethyl acetate usually had positive effects at 7.5-10.0% chemical charge (Fig. 2C).

All three important strength values of hand sheets are given in Table 3. Sodium hydroxide treatment normally improves tensile strength by approximately 4.4% ($O10Na_3$) to 57.3% ($O7.5Na_2$). Similar improvement is observed for formamide and ethyl acetate treatment of fibres. The highest tensile strength value of 42.09 Nm/g was recorded at the first recycling stage with 10% ethyl acetate treatment ($O10Et_1$); this represents an improvement of approximately 142.2% in the tensile strength of sheets compared with control samples at the same recycling stage. Increasing recycling stage usually negatively affected the tensile strength, but the strength values were still higher than for the control samples.

Largely similar results were obtained for the burst strength of sheets: all three chemical treatments had positive effects on burst strength. The highest burst strength of 2.60 kPa m^2/g was again recorded at the first recycling stage with 10% ethyl acetate treatment ($O10Et_1$); this represents an improvement of approximately 100% in burst strength compared with control samples at the same recycling stage.

The results revealed that certain chemical treatments markedly improved both tensile and burst strengths, but some variation was observed for tear strengths. The largest improvement (72.9%) in tear strength was obtained at the third recycling stage with 5.0% formamide treatment ($O5Fa_3$), followed by $O10Et_3$ (33.9%) and $O10Na_3$ (29.2%). It is important to note that the highest tear strength value (9.09 Nm^2/g) was obtained at the second recycling stage for the control samples.

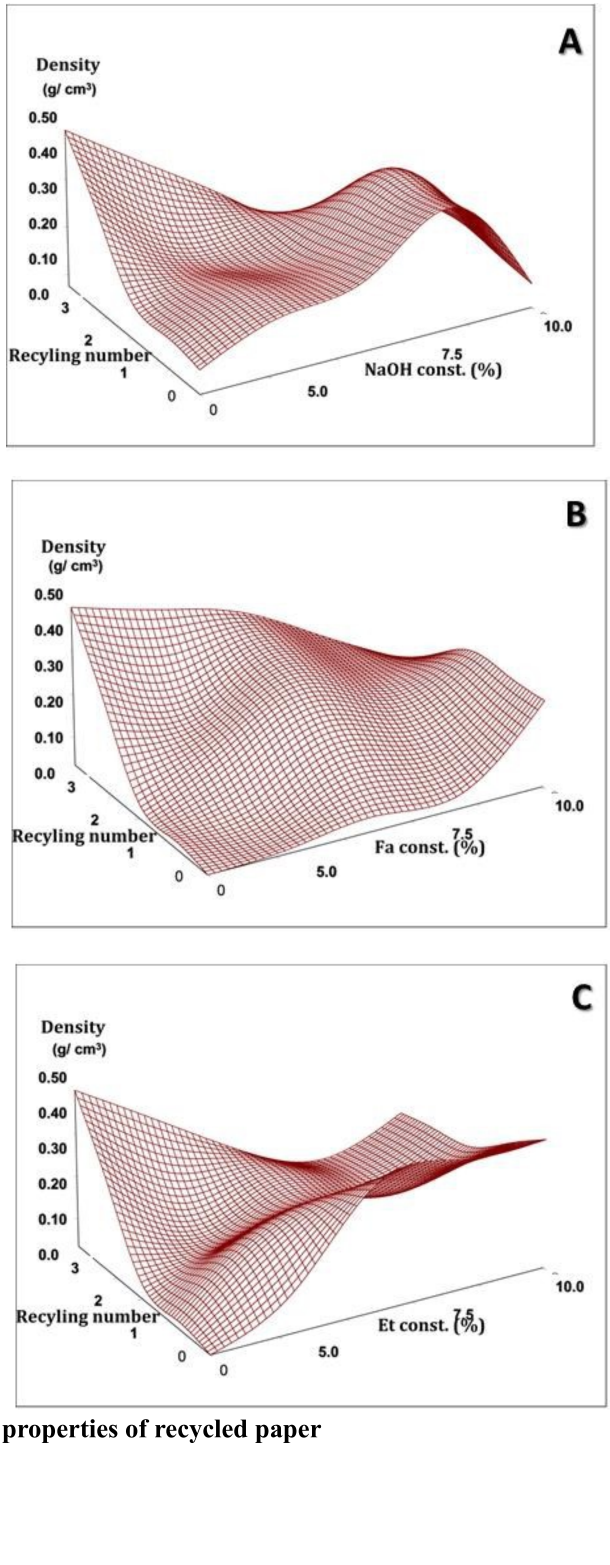

Fig. 1. Density properties of recycled paper

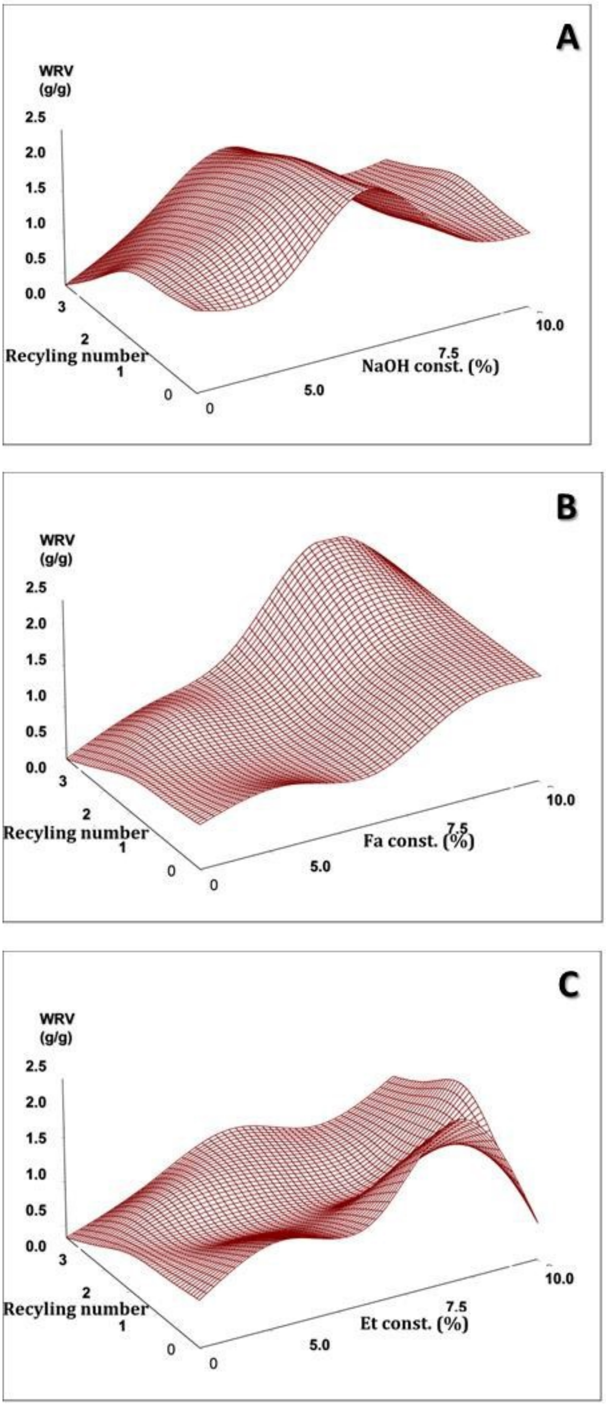

Fig. 2. Water retention properties of recycled paper

The strength results clearly show that the tensile and burst strengths of sheets can be improved by certain chemical treatments, but there is no correlation with tear strengths. However, numerous studies show that there is no correlation between the strengths of pulps, especially in the case of tear strength.

It has been reported by a number of researchers that types of pulp have different responses to recycling. Moreover, it is speculated that there should be some variation of strength properties and there is no direct correlation between them for recycled sheets [Biermann 1993; Scott and Abbott 1995; Fernandez and Young 1996]. Recycling not only affects the fibres' physical properties (length, diameter, etc.), but it also affects chemical degradation or depolymerisation. In addition to the length and strength of single fibres, the chemical composition of fibres and amorphous regions in the cellulose are significant factors. In our study, the difference between pulp (OCC) and fibre characteristics (chemical, semi-chemical and mechanical) appears to lead to different effects on the tear strength of recycled fibres. Howard and Bichard [1992] and Howard [1995] found that refined chemical pulps are more susceptible to reduction in strength, but mechanical pulps display even small gains in strength. The reason for the lower rigidity of sheets in case of recycled chemical pulps is probably the loss of fibre bonding (E modulus). For mechanical pulps, the reason is the loss of fibre thickness owing to flattening. Howard and Bichard [1992, 1993] reported that a blend of mechanical and chemical pulp (i.e. OCC) revealed that the effects of recycling on sheet properties occur at different rates.

It appears that the chemical charge improves the hydrophilic nature of fibres and bonding potentials. However, there are still questions concerning the strength development mechanisms for tear strength. It is difficult to evaluate the development of tear strength because of the possible impact of complex recycling procedures, which may have effects on all fibre properties.

The tensile strengths of sheets, with correlations with recycling stage and chemical charge, are shown in Figure 3A-C. It can be observed that all three chemicals – sodium hydroxide, formamide and ethyl acetate – have positive effects on tensile strength. Especially ethyl acetate has positive effects in all recycling conditions (Fig. 3C), whereas sodium hydroxide has positive effects up to 7.5% chemical charge and then produces some decrease in tensile strengths (Fig. 3A). In the case of formamide, marked improvement was found only at a 5.0% charge (Fig. 3B).

The graphs of the burst strengths of sheets (Fig. 4A-C) have a similar shape to those of tensile strength. Sodium hydroxide (Fig. 4A) and formamide (Fig. 4B) have marked effects at 5.0% and 7.5% chemical charge respectively. However, ethyl acetate shows positive correlation with chemical charge and recycling stage under all conditions (Fig. 4C).

Considerably different plot shapes were observed for the tear strengths of paper, compared with tensile (Fig. 3) and burst strengths (Fig. 4). It can be seen

Table 3. Strength properties of chemically treated recycled pulps

Trt.	Tensile strength (Nm/g)	Change rel. to O_{1-3} (%)	Burst strength ($kPa \cdot m^2/g$)	Change rel. to O_{1-3} (%)	Tear strength (Nm^2/g)	Change rel. to O_{1-3} (%)
OC_1	17.11	0	1.30	0	7.20	0
OC_2	16.23	0	1.27	0	9.09	0
OC_3	17.01	0	1.13	0	4.69	0
$O5Na_1$	19.64	14.8	1.34	3.1	6.49	-9.7
$O5Na_2$	24.51	51.0	1.78	40.2	6.39	-9.9
$O5Na_3$	18.86	7.2	1.25	10.6	5.66	20.7
$O7.5Na_1$	25.28	47.8	1.62	24.6	8.12	12.8
$O7.5Na_2$	25.53	57.3	1.85	45.7	5.57	-38.7
$O7.5Na_3$	21.42	23.1	1.37	21.2	4.76	1.5
$O10Na_1$	21.99	28.5	1.51	13.9	8.97	24.6
$O10Na_2$	19.89	22.4	1.28	0.8	7.45	-18.1
$O10Na_3$	18.18	4.4	1.24	9.7	6.06	29.2
$O5Fa_1$	19.68	15.0	1.33	2.3	7.59	5.4
$O5Fa_2$	26.25	61.7	1.75	37.8	6.28	-30.9
$O5Fa_3$	22.71	30.4	1.52	34.5	8.11	72.9
$O7.5Fa_1$	22.26	30.1	1.63	25.4	6.28	-12.8
$O7.5Fa_2$	20.27	24.8	1.46	14.9	4.19	-52.1
$O7.5Fa_3$	23.02	32.2	1.34	18.6	4.35	-10.7
$O10Fa_1$	26.18	56.6	1.54	14.9	8.11	12.6
$O10Fa_2$	23.42	44.3	1.48	16.8	4.83	-46.9
$O10Fa_3$	23.59	35.5	1.36	8.8	5.76	22.8
$O5Et_1$	27.59	61.3	2.16	66.1	5.23	-25.0
$O5Et_2$	28.25	74.0	1.83	44.1	5.40	-42.5
$O5Et_3$	30.27	73.9	1.69	49.6	5.40	15.1
$O7.5Et_1$	36.61	113.9	2.39	83.8	4.91	-31.8
$O7.5Et_2$	31.16	91.9	2.08	63.8	5.49	-39.6
$O7.5Et_3$	26.88	54.4	1.65	46.1	5.23	11.5
$O10Et_1$	42.09	145.9	2.60	100	5.23	-27.4
$O10Et_2$	34.96	115.4	2.27	78.8	5.76	-36.6
$O10Et_3$	28.55	63.9	1.99	76.1	6.28	33.9

OC: Control, **1–3:** Recycling stage, **Na:** Sodium hydroxide; **Fa:** Formamide; **Et:** Ethyl acetate; **5, 7.5, 10:** % chemical charge (based on oven dry pulp).

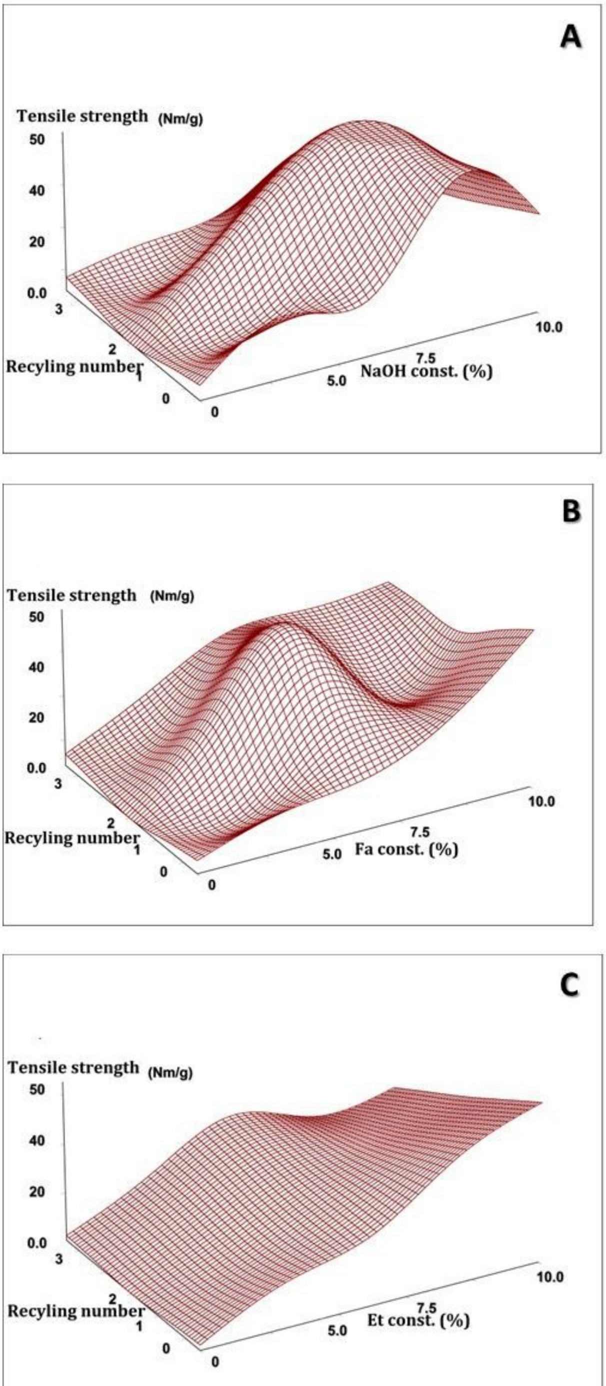

Fig. 3. Tensile strength of recycled paper

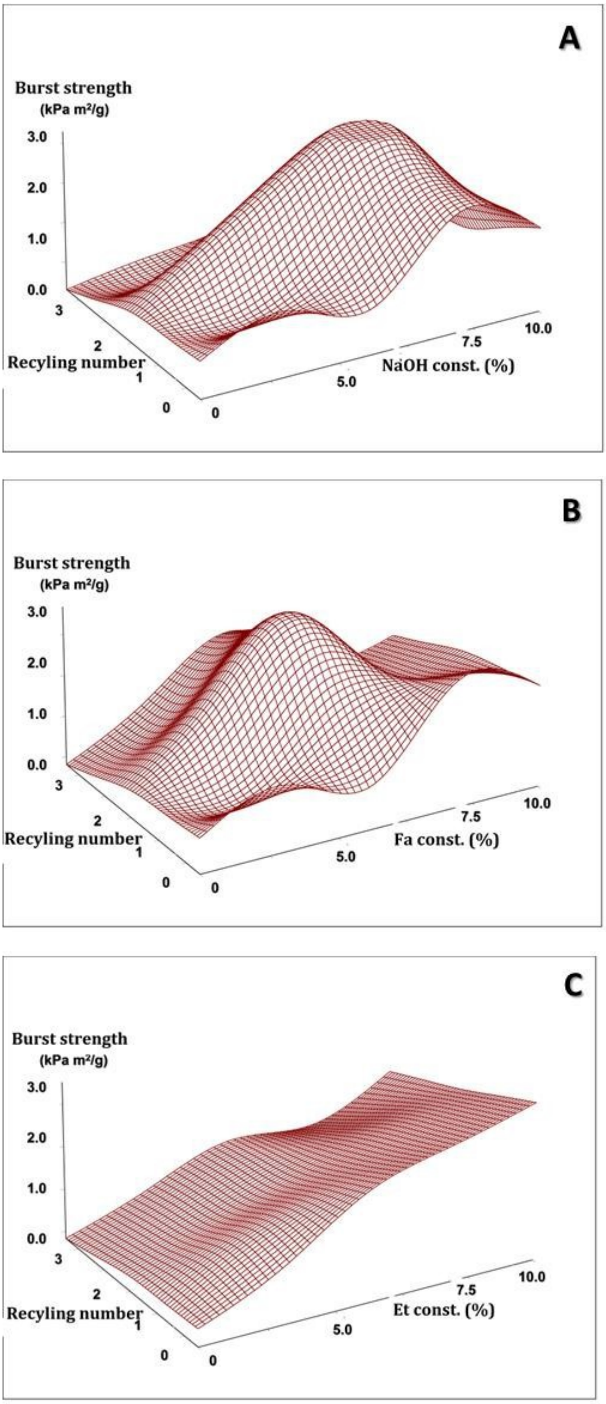

Fig. 4. Burst strength of recycled paper

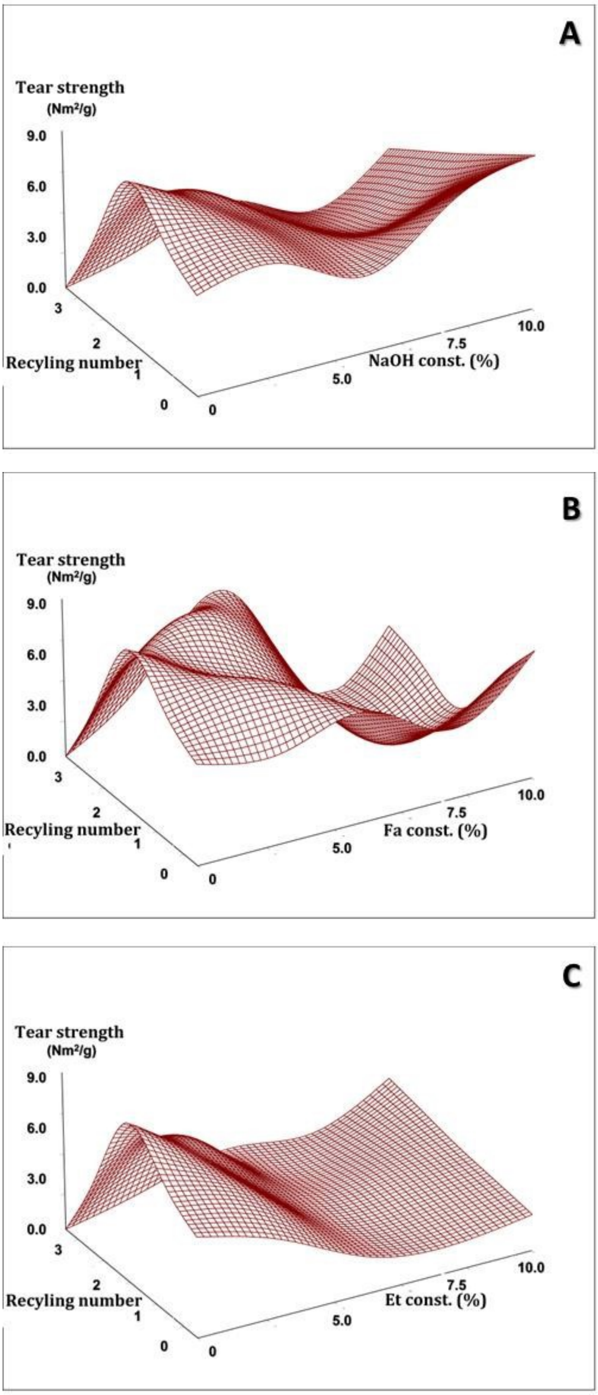

Fig. 5. Tear strength of recycled paper

that sodium hydroxide concentration negatively affects tear strengths up to 7.5% chemical charge (Fig. 5A). Further increasing the concentration leads to some improvement in tear strength. With formamide, recycling stage and chemical concentration showed no correlation with tear strength, although some variation and decreasing effects were observed (Fig. 5B). Interestingly, ethyl acetate treatments (Fig. 5C) had clearly negative effects on the tear strengths of sheets, in contrast to tensile (Fig. 3A and 4A) and burst strengths (Fig. 3B and 4B).

A number of findings reported in the literature have shown that tear strength in particular is subject to very complicated effects in terms of fibre properties [Fernandez and Young 1996].

However, recycling affects not only cellulose, but also hemicellulose degradation or depolymerisation. In addition to single fibre length and strength, the hemicellulose content of the pulp, especially xylan content, and amorphous regions in the cellulose are also significant. It seems that the chemical charge improves the hydrophilic nature of fibres. However, there are still questions concerning the mechanisms by which hemicelluloses enhance strength. It is difficult to evaluate the effects of hemicelluloses on strengths because of the possible impact of complex recycling procedures, which may affect carbohydrates to various degrees. The results found for tear strength of pulps are clearly consistent with other findings reported in the literature.

Conclusions

The use of recycled paper products in the papermaking industry is beneficial for sustainability and for protecting forest resources. There is growing interest in the use of recycled fibres in the paper industry. For success in paper recycling processes, it is important to know the physicochemical properties of the secondary cellulose fibres. On this basis, some interventions during paper recycling can improve the quality and yield of paper products, while some undesirable effects can be prevented. However, high-quality paper products can be obtained only from improved recovered cellulose fibres.

Recovered cellulose fibres have usually been used in low-quality paper products. However, an alternative approach to the use of recovered cellulose fibres can provide added value in paper products.

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List of standards

SCAN-C 62:00 Water retention value (WRV)
TAPPI T205 om-88 Forming hand sheets for physical tests of pulp
TAPPI T220 sp-96 Physical testing of pulp hand sheets
TAPPI T403 Bursting strength of paper
TAPPI T414 Internal tearing resistance of paper (Elmendorf-type method)
TAPPI T460 om-02 Air resistance of paper (Gurley method)

TAPPI T494 om-96 Tensile properties of paper and paperboard (using constant rate of elongation apparatus)
TAPPI T525 om-96 Diffuse brightness of paper, paperboard and pulp (d/0) – ultraviolet level C

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