Effects of Flotation Deinking on the Removal of Main Colors of Oil-Based Inks from Uncoated and Coated Office Papers

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This paper presents the results of trials to evaluate the color-stripping behaviour of offset printing colors and the effect of coating on deinking performance. Oil-based four-color inks were separately printed as base printing on coated and uncoated white poster paper, and then extensive pulping and deinking processes were carried out. Standard offset printings were conducted on paper samples using **c**yan, **m**agenta, **y**ellow, and blac**k** colors (CMYK), and the effects of deinking efficiency on the removal of each color were analysed. The pulps were made at Micro-Maelstrom Laboratory Pulper at specified conditions with and without bleaching agents. Formamidin sulfinic acid and hydrogen peroxide formulation were used as reductive and oxidative bleaching agents, respectively. Flotation deinking and thickening of pulp were practiced using Degussa flotation and thickening cell. The changes in the optical properties, such as brightness, whiteness, and color value (CIE L^*, a^* b^*) of deinked pulp were determined. It was found that optical properties of color-stripped pulps from coated papers were better than that of uncoated office papers. However, yield was quite low in coated papers since fillers were lost during flotation process. Cyan color was found to be the most difficult one in four main color printing inks stripping out in deinking process.

Keywords: Paper recycling; Flotation deinking; CMYK offset printing; Color stripping; Coating

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INTRODUCTION

The paper industry and academic scientists have been paying increased attention to paper recycling and the search for alternative fibre resources. Recovered paper is reported to supply around 53 percent of the world's total fibre furnish to meet the needs for paper and paperboard production (FAO 2011). Pulp made from non-woody plants has been reported to be only 4 percent of the total world's pulp consumption (Hurter and Riccio 2006; Tutuş and Karademir 2002). However, the proportion has been quite significant in some parts of the world in the past. In China and India, for instance, wastes from agricultural activities have been utilized to produce up to 56 to 73 percent of countries total pulp consumption, respectively (Hurter and Riccio 2006; Copur *et al*. 2007).

In the past 50 years, efforts have been increasingly exerted in the protection of forests and the environment. The better utilization of waste papers, finding new alternative fibre resources, and some genetic modifications on trees for industrial plantations have been pressing issues, and there are and have been numerous studies going on these fields (Byström and Lönnstedt 1997; Pilate *et al*. 2002; Berglund and Söderholm 2003; Ververisa *et al*. 2004). The usage of recovered fibers is especially important in countries with poor forest resources, since the recycling of paper can provide enough fibre to supply the increasing demand in a sustainable manner without damaging the environment (Karademir *et al*. 2002).

Waste papers contain a number of different materials, including fillers, starch, wet/dry strength chemicals, sizing agents, coating chemicals, some adhesives, coloring additives, and various inks. To recover high quality fibres from recycling, the processes should be designed to get rid of all these materials from papers without damaging both mechanical and optical properties of fibres. The solid waste material, *i.e.* the deinking sludge, needs to be properly disposed (Imamoglu and Karademir 2002).

Recovered poster paper and other documents may be present in waste paper in sufficient quantities to effect de-inking processes. Poster papers are mainly printed with CMYK (**c**yan, **m**agenta, **y**ellow, and blac**k**) colors by off-set printing systems. In order to produce economically attractive furnish and a good quality white pulp, they must first be color-stripped by use of a suitable method. Traditionally, sodium hypochlorite has been considered to be an efficient color stripping agent. However due to increasing environmental awareness, the industry is looking for an alternative process that does not use chlorine compounds (Hache *et al*. 1994). In keeping with the move away from chlorine-based bleaching agents, hydrogen peroxide, FAS, and sodium hydrosulphite have become the preferred chemicals (Vincent *et al*. 1997). Pulping, flotation, and post-bleaching are three main operations applied in recycling operations. The pulping operation involves the disintegration of dry stock. This stage determines the efficiency of subsequent stages (Renders 1993). A combination of chemical and mechanical forces is responsible for the detachment of ink from the fibre surface (Stack *et al* 2005). Several chemicals are used in the pulper, including alkali and a color-stripping bleaching agent. The reason for adding the chemicals to the pulper is to assist in the removal of undesirable materials such as ink and stickies from the paper and to make them accessible for flotation deinking. The flotation unit has often been referred to as the heart of the deinking system (Ferguson 1992). In the flotation unit, inks and coloring materials are targeted to be detached from fibres and forced to go a phase separation then removed by skimming.

The combined use of oxidative hydrogen peroxide and reductive FAS (formamidine sulfinic acid) for brightening recycled fibres is well established (Kronis 1996; Mahagaonkar *et al*. 1998; Imamoglu 2002; Imamoglu 2006; Sheng *et al.* 2011). There is, however, an increasing emphasis being placed on the color content of higher quality poster waste paper which is printed with the four main colors. The aim of this study was to explain the role of printing color type and coating on the color stripping efficiency. For this purpose the main four colors (cyan, magenta, yellow, and black, abbreviated as CMYK) of oil-based inks were printed on coated and uncoated white poster paper, and the deinking experiments were carried out. It was found that the color stripping of coated papers was significantly easier than that of uncoated office papers. Optical properties of color stripped pulps from coated papers were better than that of uncoated office papers. It is known that increasing the proportion of glossy magazines (coated paper) in the furnish resulted in a deinked pulp with higher brightness (Mahagaonkar *et al*. 1998). Deinked pulp yield was quite low in coated papers in comparison to uncoated papers, since coating pigments were lost during the flotation process. In addition, cyan colour was found to be the most difficult of the four printing inks to be stripped out.

EXPERIMENTAL

Printing

Sheets used in this study were 90 g/m² alkali poster papers with the size of 50 × 70 cm. Both coated and uncoated samples were evaluated. Sheets were printed on a Mitsubishi 3H5ED+TC offset printing machine to one side of the paper surface with the main printing colors of magenta, cyan, yellow, and black. Sun Chemical Uninova Q5777 mineral oil based printing ink was applied according to the ISO 12647-2:2004 standard, and two common drying mechanisms of oxidation-polymerisation and absorption were used. The percentage of uniform surface printed was 100%. Print density measurements on printed paper were done with a Gretag Macbeth SpectraEye device. Ash content (Tappi T211) and some properties of base and printed papers were measured according to the applicable standards, and results are listed in Table 1.

Table 1. Some Properties of Base and Printed Papers

Paper	Printing Color	Print Density	Paper Thickness (µm)	*L**	*a**	*b**	Brightness with UV filter (%)	Brightness without UV filter (%)	CIE Whiteness (%)	Ash Content (%)
UnCoated	UnPrinted	-	110	93.74	3.4	-13.7	87.45	103.56	146.94	26.42
	Magenta	1.23		52.06	49.4	-5.8	20.57	22.85	58.75	
	Black	1.32		36.48	0.6	1.8	8.44	8.73	-8.43	
	Yellow	1.32		80.97	6.9	69.4	10.38	11.19	-257.40	
	Cyan	1.12		56.95	-22.7	-38.9	56.29	58.45	285.11	
Coated	UnPrinted	-	70	93.90	2.0	-9.4	83.98	97.60	127.94	40.10
	Magenta	1.79		41.73	64.8	-5.1	12.60	13.79	49.82	
	Black	1.99		15.88	-0.2	-1.0	2.13	2.19	18.23	
	Yellow	1.65		78.53	9.8	93.0	3.55	3.69	-327.60	
	Cyan	1.61		54.25	-30.4	-43.1	56.75	57.98	320.39	

Pulping

100 g oven-dry papers for each process were cut into 2.5 × 2.5 cm squares and transferred into the 2 L capacity laboratory pulper (Micro-Maelstrom) (Fig. 1). Temperature in the pulper was set at 40 °C, and the consistency was adjusted to 15% by adding a calculated amount of hot water. Samples were allowed to wait for 5 minutes to become wet and swollen, and then 10 minutes of pulping was done with or without bleaching chemical agent. Reductive (FAS) and oxidative (H_2O_2) agents were used with a certain formula as bleaching chemical agents. Three main processes, namely pulping without chemicals, pulping with a reductive bleaching agent followed by flotation, and finally pulping with an oxidative bleaching agent followed by flotation were established. In order to understand the changes of values, some optical properties of printed or

unprinted base paper were measured. Alkalinity, to facilitate ink release by swelling the fibres and by saponification of the ink binders, was provided with NaOH. Hydrogen peroxide stabilisation was provided by use of sodium silicate. The quantities of the chemicals charged in the deinking formulations were calculated as a percentage of the o.d. weight of paper fed to the pulper. The chemical composition of the suspension was maintained at a constant level for each of the trials in pulping. Charged chemicals based on dry paper for OxiP process were; 1 wt.%H_2O_2 + 2 wt.% NaOH+ 2 wt.% Na_2SiO_3 and for RedP process were; 0.5 wt.% FAS + 0.25 wt.% NaOH. The pH values of pulp suspensions were measured (Table 2) at the beginning (pH-1), at the end of the pulping (pH-2), and at the end of the flotation (pH-3).

Table 2. pH Measurements of Process

Paper		pH Measurements		
		Pulping	Reductive Pulping +Flotation	Oxidative Pulping +Flotation
UnCoated	pH-1	7.54	9.25	11.82
	pH-2	11.75	7.14	11.33
	pH-3	11.49	10.44	7.35
Coated	pH-1	7.80	9.35	11.93
	pH-2	11.88	7.20	11.55
	pH-3	11.78	10.50	7.55

a b c

Fig. 1. Micro-Maelstrom pulper rotor (a), pulping process (b), and flotation process (c)

Flotation

A prepared pulp suspension was transferred into a 10 L capacity Degussa Flotation Cell (Fig. 1). A calculated amount of calcium chloride ($CaCl_2$) was dissolved in hot water and poured into the flotation cell to adjust water hardness to 10 °dH. Based on dry paper 1 wt.% industrial soap (Olinor-RS 4200 mixed fatty acid) emulsion was also added to the flotation unit before starting the mixer engine. The consistency of the pulp suspension in the flotation cell was adjusted to 1% by adding enough hot water at 40 °C.

Mixing with a perforated disc (100 mm radius) at 1450 rpm was maintained in the flotation cell. 2.5 L/min regulated airflow was maintained to the base of flotation cell just under the perforated mixing cell, which generated bubbles, removing inks from the suspension. A good phase separation was achieved, resulting in a colored, foamy froth on the surface of the suspension. The flotation was sustained for 10 minutes, during which time the froth was removed every 20 seconds with a scraper.

Preparing Pads and Optical Test

Deinked pulp was taken from the flotation cell and put in a Degussa thickening cell to remove waters remained from the flotation process. The mixture was drained through a 200-mesh wire screen in the thickening cell. Finally, dry matter of thick pulp, which was around 30% consistency, was determined and stored in the refrigerator at <4°C until used. The ash content of deinked pulps and rejects were determined according to TAPPI Method 211-om93. Total yield was calculated for each of the trials based on dry paper fed into the pulper (Table 3).

Table 3. Pulp Yield and Ash Content of Processed Pulp

Paper used	Printing color	Pulping without chemical		Reductive Pulping +Flotation		Oxidative Pulping +Flotation	
		Pulp yield (%)	Ash content (%)	Pulp yield (%)	Ash content (%)	Pulp yield (%)	Ash content (%)
UnCoated	UnPrinted	99,85	20.69	91.91	19.82	90.79	19.21
	Cyan	99,62	18.85	75.13	5.27	73.79	4.85
	Magenta	99,73	17.42	73.37	5.81	73.79	4.85
	Yellow	99,95	17.88	72.89	7.75	70.75	4.70
	Black	99,00	18.00	73.73	10.05	73.32	6.55
Coated	UnPrinted	96,74	32.23	83.39	33.78	60.99	11.23
	Cyan	99,88	36.47	48.16	7.97	49.93	4.85
	Magenta	99,82	31.84	49.55	8.08	52.82	4.26
	Yellow	99,00	36.00	48.50	6.65	51.09	2.23
	Black	99,00	37.86	51.59	8.68	54.16	4.18

Filter pads for optical tests of treated pulps were prepared in accordance with the modified INGEDE-1 method. A specified amount of thickened pulp was initially mixed with deionized water in a beaker with mild mixing for 2 minutes. The consistency of the fibre suspension was then set at 0.3% by adding more water, and the pH of the suspension was adjusted to 7. Prepared pulp suspension was filtrated over black filter to evaluate pad with the basis weight 225 g/m², followed by drying at 95 ºC for 10 minutes with the drying unit of a Rapid Köthen handsheet machine.

Brightness of filter pads was measured according to Tappi T 452 om-98 standards under a 457 nm wavelength. Measurements were done using a UV filter to avoid the disturbance of residual materials having some fluorescence effects. Color measurements of filter pads were achieved according to Tappi T 527-om 94 method, giving L^*, a^*, b^* values and Color difference (ΔE^*). Finally, filter pads CIE whiteness was determined in accordance with TAPPI Method T 562 pm-96, giving CIE (Commission Internationale de I'Eclairage) values.

RESULTS AND DISCUSSION

Printing and Pulping

Filler content in uncoated and coated office papers was found to be 26.42% and 40.10%, respectively. In general, fillers in office papers are distributed in the inner structure of the sheet matrix. In the case of coated papers, most of the mineral content actually is present near the sheet surface, together with other coating ingredients such as starch, binders, and so on. Therefore, coated papers have smoother surfaces and lower air permeability compared to uncoated sheets. Micro-capillary structures of such papers also differ remarkably. All these factors significantly affect the behaviours of liquid droplets over paper surfaces (Aydemir 2010) and the interaction between paper and inks (Aydemir *et al*. 2010). Printing ink consists of pigments, carrier (water, oil, or solvent), and binders. Carrier medium of an ink droplet over a coated paper surface was reported to be absorbed at a faster speed due to higher micro capillarity in the coated layers (micro-capillaries between filler particles). It was also reported that deeper troughs on the paper surface led to the deeper penetration of UV curing ink pigments (Li 2011). In this case, pigments would not get deep inside the coated layers, but actually stay near the paper surfaces, which give higher print density with less ink consumption. Ink penetration, on the other hand, can be deeper in the uncoated papers compared to that of coated sheets if no sizing agents interfere with the mechanism (Aydemir *et al*. 2010; Karademir and Hoyland 2003). These reports suggest that printing of coated papers can be done at a faster speed with less ink consumption. It was also suggested that pigments over the coated papers actually accumulate mostly in the coating layers, between fillers, and have fewer chances to face with fibres in the sheet structure.

During pulping process, the fillers in sheets get separated from fibres matrix and then in the flotation process separated fillers and other contaminant are removed with froth, leading to lower yield at the end. Therefore, the amount of filler lost is larger in the process of coated papers because of having more mineral content.

Optical Properties

Brightness of pulps was measured according to Tappi T 452 om-98 method with and without using a UV filter. Figure 2 shows that the brightness of evaluated pads made from coated paper increased as a result of pulping and flotation treatments.

When using a UV filter, the brightness value of unprinted coated samples showed no significant changes due to the processing. By contrast, when no UV filter was used (*i.e.* standard brightness measurements), the brightness value of same samples were found to be decreased at the end of pulping process from 97.60% to 94.87 % because of the removal of some optical brightening agent.

Cyan printed samples gave a higher brightness than all other printed samples. This result was thought to be due to the reflective ingredient present in the cyan ink used. Since brightness levels were measured according to TAPPI Official Test Method T525 om-92, which is a measure of diffuse reflectance at 457 nm (*i.e.* a blue light reflectance), cyan or blue printed papers gave naturally higher brightness levels. It is well known that the higher the blue-light reflectance generally the whiter the products will appear.

Magenta-printed coated paper was seen to be recovering its brightness at the end of process better than other printed coated papers did, except for the cyan-printed paper.

Figure 3 shows the development in the pulp brightness of all printed, uncoated papers. The range of increase is similar to that of uncoated samples, but the final brightness values of coated papers were remarkably better than those of uncoated office papers (Figs. 2 and 3).

Brightness values of both uncoated and coated papers printed in yellow, magenta, and black were plotted against treatments they received in the same graph, as seen in Fig. 4. It clearly reflects the effects of coating on deinking processes. Printing inks seem to be separated and removed easily from coated sheets giving higher recovery in pulp brightness. Inks in uncoated office papers are attached to fibres to some extent and exhibited some resistance against deinking. Therefore, residual inks in pulps of uncoated papers were expected to be higher, leading to a lower brightness value.

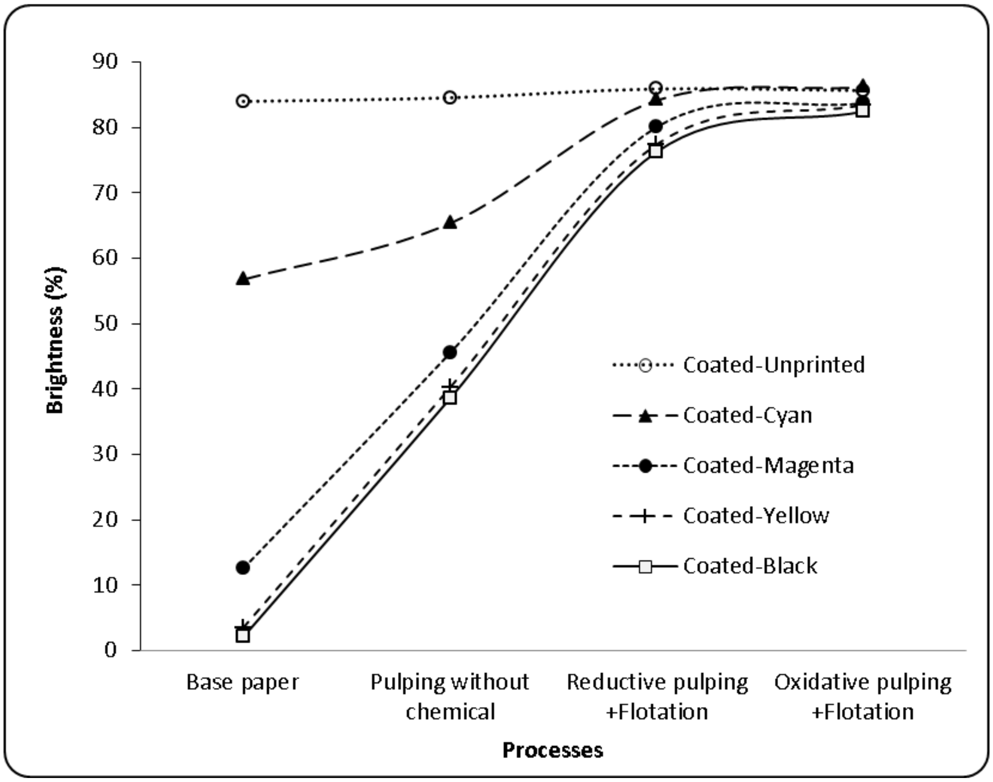


Fig. 2. Effects of processes on the brightness of coated papers

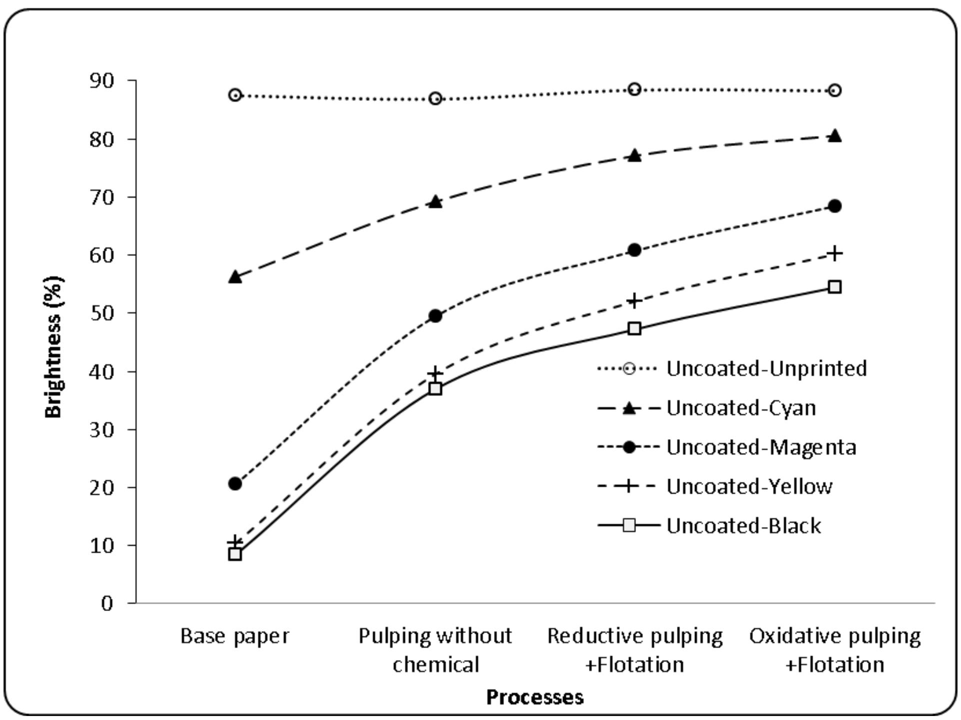


Fig. 3. Effects of processes on the brightness of uncoated office papers

All tests strongly indicated that deinking of coated papers gave better results than uncoated office papers in terms of final brightness of pulps. As clearly seen, the pulp yield was lower for coated papers than uncoated papers due to high filler content.

Sometimes the brightness of pulp is inadequate to explain how the colors are removed from pulp. For this reason CIE *L** *a** *b** values of pulps should be considered.

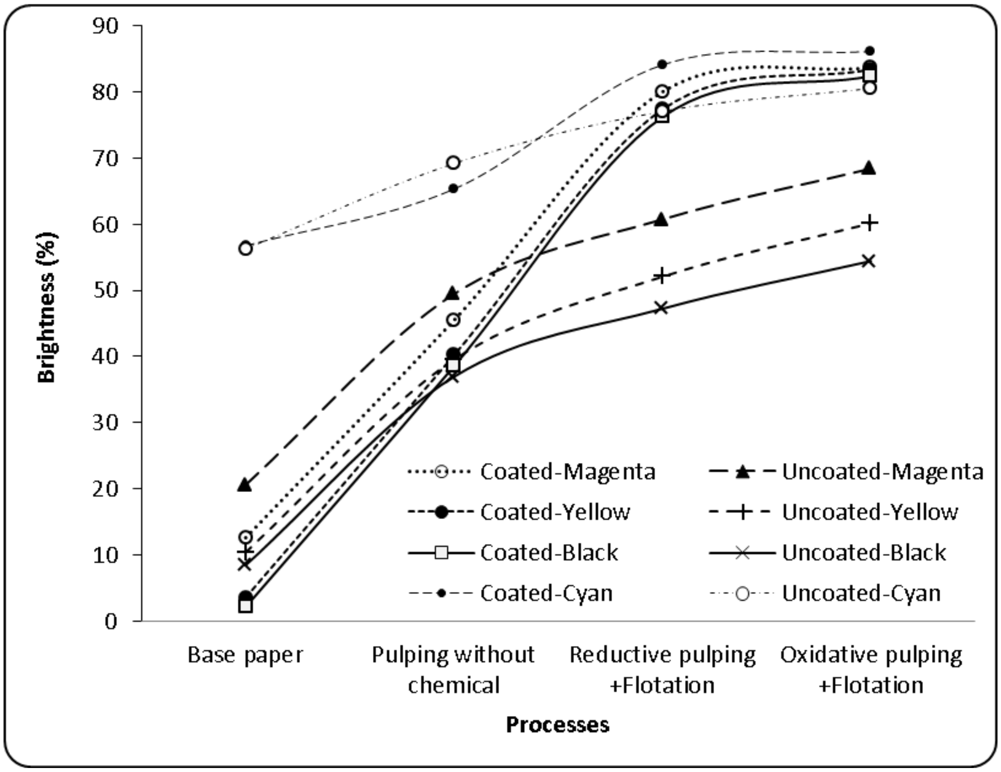


Fig. 4. Coated papers showing better recovery of brightness

Figure 5 shows the effects of pulp treatment on CIE *a** and *b** values of magenta-color papers. Positive values of CIE *a** represent the red color of paper. Pulping stages decreased the CIE *a** value of coated magenta paper from 64.8% to 17.2% and CIE *a** values of uncoated magenta-printed paper from 49.4% to 17.3%. Color removal of coated paper is always higher than uncoated paper because color pigments are present on the coating surface and are more easily dispersed in water compared to fibres. After the FAS-reinforced pulping and flotation stages, the CIE *a** value of coated magenta paper and uncoated magenta paper decreased to 4.9% and 13.6%, respectively. Hydrogen peroxide-reinforced pulping and flotation stages further decreased the CIE *a** values of the aforementioned samples down to 3.6% and 10.9%. In short, hydrogen peroxide was more effective than FAS relative to magenta color removal.

CIE *b** values at the negative coordinate, namely the blue color of paper, was increased by magenta color removal. This situation explains the highest increase in brightness (blue band 457 nm) of paper (approximately from 20% to 75%).

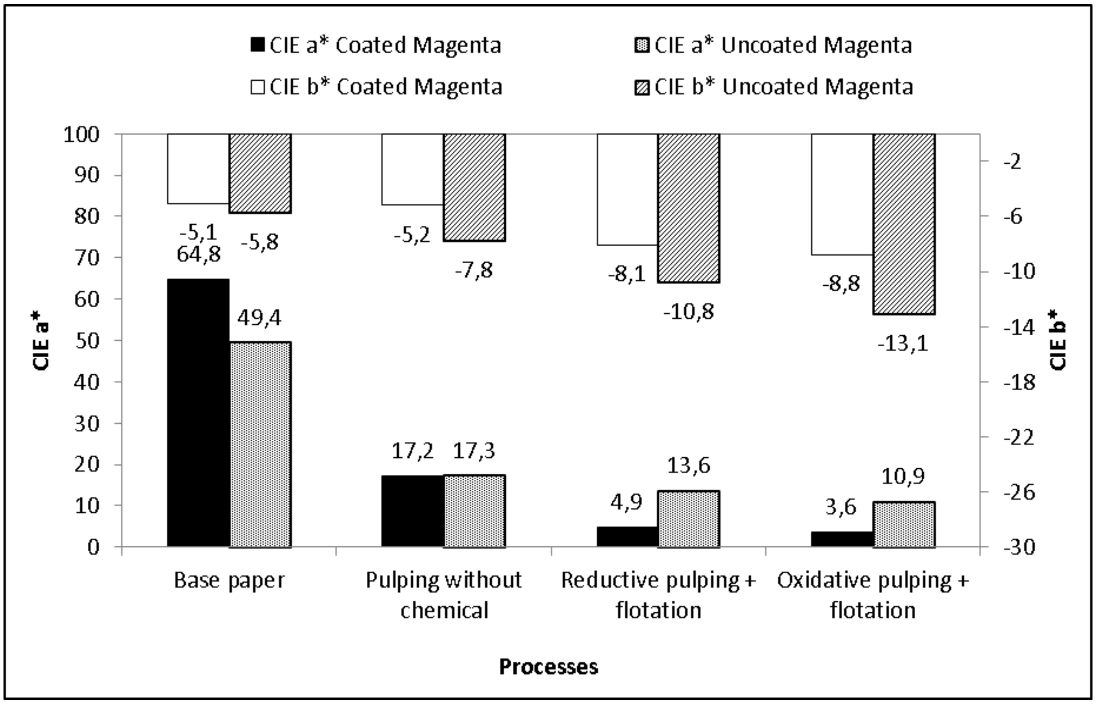


Fig. 5. The effects of pulp treatment on CIE *a** and *b** values of magenta color papers

Figure 6 shows the effects of pulp treatment during recycling on CIE *a** and *b** values of yellow-color papers. Positive values of CIE *b** indicate the yellow color of paper. Recycling processes decreased the yellowness of pulps. After flotation stages, the yellowness of pulps was decreased to sub-zero, and hydrogen peroxide was more effective than FAS on yellow color removal. CIE *a** values at positive direction namely cyan color was decreased along with the yellowness.

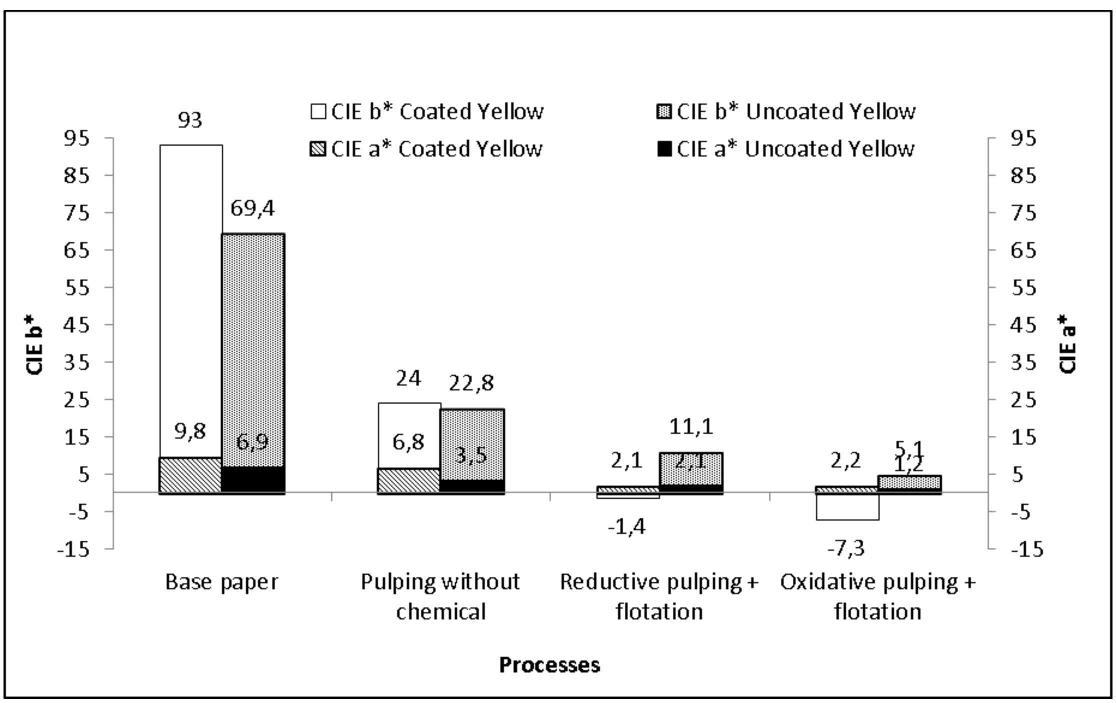


Fig. 6. The effects of pulp treatment on CIE *a** and *b** values of yellow color papers

Figure 7 shows the effects of pulp treatment during recycling on CIE *b** and *a** values of cyan color papers. CIE *b** at negative position indicates blue and CIE *a** at negative position indicates green color. With pulping and deinking stages, the blue and green color contents of paper decreased. Hydrogen peroxide and FAS had similar effects on blue color removal from pulp.

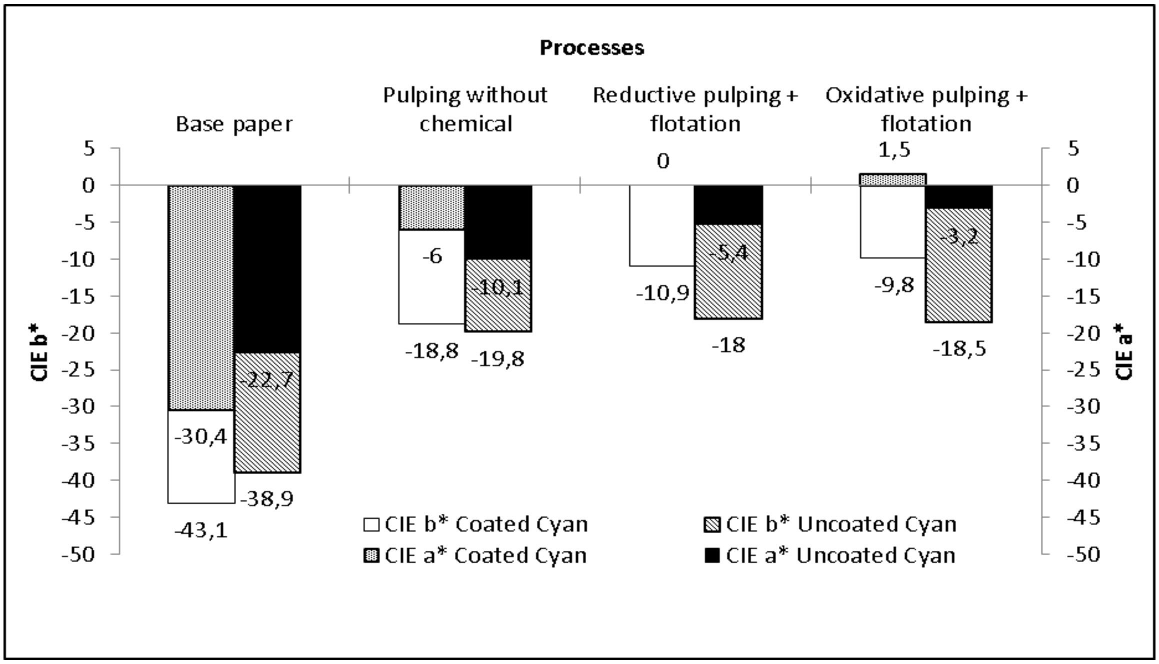


Fig. 7. The effects of pulp treatment on CIE *a** and *b** values of cyan color papers

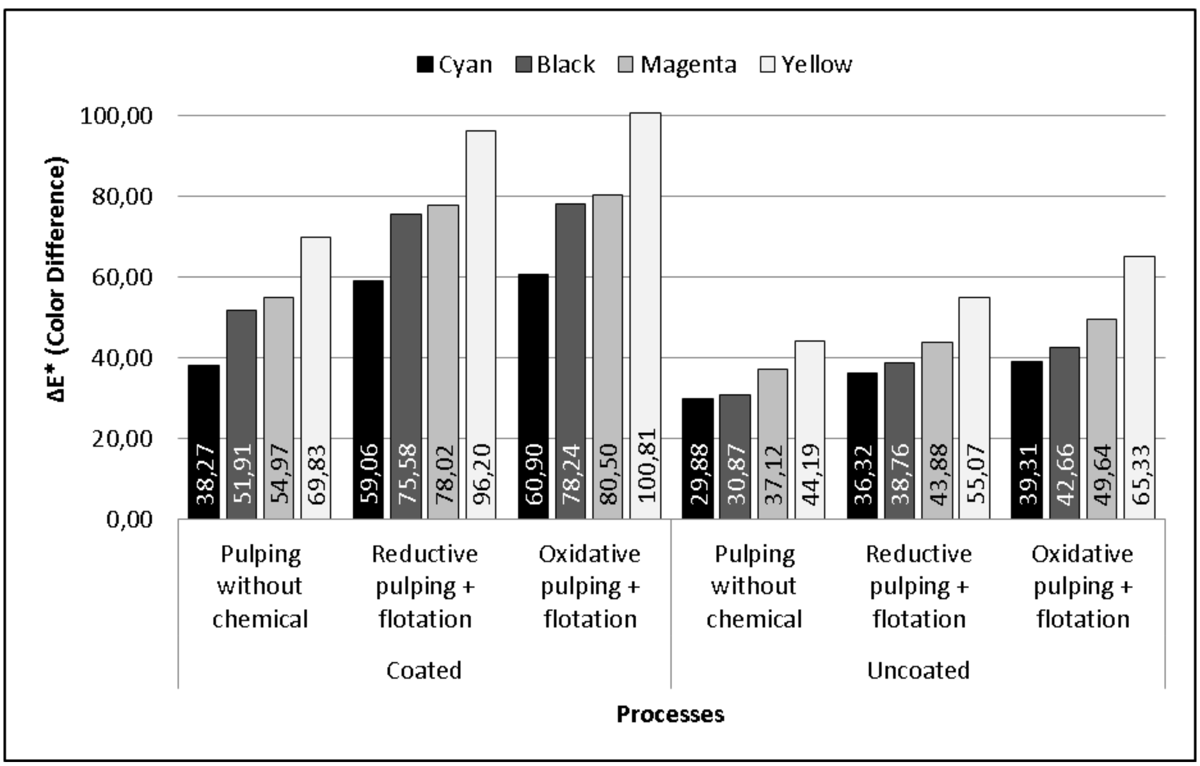


Fig. 8. Color removal of cyan, black, magenta, and yellow printed paper according to ΔE^*

When the removal difficulty of yellow, cyan, magenta, and black color printed papers were compared according the ΔE^* values (Total Color Difference (T 527 om-94): $\Delta E^* = ((\Delta L^{*2}) + (a^{*2}) + (b^{*2}))^{1/2}$), yellow color was removed easily, but the cyan and black colors were more stable than the other colors, as shown in Fig. 8.

Changes on CIE whiteness (CIE *W*) and lightness (CIE *L**) values of both uncoated and coated papers as a result of treatments were analyzed. Just some values of black printed samples are displayed in Fig. 9. It can be seen that recovery of measured parameters and final values were distinctly better for coated samples. It is again stressing that more physical and chemical attachments between ink components and fibers were developed in uncoated papers, which made the deinking of such papers more difficult, giving lower optical values than coated papers gave (Fig. 9).

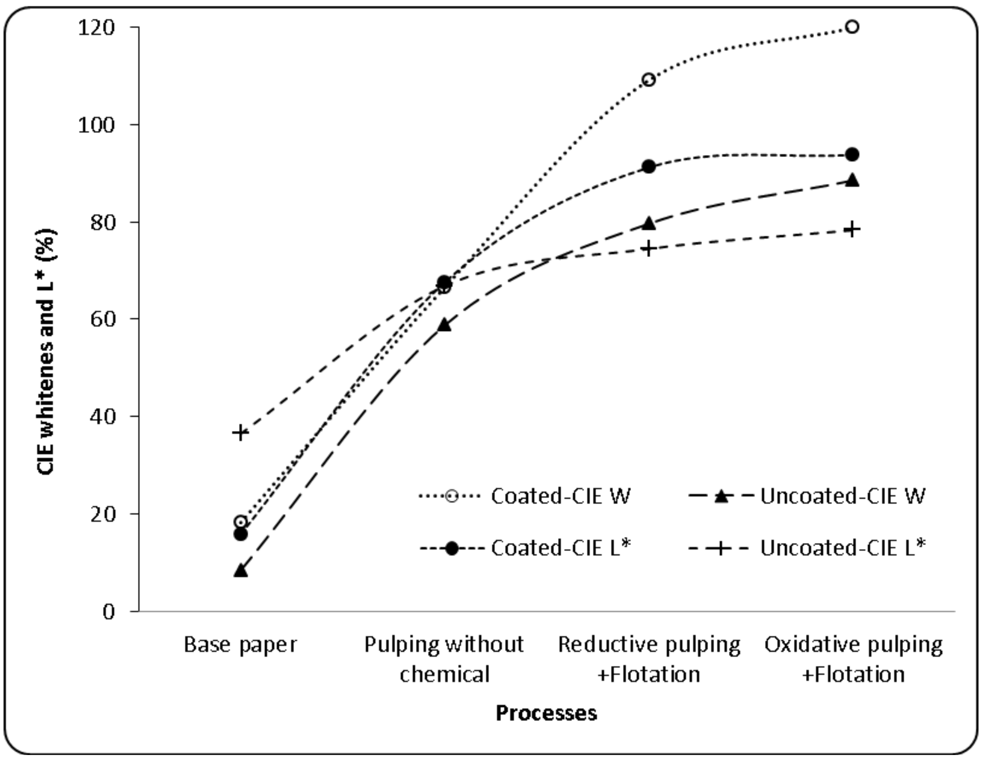
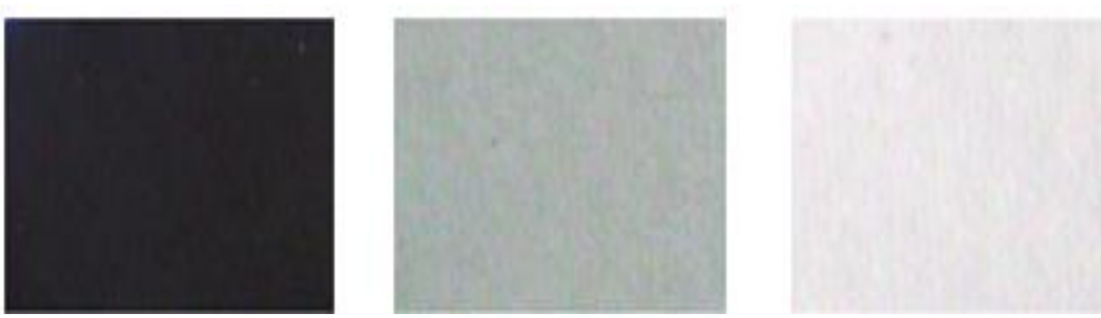


Fig. 9. Changes on some optical properties of black printed uncoated and coated papers

Pictures of control samples for uncoated papers printed in black, fibre mat after first pulping and fibre mat after final bleaching are displayed in Fig. 10 below.



a **b** **c**

Fig. 10. Black printed uncoated paper (a), after pulping (b), and final flotation (c)

CONCLUSIONS

As the content of colored paper in raw furnish increases, its price decreases. So while post-consumer mass-printed colored poster paper represents an economically attractive furnish, it must first be color-stripped or removed by a suitable method to produce a good quality white pulp. This study has led us to the following conclusions:

1. Color-stripping of coated papers was easier than uncoated poster papers. The coating layer took up most of the ink component and, in a sense, did not let them contaminate the fibres.
2. During pulping with reductive and oxidative bleaching agents, most of the inks and colorant components were removed with coating layers, and the resultant pulps exhibited better optical properties than pulps from uncoated poster papers.
3. Yield was expectedly lower for coated papers due to lost mineral content. It was seen that during the flotation process most of the fillers and coating pigment materials were removed with froth. 25% and 50% yield loss was calculated for uncoated printed and coated printed samples, respectively.
4. In terms of optical values, oxidative treatment was more effective than reductive treatment.
5. Ideally to evaluate good quality white pulp, cyan-color printed paper and coated paper, whatever its printing color, should not be excluded from furnishes.
6. Cyan-printed samples gave a higher brightness than all other printed samples. It was thought to be the reflective ingredient present in the cyan ink used. Since brightness levels were measured according to TAPPI Method T525 om-92, which is a measure of diffuse reflectance at 457 nm (*i.e*. a blue light reflectance), cyan or blue printed paper gave a naturally higher brightness level.

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