The Optical Properties of Deinked Pulp

Ivana Bolanča and Zdenka Bolanča†
Faculty of Graphic Arts, Zagreb, CROATIA

Optical properties of handsheets made from pulp resulting from deinking flotation of multicolored and desaturated prints from digital and conventional offset printing have been observed. The prints were made on different models of digital offset machines. One series of prints was naturally aged for the period of one year and the other was accelerated aged in a climatic chamber. The results show that the model of the printing machine, even with the same technology, can affect the size of particles and the optical properties of the handsheet. The quality of deinking pulp and the efficacy of the ink removal in relation to the optical parameters such as brightness, reflectance in the determined areas of wavelengths and color are discussed in this work.

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Introduction

Recycled fibers are important raw material in paper production. The recent increase in recovery rates of waste paper require an increase in consumption of recycled fibers in higher quality grades such as magazine and office paper for non impact printing. For recycling, deinking flotation is the most common method used.

Deinking is a process for detaching inks from fibers using chemical and mechanical conditions, and is followed by the flotation process for removal of the printing ink particles.[1] The efficiency of this process depends on many factors like: type of printing technique, kind of printing ink, properties of the substrate, type and amount of chemicals used in the various processing stages, and the hydrodynamics of flotation.[2–4]

Crosslinkable printed inks (oxidative drying and radiation curing), are difficult to remove from the fiber. Examples include ultraviolet inks and varnishes which break up in the disintegrator into particles too big to float. In addition, their crosslinked surface makes them resistant to attachment by collector chemicals.[5] Particle sizes of water based flexo inks after disintegration are too small for flotation. In addition these particles are hydrophilic and cannot agglomerate with the collector chemicals.[6]

Toners from some digital prints detach in flat plate-like structures and are too large to be removed by flotation.[7,8] Removal of plastics, coating binders, contact adhesive, and hot melts is important, because these materials can cause problems in the paper production and later in the printing process.

Letterpress printing inks are easy to remove from prints. To process these prints effectively into bright pulps a combination of different methods is necessary, such as flotation after ink particle fragmentation or detachment by dispersing or kneading.[9] Rotogravure prints made with toluene based inks are easier to deink than offset printed paper.

Deinking of coated paper is more successful than of uncoated substrate. Deinkability of uncoated paper is influenced by the absorption of the ink into the fiber network of the paper.[10]

The efficiency of the deinking operation depends on paper and print aging too. Deterioration in quality of an aged paper can manifest itself in chemical permanence and decrease in mechanical durability.[11] The permanence of paper or prints depends on the chemical resistance of its components and the influence of external factors. The durability depends mainly on the physical and mechanical characteristics of the raw materials, impact of microclimatic factors such as heat, or radiation, and on contamination by ions and gas from the environment as well as action of microorganisms.[12–15]

The natural aging process of paper and prints causes degradation of cellulose. The presence of moisture, oxidative agents and microorganisms and especially the presence of acidic substances, are important in this process. The results in this case are the hydrolysis of cellulose that appears as shortening of its chain along with changes in crystalline content.[16,17]

Acid catalyzed hydrolysis of cellulose has been recognized to be the primary reaction in the accelerated deterioration of paper. For study of accelerated aging of paper new methods are being developed, and recently a mathematical model for temperature dependence was presented from Rychly et al.[18]

Some authors jave been occupied with investigations of recycling efficiency of natural and accelerated aged prints. Alkaline and neutral chemical deinking for treatment of natural and accelerated aged flexographic prints are used Ryu and Lee.[19] More successful is neu-

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†Corresponding Author: Z. Bolanca, Zbolanca@yahoo.com

TABLE I. Condition of Pulping and Flotation

Parameter	Pulping	Flotation
NaOH %	1.0	–
H_2O_2 %	1.0	–
Water glass %	2.0	–
DTPA %	0.2	–
Surfactan t%	0.4	–
Consistency %	10.0	0.6
Time min	45	8
Temperature °C	40	30

tral deinking process in combination with alkaline treatment. Detachment of printing from different types of aged paper was investigated by Sjostrom and Callmel.[20]

Recycling of waste paper exposed to aging during the summer months, defined as the summer effect, was studied too.[21] The results show that the summer effect is due to the aging and thermal drying of printing inks.

The important characteristics of secondary raw materials are those which influence the printability and the runability of the paper and the quality and durability of the end product. Paper properties may be classified into those affecting surface, optical and strength characteristics.

The surface characteristics of paper depend on factors which include the nature of the fibers and their treatment during production. Some important strength properties include tensile strength, burst strength and tear resistance. Recycled fibers have lower strength properties than virgin paper.[22] The reduction in swelling and the loss of fibers' flexibility after drying diminish the strength potential of recycled fibers. Contaminants which occur in recycled pulps (stickies) and age degradation also contribute to the reduced strength of recovered fibers.

Some optical properties of handsheets obtained after the chemical deinking flotation of digital offset prints are presented in this article. Optical properties have been discussed in relation to the kinds of the printing techniques, model of the printing machine, conditions of the print aging before recycling, and the method of handsheet preparation.

Experimental

Colored and desaturated prints were used for recycling. For the production of prints in the offset digital technique, different models of Indigo E-Print machines were used, such as: rotary digital offset machine Galos DO 330 with six ElectroInks, sheet fed digital offset machine 1000 + and TurboStream with four ElectroInks in both cases. Printing was also performed on the sheet fed Heidelberg SpeedMaster printing machine. A unique test form was used in printing containing wedges of tonal values from 0–100% coverage in steps of 10% for CMYK.

Prints used for recycling were made on different printing substrates of the same grammage. The printing substrates used were uncoated (sample a) and double side coated paper for digital printing (sample b).

One series of prints was naturally aged for periods from one year and the other was accelerated aged in a climatic chamber at 80°C and 65% relative humidity for periods of 10, 20, and 30 days.

In the recycling process alkali chemical deinking was used. The conditions for the recycling of prints are presented in Table I. The workflow of the deinking flotation is presented schematically in Fig. 1.

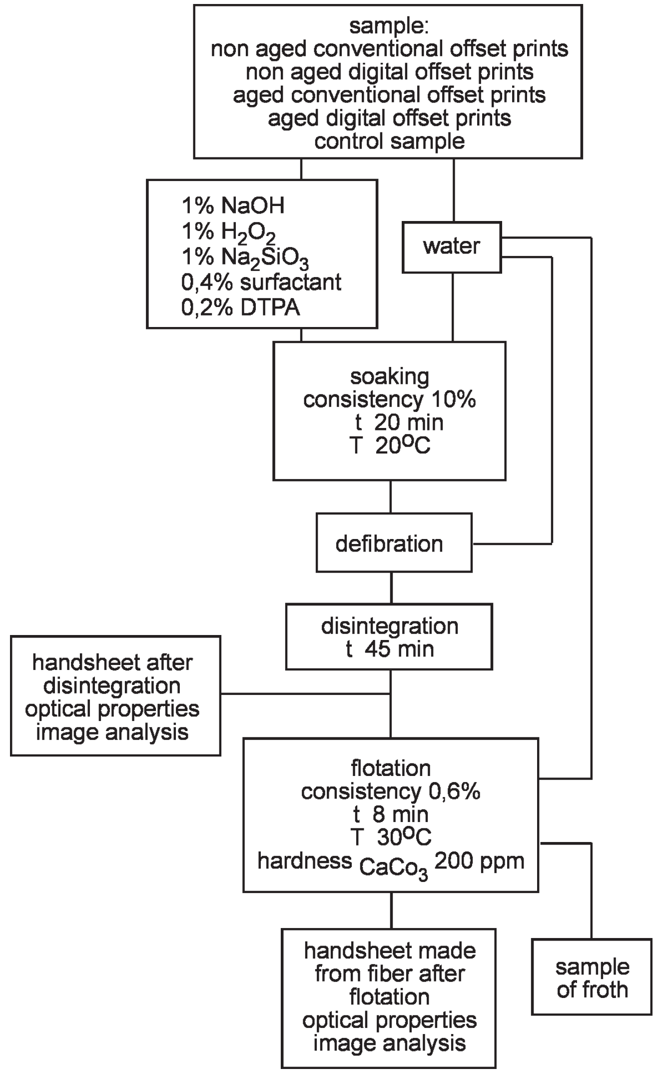


Figure 1. Workflow of the deinking flotation process.

During sample soaking, deinking chemicals were added. The consistency of the suspension was 10% with respect to the dry prints. The disintegration stage was 45 minutes. The suspension was diluted to 0.6% pulp consistency. An optimal level of hardness, 200 ppm $CaCO_3$, was maintained in the flotation cell. The flotation time was 8 minutes.

The handsheets were made using a laboratory sheet former, and by means of a Büchner funnel. The optical properties of handsheet were determined by using ISO standard methods.

Residual ink spot size, ink spot number, and ink areas were assessed with image analysis software Spec*Scan® from Apogee Systems Inc. Scanner optical resolution was set to 600 dots per inch. Threshold value –100, black level –65 and white level –75 were chosen manually after comparing computer images to handsheets. Ink particle size intervals were established with the software according to TAPPI methods T213 and T437.

Results and Discussion

The first impression of a paper is formed by its color, whiteness and gloss. The parameter frequently used for study of the mechanism of deinking flotation of differ-

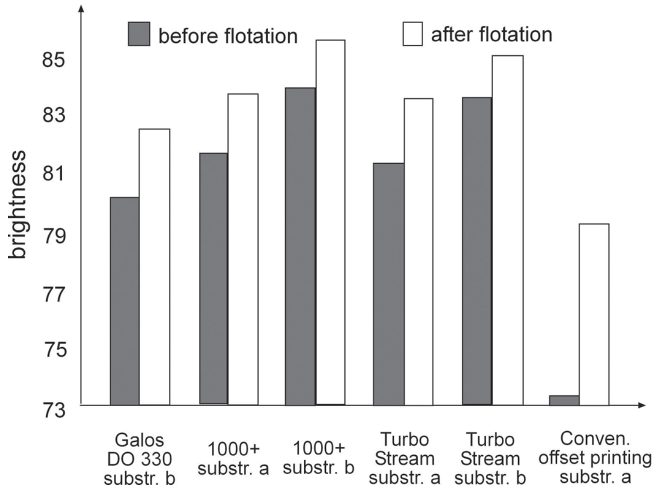


Figure 2. Brightness of handsheets before and after flotation

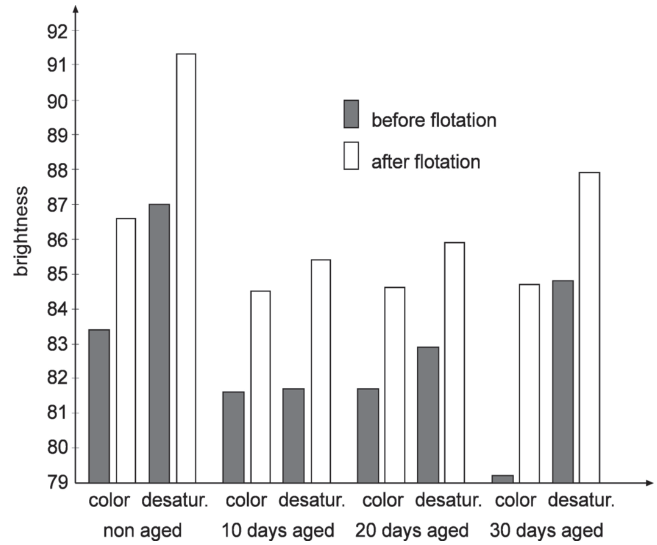


Figure 3. Brightness dependence on the characteristics of the prints and the time of aging

ent kind of prints under particular conditions and to evaluate the optical quality of deinked pulp is the spectral reflectance factor, R_{457}, used as ISO brightness. Figure 2 presents the brightness of the handsheets before and after flotation of prints made by different kinds of digital offset printing machine in relation to the handsheets derived from prints made by a conventional offset sheet fed machine.

The results show that the brightness of the handsheet is influenced by the printing technique and the kind of digital offset printing machine, as well as the kind of substrate. The smallest value of brightness is measured for the handsheet obtained by recycling of prints from a conventional offset machine. However, in this case the greatest brightness gain is obtained when handsheets before and after flotation are compared. The smallest differences in brightness are noticed for handsheets made by the recycling of prints from the web digital printing machine compared to the sheet fed digital offset printing machine, except insofar as somewhat greater values of brightness are obtained in those cases when specially coated paper designed for digital printing was used for printing.

Brightness of the handsheet obtained by recycling of the digital offset color and desaturated prints is presented in Fig. 3 (a desaturated print is a print on which the grey tones are reproduced by multicolor (CMYK) printing). The prints were made on the Indigo E-Print 1000+ machine. The accelerated aging of some of the prints was carried out for 10, 20, and 30 days before recycling.

Greater handsheet brightness can be observed in the case of desaturated prints which were not aged before recycling. Greater increase of brightness appears in comparison to handsheets before and after flotation from color prints.

By accelerated aging of color prints for 10, 20, and 30 days before recycling, the value of the brightness increase can be influenced. The corresponding value decreases somewhat with the increase of the aging time. The brightness increase is greater when compared with the desaturated prints which points to somewhat greater efficacy of the deinking process itself in this case.

The brightness of the handsheets after disintegration and flotation of color prints produced on different models of digital offset Indigo E Print machines and sub-

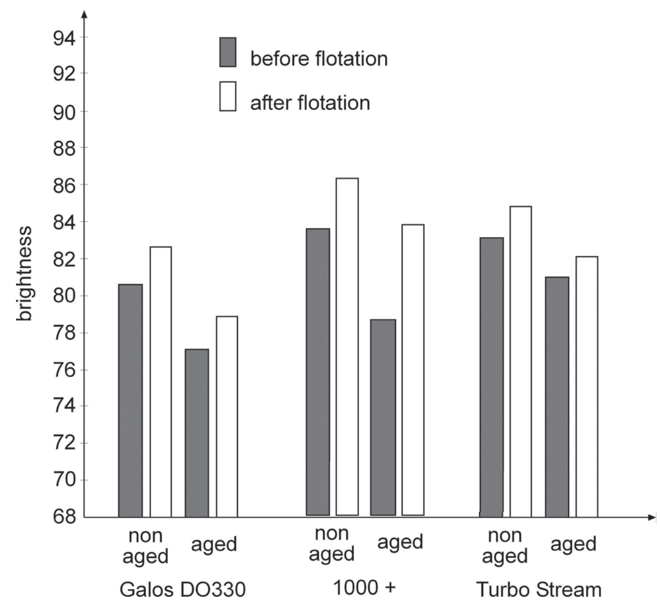


Figure 4. Brightness of handsheets obtained by recycling of natural aged prints

mitted to the natural aging for the period of one year is presented in Fig. 4.

By recycling the aged prints on the substrate b, made with different models of digital offset machines, the handsheets with lesser value of brightness are obtained compared to the non-aged prints. Greater differences in brightness are not noticed, except for somewhat greater difference values obtained on processing prints made on the Indigo E-Print 1000+. The presence of particles of ElectroInk of large surface area, which causes optical inhomogeneity of the handsheet, are characteristic of the handsheet in this case after disintegration, which is the reason for relative high handsheet brightness obtained by the processing of non-aged prints. By aging of the prints, the residual particles after disintegration become smaller and many of them are in the

TABLE II. Brightness Top and Screen Side of Handsheet

Sheet former			Buchner funnel		
Top side	Screen side	Difference	Top side	Screen side	difference
86.0	85.8	0.2	84.7	84.3	0.4
88.0	87.7	0.3	86.8	86.6	0.2
87.4	86.8	0.4	81.0	87.7	0.3
88.7	88.2	0.5	89.4	89.2	0.2
85.5	85.0	0.5	87.9	87.5	0.4

regime of optimal size for flotation detachment, which, as a result, provides a somewhat grater brightness gain.

In measuring brightness of the handsheets differences in values for the top and screen side of the sheet have been noticed. Generally, for the formation of the sheet for brightness measurements, two handsheet forming methods have been used: one with a sheet machine, and the other by forming a pad with a Büchner funnel. Some of these results are presented in the Table II.

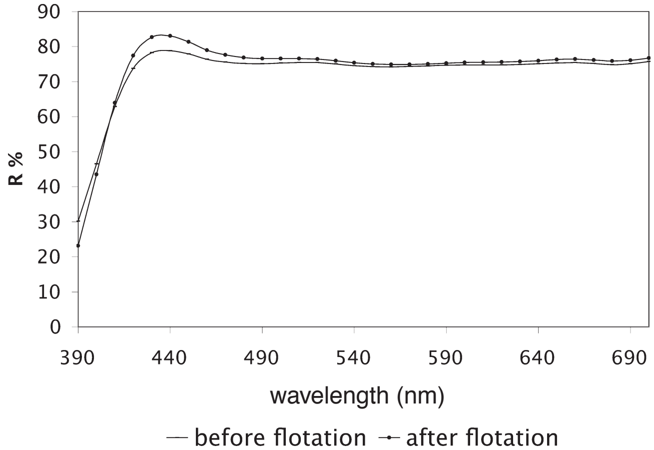
The results slightly show lower brightness on the screen side of the handsheet. Color prints were used for recycling. The differences in brightness on top and screen sides for five different samples of handsheet which are formed by means of the sheet machine is 0.2 – 0.5 points. By using the Büchner funnel for sheet formation, smaller differences have been obtained between brightness of top and screen sides of the handsheet.

In regard to the fact that only the blue part of the visible spectrum between the wavelengths 400 and 500 nm is included in the brightness measurements, Figs. 5(a) and 5(b) present the reflectance curves for handsheets before and after flotation obtained by processing of prints from the digital offset machine 1000+ on substrate b (Fig. 5(a)) as well as prints from a conventional offset press on substrate a (Fig. 5(b)).

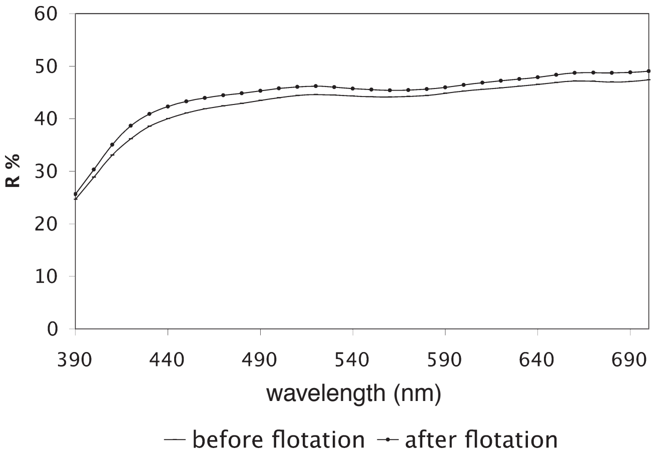
Handsheets made after disintegration of prints from digital offset printing with the 1000+ machine on substrate b show a reflectance growth. Reflectance increase is present in the shorter wavelength region, which is a characteristic of the original substrate used in printing. It could be thought that it is the consequence of the addition of optical whitening agent during the manufacturing of original paper. Handsheets after flotation have increased reflectance along the whole wavelength range, somewhat more so in the shorter wavelength region relative to the handsheet before flotation. The increased handsheet reflectance after flotation shows the slightly improved characteristics resulting by this process. Handsheets made after disintegration and flotation of prints from the conventional offset printing on substrate a have a somewhat different trend in the reflectance curve compared to the sample described before. The reflectance values are only about 45% with a slight increase from smaller to greater wavelengths. Thereby one can notice a strong influence of the original substrate used in printing.

In Fig. 6 the handsheet spectra before and after recycling of color and saturated digital offset prints (Indigo E-Print 1000+ machine) on substrate b are presented.

Greater influence of the different kinds of non-aged printing on the reflectance properties of the handsheets before and after flotation was not noticed. By aging of both kinds of prints, before and after recycling for 10 days, similar changes appear in the difference of reflection curves of the handsheets before and after flotation. By increasing the time of accelerated aging of prints, the influence of that process becomes more apparent.



(a) digital offset printing – substrate b



(b) conventional offset printing – substrate a

Figure 5. Spectral reflectance curves for handsheets derived from recycled digital and conventional offset prints

By recycling desaturated prints aged for 30 days a difference in the reflection curves before and after flotation, somewhat greater in respect to the color prints is obtained. The increase of handsheet reflection points to improved properties of the measured sample and demonstrates the efficacy of the deinking flotation, which was also confirmed by the brightness values presented above. However, it can be generally be said that the shapes of reflection curves of the handsheet before and after recycling are similar to the shapes of the reflection curves of the original printing substrate.

It is possibility to evaluate colored samples from the recycled paper processing operation in order to determine their color. Figure 7 presents a^*b^* plots for handsheets after disintegration and flotation.

As can be seen from the presentation, according to the measuring criteria of the CIE color system, the color of the handsheets after flotation does not differ greatly across the series studied. The more intensely colored handsheets are obtained by recycling prints from web digital offset printing under the experimental conditions.

From the handsheet series obtained after flotation of color and desaturated aged prints and presented in Fig. 7(b), one could conclude that accelerated aging of prints before flotation only trivially influences the color of handsheet. No significant difference in the coloration

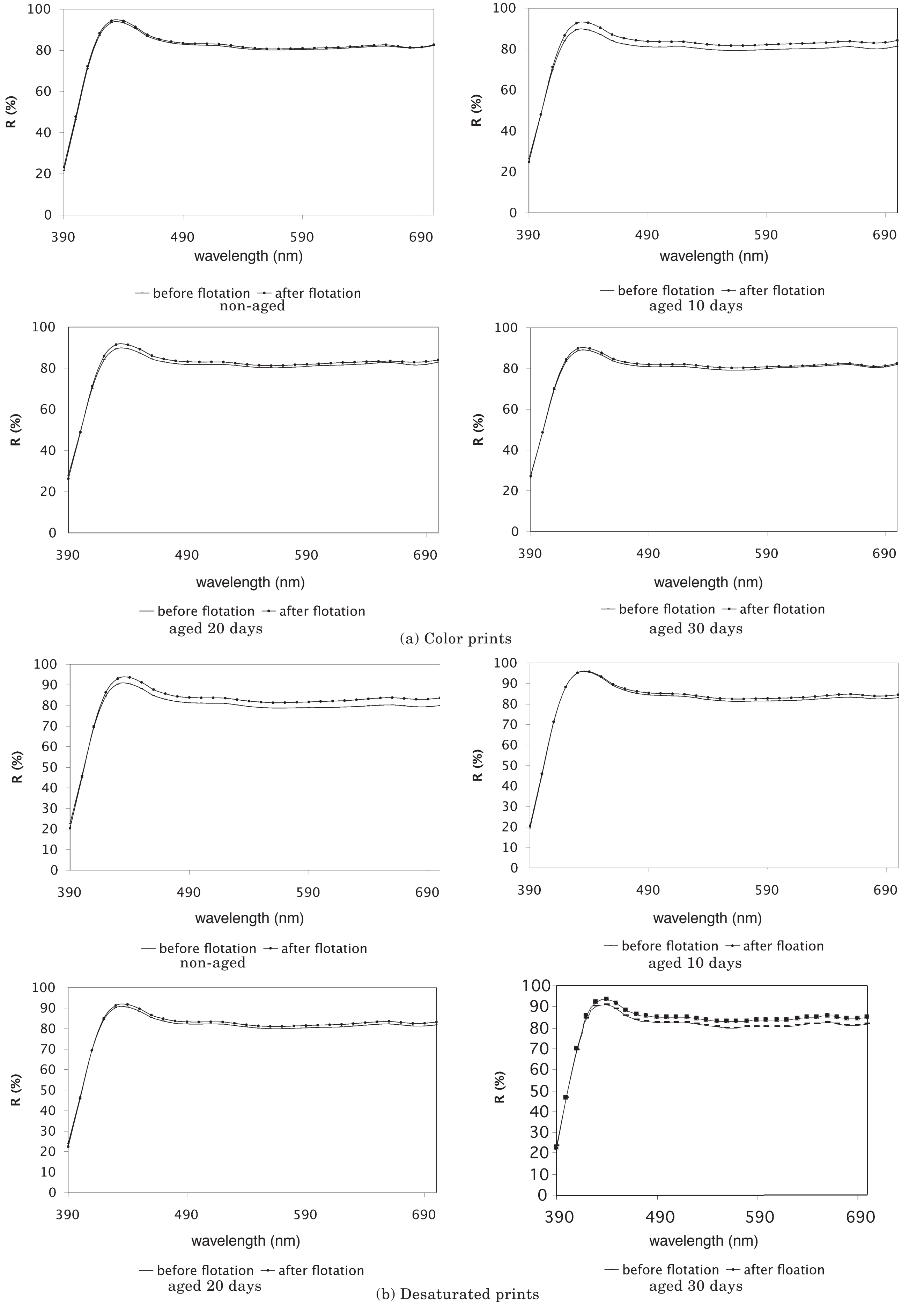
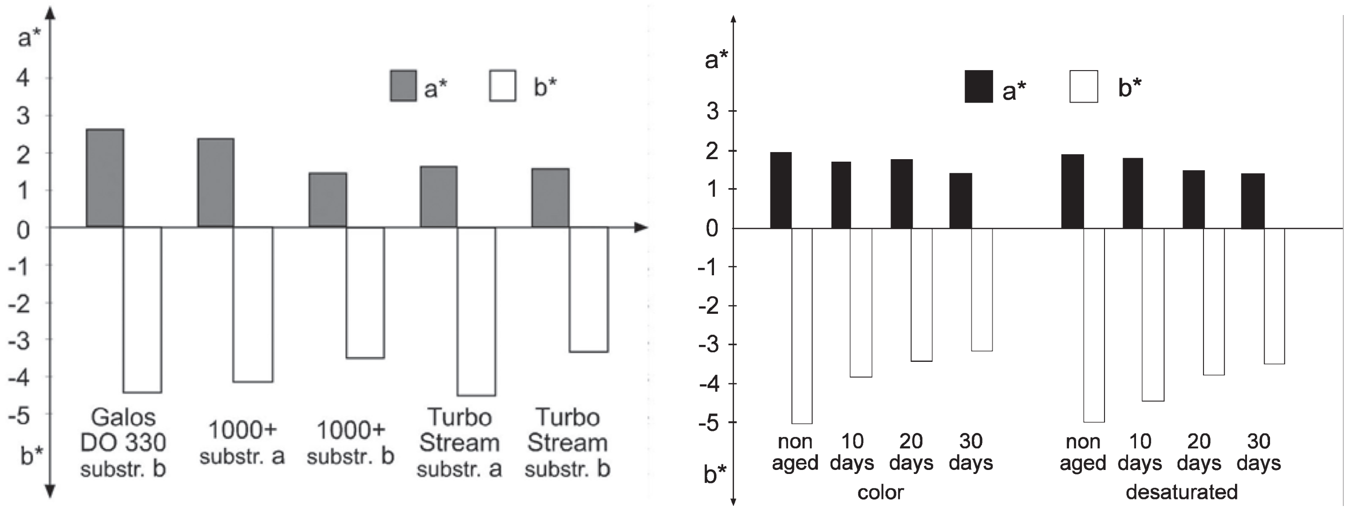
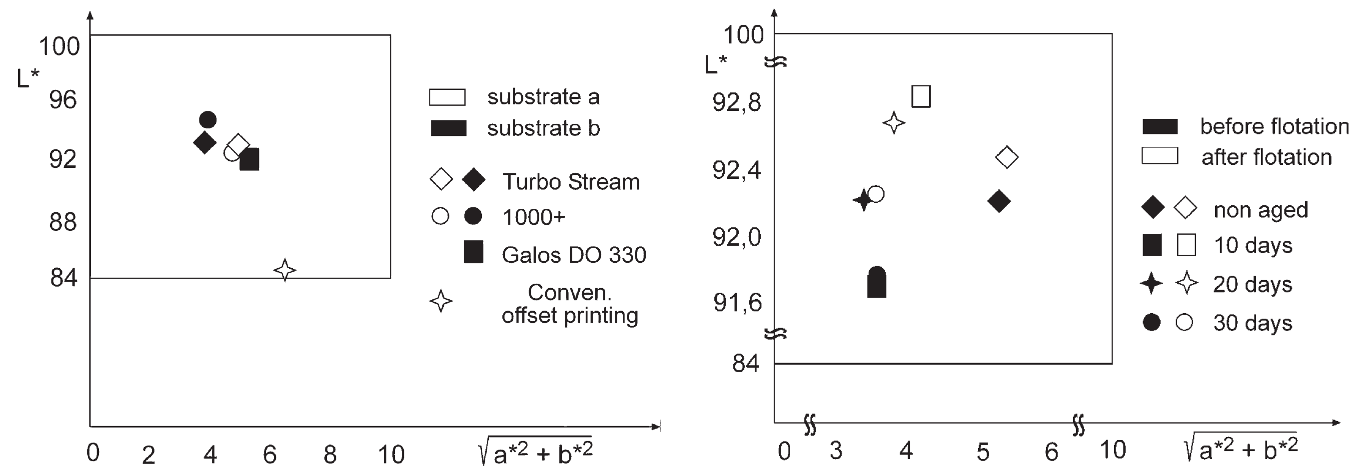


Figure 6. Spectral reflectance curves for handsheet from accelerated aged multicolor and desaturated digital offset prints



(a) different models of digital offset machine (b) accelerated aged prints

Figure 7. *a*b** plot for handsheets from digital offset prints after flotation



(a) different models of digital offset machine (b) accelerated aged prints

Figure 8. Lightness versus chroma

of handsheets is obtained when color and desaturated prints are used in recycling.

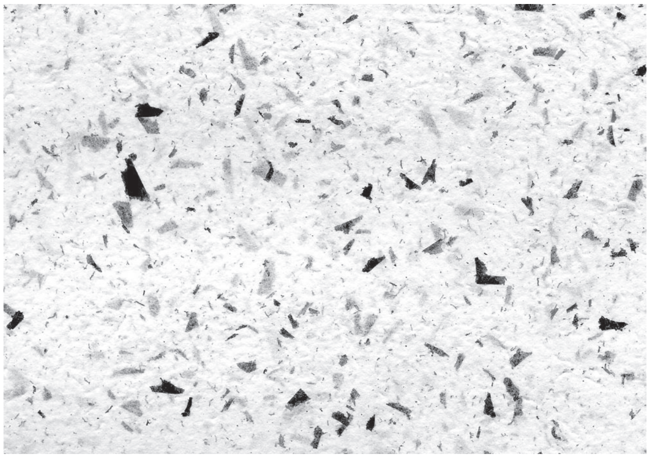
An interesting variable reflecting the removal of color from prints, with respect to the possible usage of the recycled fiber for the production of fine graphic paper, is embodied in the definition of the white and very near white paper. White or near white papers are those for which $L^* > 84$ and $(a^{*2} + b^{*2})^{1/2} < 10$, according to TAPPI Test Method T524. In Fig. 8(a), the results obtained for handsheets after recycling of prints made on different digital offset machines and on different printing substrates are presented. The same test method was used for handsheets made before and after flotation of the non-aged and prints accelerated aged for different periods of time (Fig. 8(b)).

From the results presented, one can see that the stages of the process used for deinking flotation had little effect on color. This means that while the deinking process can remove inks it has little effect on dyes. Furthermore, in an experiment in which hydrogen peroxide was used the expected color stripping function was not observed.

Image analysis is useful for detecting optical inhomogeneities. Generally, these are dirt specks, including ink particles, in the handsheet. Figure 9 presents a part of results, those which can demonstrate in the best way the influence of incoming materials on the success of the processes under study.

General characteristics of the results presented in Figs. 9(a) and 9(b) include the presence of greater numbers of bigger particles of ElectroInk in one case as opposed to the presence of numerous very tiny particles of conventional offset ink on handsheet after pulping in the other case. This fact can be explained by relative weak efficacy of flotation, which also is confirmed by the observed optical characteristics of the handsheets before and after recycling. The reason for such particle distributions can be looked for in the principles of the printing techniques used, some details in which machines for digital offset printing mutually differ, as well as in characteristics of the substrate employed.

As is known, the principle of digital offset printing is identical for all the models of these machines. However,



Dirt Spot Size	Count	Area (sq.mm)
>= 5.000	1	6.703
4.00--5.00	5	22.233
3.00--4.00	3	9.858
2.50--3.00	2	5.289
2.00--2.50	6	13.143
1.50--2.00	6	10.057
1.00--1.50	12	14.319
0.80--1.00	12	10.661
0.60--0.80	12	8.210
0.40--0.60	26	12.763
0.30--0.40	13	4.625
0.25--0.30	9	2.448
0.20--0.25	8	1.740
0.15--0.20	21	3.613
0.10--0.15	36	4.554
0.09--0.10	9	0.851
0.08--0.09	11	0.941
0.07--0.08	6	0.457
0.06--0.07	20	1.310
0.05--0.06	25	1.375
0.04--0.05	37	1.636
0.03--0.04	46	1.570
0.021--0.03	69	1.753
0.013--0.021	117	1.955
0.006--0.013	322	3.045
0.001--0.006	679	2.036

(a) handsheets after pulping of prints from digital offset machine and substrate b

Dirt Spot Size	Count	Area (sq.mm)
>= 5.000	0	
4.00--5.00	0	
3.00--4.00	0	
2.50--3.00	0	
2.00--2.50	0	
1.50--2.00	0	
1.00--1.50	0	
0.80--1.00	0	
0.60--0.80	0	
0.40--0.60	0	
0.30--0.40	0	
0.25--0.30	0	
0.20--0.25	0	
0.15--0.20	0	
0.10--0.15	1	0.122
0.09--0.10	1	0.091
0.08--0.09	3	0.254
0.07--0.08	2	0.147
0.06--0.07	8	0.516
0.05--0.06	16	0.867
0.04--0.05	21	0.930
0.03--0.04	72	2.473
0.021--0.03	153	3.749
0.013--0.021	214	3.559
0.006--0.013	558	5.084
0.001--0.006	1615	4.781

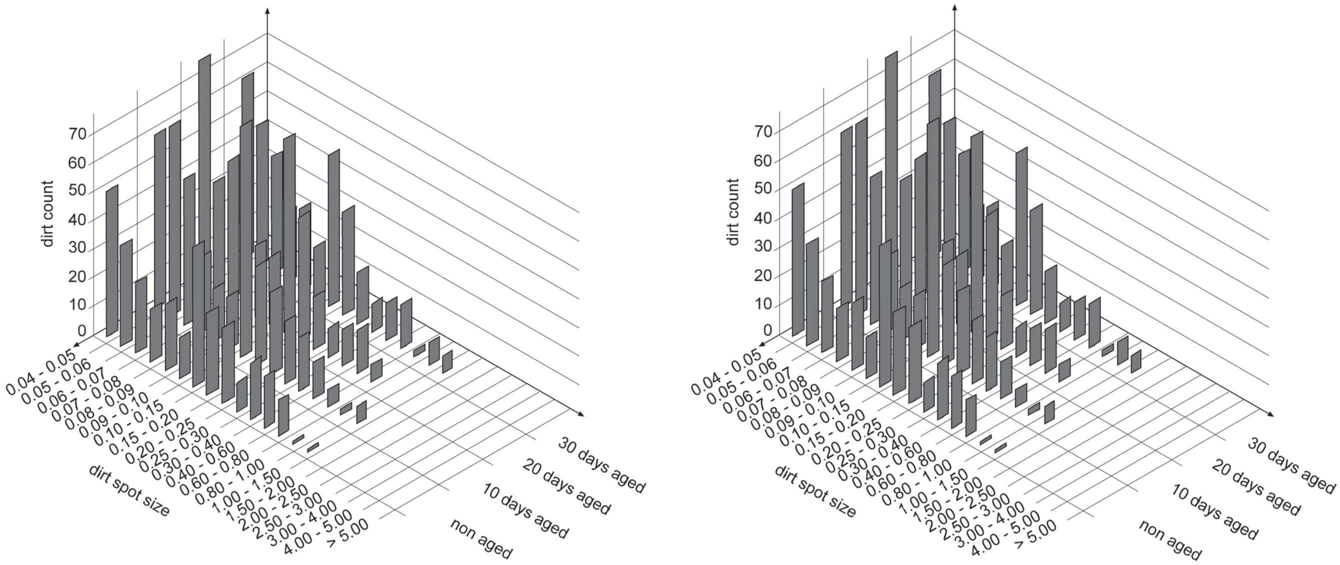
(b) handsheets after pulping of prints from conventional offset machine and substrate a

Figure 9. Size distribution of dirt particles after pulping

there is a difference between this technique and classical offset in the drying of the prints, which can be the cause of appearance of larger ink particles on the handsheets. In digital offset printing the ElectroInk is laminated into an ink–polymer film, and peeled off the blanket and applied to the paper with help of transfer oil. This process occurs sequentially for each color. Thus in conventional printing the ink binds directly with the paper, but the ElectroInk dries to a film on the blanket before it reaches the substrate and does not penetrate into the paper.

Also there are differences in operational detail among the various machine models using the Indigo digital technique. The differences among series 1 and 2 include the application of binary ink development technology in the latter one. In this technology there are inking rollers which transfer ink onto the base cylinder in one layer; the developer units are separated for each ink which results in a faster working speed for the machine. There are also differences in the second transfer of ElectroInk depending on the machine model. The sheet fed machines use so-called multishot technology, which means that the sheet of paper repeatedly passes through the machine until all colors are printed. Such a process can not be performed in web printing. In a web digital printing machine one-shot technology is used, which means that the inks are superimposed one above the other on the transfer roller, rather than on the printing substrate. In this case all the colors are transferred on the printing substrate simultaneously.

Ink particles, categorized according to size, for the handsheets made after disintegration of color and desaturated aged digital offset prints are presented in Fig. 10.



(a) Accelerated aged multicolor digital prints (b) Accelerated aged desaturated digital prints

Figure 10. The influence of the aging process on residual ink particle size

As can be seen from these results, the number of particles in lower size classes has increased with aging in relation to macro particles. However this trend is not unambiguous over the investigated periods of 10, 20, and 30 days, as is also illustrated by the data presented in Table III.

From the results presented in Table III the increased dirt count and area in both size classes >0.04 mm^2 and <0.04 mm^2 for handsheets made after the disintegration of the aged desaturated prints in relation to the non-aged ones is visible. The process of print aging generally has less influence on the increase of dirt count >0.04 mm^2 (at mostly 28.1%) in relation to the dirt count <0.04 mm^2 (at mostly 49.2% in dirt count, and 55.1% in dirt area respectively). By accelerated aging of prints for 10 days, the share of dirt count <0.04 mm^2 has been increased 46.4% after disintegration and by 65.4% on the handsheet after flotation, relative to the non-aged print. Greater differences in the dirt count area have not been observed on further increasing aging time to 20 days. By further increase of the accelerated aging time to 30 days, a decrease in dirt count and area, in relation to the print with accelerated aging for 20 days, has been noticed. However the increase of the total dirt count by 46.1% and the total dirt area by 5.2% in relation to the non-aged print is still apparent. It is interesting in this case, that the area occupied by the particles >0.04 mm^2 is smaller by 10.5%, and the surface of the particles <0.04 mm^2 is greater by 50.8% in relation to the recycled non-aged print.

The results obtained on the particle size distribution and the number of particles as influenced by the duration of the process of the accelerated aging of digital prints explain the observed values of recycled handsheet brightness. Particles in lower size classes, the number of which decreases on increasing the period of accelerated aging of prints, contribute to the increase in handsheet brightness.

Generally, a better quality secondary raw material can be obtained by mixing a smaller proportion of digital offset prints with conventional ones whether using conventional deinking flotation or using the enzymatic deinking.[24,25]

TABLE III. Dirt Count and Particle Area in Dependence on Accelerated Aging of Handsheets

(a) After Disintegration

	Dirt count >0.04 mm^2%	Dirt count <0.04 mm^2%	Area >0.04 mm^2%	Area <0.04 mm^2%
Non-aged	323	1316	54043	11593
Aged 10 days	449	2457	64359	24366
Aged 20 days	399	2626	53273	25823
Aged 30 days	371	2338	53226	22647

(b) After Flotation

	Dirt count >0.04 mm^2%	Dirt count <0.04 mm^2%	Area >0.04 mm^2%	Area <0.04 mm^2%
Non-aged	179	607	35595	5799
Aged 10 days	276	1754	37925	16809
Aged 20 days	264	1700	35910	16835
Aged 30 days	210	1248	31873	11783

Conclusions

Based on the investigation results, we conclude that particles of ElectroInk of different sizes and shapes relative to the particles of the conventional offset ink are formed during the processes of disintegration of prints from a digital printing process based on electrophotography and from conventional offset printing. The particles of ElectroInk are flat and have a greater surface which decreases the efficacy of the flotation process.

It has been proven that the quality of the recycled fibers indicated by the values of optical responses and image analysis of handsheets varies among the source prints obtained from different models of Indigo digital offset printing machines, which differ among each other only in details.

It has also been found that by processing accelerated aged prints by the method of alkali chemical deinking flotation, the dirt count and the area on handsheet was influenced before and after flotation. In subsequently recycled prints accelerated aged for 10, 20, and 30 days, the increase of dirt count <0.04 mm^2 and the area occupied by these particles on handsheets cast before and after flotation should be specially pointed out in relation to the non-aged print which influences the observed optical properties of handsheet.

The process of accelerated aging has a different influence on the observed optical parameters (brightness, a^*b^*, lightness versus chroma) and on dirt count and area, of handsheets obtained by deinking flotation of color prints in comparison to desaturated ones.

Handsheets obtained after the recycling of all of the digital prints satisfy the criteria of white or near white substrate for graphic applications; however the optical homogeneity of handsheets from recycling of non-aged digital offset prints can be noticed.

The results verify that by measuring the dirt count and area and optical parameters over all the processing phases, an understanding of the process of ElectroInk removal and of the way to modify this process can be obtained in order to achieve the basic aim of improving the quality of the recycled fibers.

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