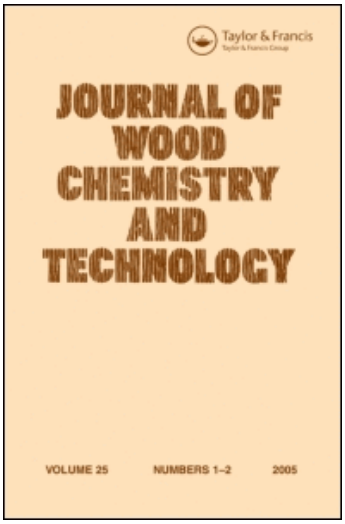

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Retention of Optical Brightening Agents (OBA) and their Brightening Efficiency on HYP-Containing Paper Sheets

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Abstract: Optical brightening agents (OBA) or fluorescent whitening agents (FWA) are commonly used in the paper industry to improve the optical performance of paper products. The increased use of bleached chemi-thermo-mechanical pulp (BCTMP) or high-yield pulp (HYP) in printing and writing paper grades has initiated research topics on the brightening efficiency of OBA on mechanical pulp-containing furnishes. In this study, process parameters that may affect the retention of OBA were investigated, such as furnish composition, OBA charge, contacting time, water hardness, anionic trash, and fines in HYP. It was found that OBA had lower retention on HYP fibers than on bleached kraft pulp (BKP) fibers. Efforts were also made to understand the underlying mechanism.

Keywords: Optical brightening agent (OBA), fluorescent whitening agent (FWA), effectiveness, OBA retention, HYP, BCTMP, brightness, CIE whiteness, printing and writing paper

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INTRODUCTION

High-yield pulp (HYP) or bleached chemi-thermo-mechanical pulp (BCTMP) is being used at an increasing rate to replace bleached hardwood kraft pulp in the manufacturing of higher-quality paper products due to cost advantages and its unique properties, for example, high bulk, good opacity, and stiffness.[1] However, HYP or BCTMP usually has lower brightness and a yellowish color shade, when compared with bleached kraft pulp. Brightness is one of the most important quality criteria for high value-added paper products. Therefore, optical brightening agents (OBA), which are also called fluorescent whitening agents (FWA), are used to improve the optical properties of paper products.[2]

OBA can absorb ultraviolet light and re-emit it as bluish light. As a result, paper with OBA looks brighter and whiter when it is exposed to daylight or normal office light that contains UV light. Because of their small molecular sizes, OBAs are known to be absorbed by pulp fibers through the pore openings. The mobility of the OBA molecule within the cell wall enables it to diffuse through the wall structure and become fixed through hydrogen bonding without the need of cationic additives. The fiber-OBA hydrogen bonds are generally formed through the hydrogen of the hydroxyl or amine groups with the oxygen or nitrogen of other polar groups.[2–4] In addition, the application of OBA can reduce the light-induced color reversion by reducing the intensity of UV radiation on fibers.[5,6]

In fine paper manufacturing, HYP can be used to substitute a portion of hardwood bleached kraft pulp. Bleached mechanical pulps contain many impurities such as oxidized lignin, dispersed wood resin, and fatty acids, and other dissolved and colloidal substances (DCS).[7–10] Part of these impurities bear negative charges and are known as anionic trash, which has been found to be detrimental to the papermaking process.[2,10,11] In addition, PCC filler is commonly used in the manufacturing of fine paper, which may also affect the efficiency of OBA application.

Most of the literature regarding the use of OBA was related to chemical pulps.[12–15] Recently we started a project to improve the optical properties of high-brightness HYP by using OBA.[16,17] The objective of this article was to study the retention of OBA on HYP and its brightening efficiency under various wet-end conditions of the papermaking process, and to understand why OBA usually has a lower retention on HYP than on bleached kraft pulp.

EXPERIMENTAL

A softwood (mainly spruce) bleached kraft pulp (SW BKP), and a hardwood (eucalyptus) bleached kraft pulp (HW BKP) were refined in a PFI mill to 470 and 490 ml CSF freeness, respectively. Three commercial aspen high-yield pulps (HYP 325/85, 325/83, and 250/80) were obtained from a mill in

Western Canada, without further refining treatment. The properties of the pulp samples were listed in Table 1. A precipitated calcium carbonate filler (Albacar HO) was obtained from Specialty Minerals, an optical brightening agents (Tinopal ABP-A) from Ciba, and a cationic polyacrylamide (Percol 239), also from Ciba Specialty Chemicals. A DCS-free HYP sample was prepared by dispersing 3 grams of HYP into a 1% pulp suspension and filtering on a 500 ml Büchner funnel with a 200-mesh Teflon screen. The filtrate was recycled to go through the fiber mat to collect the fines, and then the pulp pad was washed 2 times on the funnel with 100 ml deionized water each.

Fines- and DCS-free HYP sample was prepared using a dynamic drainage jar (DDJ) according to a TAPPI standard method (T261).

Tinopal ABP-A is a tetra-sulfonated fluorescent whitening agent that is most commonly used for wet-end addition in the pulp and paper industry. Unless specified, Tinopal ABP-A was used for all the experiments in this study, and its dosage was based on the liquid product (about 25% solid content). Weighed pulp samples (SW BKP, HW BKP, and HYP), were disintegrated for 15,000 revolutions in a standard disintegrator at 1.5% pulp consistency, and then diluted to 1% suspension. An aliquot of the pulp suspension was transferred to a 500 ml beaker, and $CaCl_2$ solution was added to reach a desired Ca^{2+} concentration. The pH of the mixture was adjusted to about 7.0, followed by the OBA addition. Magnetic stirring was provided for 0–20 min under room temperature. When needed, PCC filler (dispersed into a suspension) was then added, followed by the addition of CPAM (0.05%, on pulp). Subsequently handsheets were prepared and tested according to the TAPPI methods (T205 and T220) on a TechniBrite^{TM} Micro TB-1C brightness meter.

We developed a method to determine the retention rate of OBA on fibers by difference. The amount of OBA retained on fibers equals to the amount added minus the amount found in the filtrate. The amount of OBA in the filtrate was determined by following a UV spectroscopy method. As shown in Figure 1, fresh OBA solution has two specific absorption peaks at 280 and 350 nm. However, in a diluted solution, OBA undergoes a transformation from trans- to cis-configuration.[2] For this reason, the absorbance of OBA at 350 nm decays over time. As shown in Figure 1, the absorption peak at 350 nm

Table 1. Characteristics of the pulp samples used for the experiments

	CSF (ml)	Brightness (%)	CIE whiteness (%)	L*	a*	b*
SW BKP	470	87.2	73.6	97.2	−0.72	4.28
HW BKP	490	88.4	76.2	97.4	−0.60	3.84
HYP (325/85)	510	85.5	66.7	97.2	−1.43	5.77
HYP (325/83)	540	83.7	61.7	96.9	−1.55	6.73
HYP (250/80)	450	81.2	55.0	96.6	−1.48	7.97

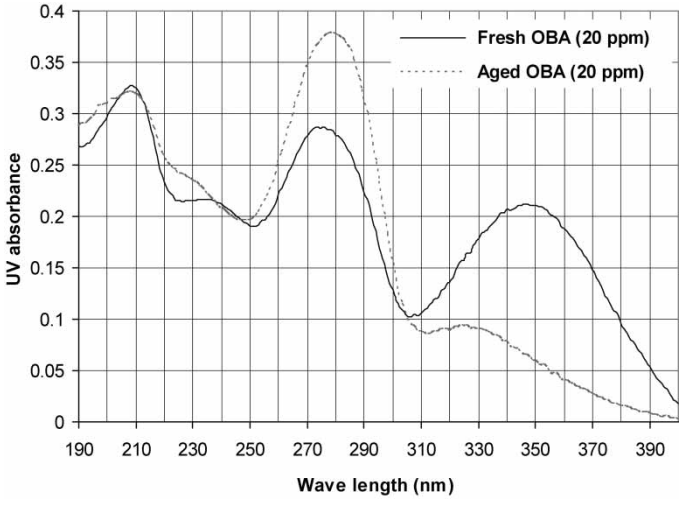

Figure 1. Light absorbance of fresh and aged (2 days, at room conditions) OBA samples (Tinopal ABP-A).

almost disappeared after two days of aging at the room conditions, and accordingly the absorbance at 280 nm increased considerably. The transformation of OBA seems to reach completion within two days under the studied conditions, and the aged solution has an intensive and stable absorbance at 280 nm that can be used for the quantitative analysis. A calibration curve was then constructed by preparing a series of OBA standard solutions that were let stay for two days, and measured for their UV absorbance at 280 nm. Figure 2 shows that the absorbance of OBA at 280 nm linearly correlates to its concentration. The absorptivity of aged OBA solution at 280 nm was determined to be 18.7 L/g/cm. Independent control trials (i.e., without the addition of OBA) showed that the UV absorbance due to lignin, and other substances in the filtrate, was negligible (less than 1%).

Filtrate samples from the handsheet making process were further filtered with a 0.1 μ membrane filter to remove micro particles, to avoid their interference with the UV measurement. The membrane filters were pre-saturated with OBA solution of concentrations similar to those of the filtrate samples and rinsed with deionized water to remove free OBA from the membrane. Desorption of OBA from the membrane was found to be a much slower process compared with the filtration process. The first 100 ml of filtrate was discarded to minimize the change of OBA concentration due to the filtration process. The membrane-filtered filtrate samples were kept under the room conditions for 2 days before the measurement of their UV absorbance at 280 nm. The filtrate from the control pulp samples without the OBA addition was used as the blank. The OBA concentration in the filtrate samples was determined according to their UV absorbance and the established

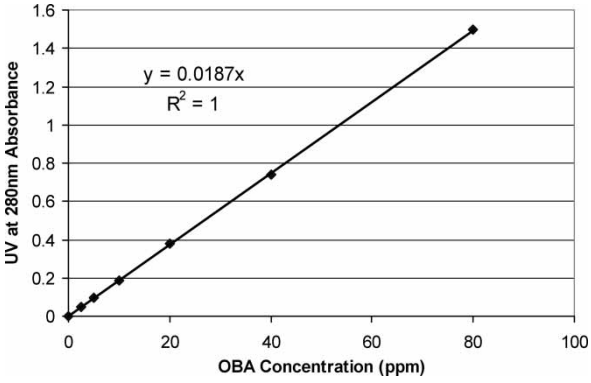

Figure 2. Calibration curve for the aged OBA solutions.

calibration curve. The OBA retention on pulp fibers were then calculated by mass balance.

For the fluorescent microscopy study, pulp fibers were gradually dehydrated with ethanol/water solutions and embedded in resin before sectioning with a diamond knife. Sections of 1 μm thickness were observed under a fluorescence microscope with excitation at 340–380 nm, and re-emission larger than 425 nm.

RESULTS AND DISCUSSION

The Brightening Efficiency of OBA on High-Yield Pulp (HYP)

We found that OBA is less effective with HYP than with fully bleached kraft pulp. For example, at an OBA dosage of 1.0% the brightness gain for the HYP (Grade 325/83) was about 4 units, while an 8-unit brightness increase was obtained for a hardwood bleached kraft (HW BKP) under otherwise the same conditions. Possible reasons for this difference could be

(1) lower retention of OBA on HYP; (2) the competition of lignin with OBA for UV light; (3) the lower brightness of HYP; (4) the negative effect of the impurities of HYP (anionic trash, extractives).

We first examined the light reflectance of HYP and hardwood bleached kraft pulp (HW BKP) with and without OBA addition. Figure 3 shows their differential light reflectance. As expected, for both HYP and HW BKP the reflectance decreased in the near UV region but increased in the blue light region due to the fact that OBA absorbed UV light and re-emit it as blue light. However, the absorption of UV light and the re-emission of blue light

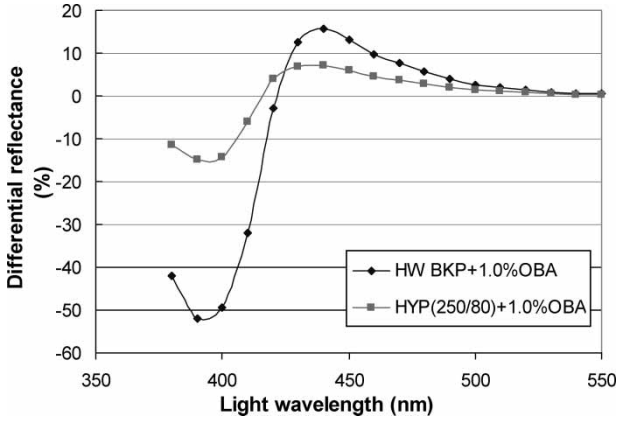

Figure 3. Differential light reflectance of handsheet samples with and without the OBA addition.

were much more intensive for the HW BKP than for the HYP, indicating that less OBA was retained in the HYP sheet or that its optical effects were being suppressed.

OBA Retention and its Effect on the Brightening Efficiency

We also determined the retention of OBA on HYP and HW BKP. As shown in Figure 4, the optical properties of the HYP-containing furnish improved significantly with OBA. For example, with 0.6% OBA, the brightness increased from 84.3% ISO to 90.5% ISO, and CIE whiteness from 68.1% to 87.0%. Figure 4 also shows that the brightening efficiency of OBA deceased, in particular when its dosage exceeded 0.6%.

Figure 5 shows the retention rate of OBA as a function of the OBA charge, based on the absorbance of the filtrate. The higher the OBA charge, the lower the retention rate of OBA. At 0.5% OBA, the OBA retention was higher than 90%, but it dropped to about 75% when the OBA charge was increased to 2%. However, the OBA retention on HYP-containing furnish was always lower than on 100% BKP (0% HYP).

We offered the following explanations to account for the lower OBA retention in the presence of HYP. Firstly, HYP fibers retain most of the lignin from wood, thus having less pore openings than kraft pulp fibers. Consequently, OBA cannot readily diffuse into fiber structures of the HYP pulp. The above hypothesis will be further discussed later in this article (based on the evidence from fluorescence microscopy). Secondly, HYP contains more fines and anionic trash, which may also contribute to the lower OBA retention.

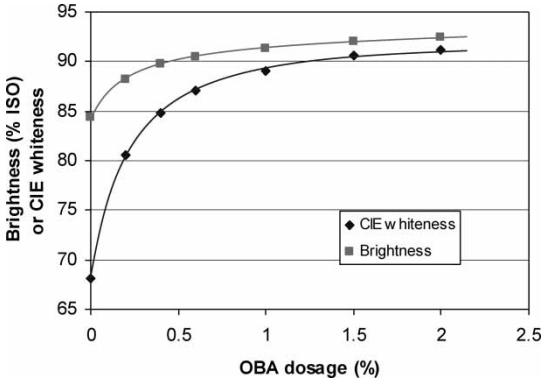

Figure 4. Effect of OBA dosages on the brightness and CIE whiteness of HYP-containing paper sheets (30% SW BKP + 40% HW BKP + 30% HYP (Grade 325/83); 1% pulp consistency; 0–2% OBA; 100 ppm Ca^{2+}; magnetic stirring for 20 minutes).

Effect of Contact Time and Ca^{2+} Concentration

OBA molecules can absorb on fibers through hydrogen bonds without the assistance from cationic additives. Figure 6 shows that the brightness and fluorescent composition of handsheet samples increase with contact time but level off after 5 min. It seems that a minimal contact time is required to ensure that there is enough time for OBA molecules to complete the interaction with pulp fibers. Under the experimental conditions (1% pulp consistency, 100 ppm Ca^{2+}, with stirring), a 20-min contact time seems to have been sufficient for the

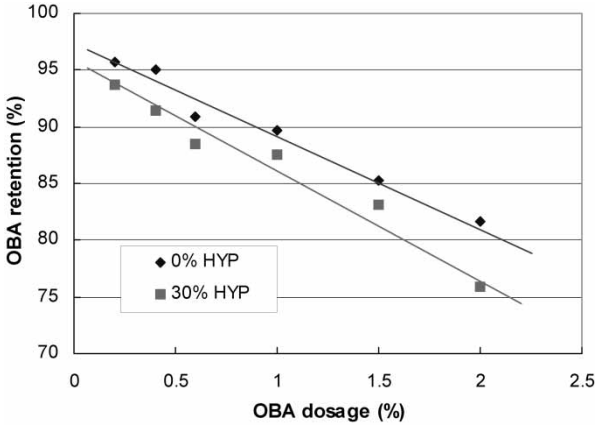

Figure 5. Retention rate of OBA on fibers versus OBA dosages (30% SWBKP + 40% HWBKP + 30% HYP (Grade 325/83); 1% pulp consistency; OBA 0–2%; 100 ppm Ca^{2+}; magnetic stirring for 20 minutes).

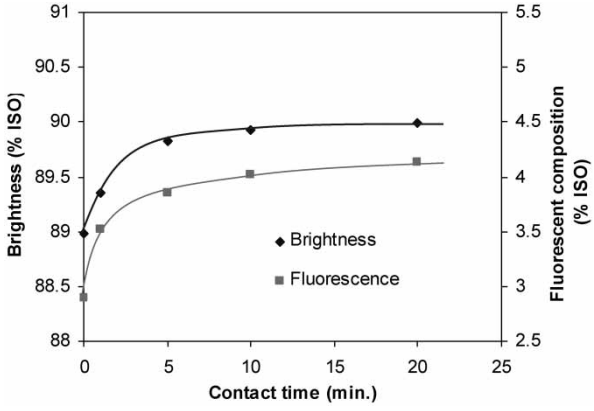

Figure 6. Effect of contact time on brightness and fluorescent composition (30% SWBKP + 40% HWBKP + 30% HYP (Grade 325/83); 1% pulp consistency; 0.2% OBA; 100 ppm Ca^{2+}; magnetic stirring for 20 minutes).

absorption and fixation to complete. The contact time may be shortened by increasing temperature but with the risk of thermal yellowing of HYP.

It is well known that pulp fibers bear negative charges. The OBA molecule has four sulfonic acid groups. Electrostatic repulsion occurs when anionic OBA is brought into contact with these negatively charged pulp fibers. The presence of metal ions such as Ca^{2+} and Mg^{2+} may be able to reduce the electrostatic repulsion between OBA and fibers by binding with the sulfonic groups.[2,3] In Figure 7, various amounts of Ca^{2+} ions were added to the pulp suspension before the addition of OBA. The results show that Ca^{2+} ions were very effective in improving the brightening efficiency of OBA. For example, without the addition of Ca^{2+}, the brightness gain

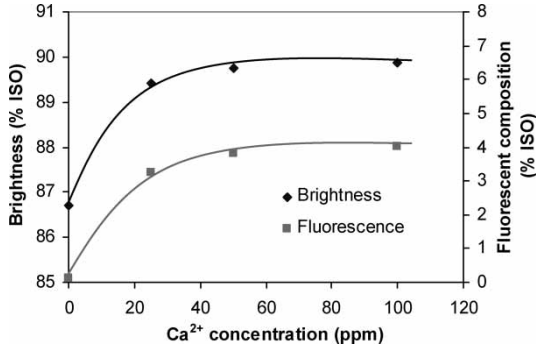

Figure 7. Effect of Ca^{2+} concentration on brightness and fluorescent composition (30% SWBKP + 40% HWBKP + 30% HYP (Grade 325/83); 1% pulp consistency; 0.2% OBA; 100 ppm Ca^{2+}; magnetic stirring for 20 minutes).

from fluorescence were only about 0.1% ISO; it increased to 3.23% ISO with 25 ppm Ca^{2+}. Figure 7 shows that the fluorescent composition and brightness increased with water hardness (Ca^{2+} concentration), but leveled off when Ca^{2+} concentration was higher than 50 ppm.

Effect of Furnish Composition

Figure 8 shows the retention rate and the brightening efficiency of OBA at various substitution rates of HYP (Grade 325/83) for hardwood bleached kraft pulp. The brightness gain from OBA deceased with HYP substitution rate. The retention rate of OBA decreased with the increase of HYP substitution rate. However, the brightening efficiency of OBA (in terms of brightness gain) does not always follow the retention curve, although a decreasing trend was observed for both of them. These results suggest that the brightness gain from the OBA fluorescence is not always proportional to its amount retained on fibers. Part of the reason is that at a higher charge, OBA may be aggregated, leading to the formation of eximer complexes. In addition, the properties of HYP such as DCS, fines, and lignin contents may also affect the OBA effectiveness.

Effect of Anionic Trash and Fines

HYP is known to contain dissolved and colloidal substances (DCS) and anionic trash[2,7–9] that may interfere with the adsorption of OBA on fibers. Washing is an effective way to remove anionic trash from pulp.[8–10] In order to verify whether anionic trash in HYP is responsible for the decreased OBA retention, experiments were performed on the DCS-free

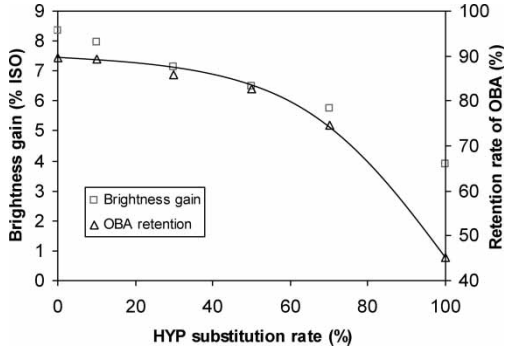

Figure 8. Effect of HYP substitution for HW kraft pulp on the OBA retention and its effectiveness in improving brightness. (100–0% BKP + 0–100% HYP (Grade 325/83), with SW BKP fixed at 30% except for the case of 100% HYP substitution; 1% pulp consistency; 1.0% OBA; 100 ppm Ca^{2+}; magnetic stirring for 20 minutes).

HYP. The results in Table 2 show that at the same OBA dosage, the retention of OBA was slightly higher for the DCS-free HYP than for the control (regular HYP). As a result, better optical properties of paper sheets were obtained, although the improvement was small.

It is also known that HYP contains much more fines than bleached kraft pulps, which may also affect the retention of OBA. Having a much larger specific surface area than fibers, fines may adsorb a larger amount of OBA and have a lower retention rate than fibers during the papermaking process. To investigate whether the fines of HYP contribute to the observed effect of HYP on the OBA retention and its brightening efficiency, OBA was applied to a fines- and DCS-free HYP sample. As shown in Table 2, at 0.5% OBA dosage, removing fines from the DCS-free HYP sample improved further the retention of OBA and thus its brightening efficiency. However, the improvement was marginal when OBA dosage increased to 1.0%.

Based on the aforementioned results, one can draw the conclusion that the anionic trash and fines present in HYP have only small effects on the OBA retention and its brightening efficiency.

Table 2. Effect of anionic trash and fines on the OBA retention

Pretreatment	OBA dosage (%)	0	0.5	1.0
Control (regular HYP)	L*	96.09	96.48	96.67
	a*	−1.30	−0.38	−0.17
	b*	6.49	4.42	3.83
	CIE WH (%)	60.80	71.05	74.22
	Brightness (%ISO)	82.82	85.70	87.02
	Fl. comp. (%ISO)	0	2.91	3.92
	OBA retention (%)	—	58.49	41.92
DCS-free HYP (with fines)	L*	96.48	96.62	96.60
	a*	−1.43	−0.26	−0.16
	b*	6.85	4.29	3.27
	CIE WH (%)	59.90	72.10	75.39
	Brightness (%ISO)	82.61	85.93	87.50
	Fl. comp. (%ISO)	0	3.15	4.14
	OBA retention (%)	—	62.09	47.69
Fines- and DCS-free HYP	L*	96.38	96.51	96.54
	a*	−1.30	−0.35	−0.07
	b*	6.40	3.97	3.25
	CIE WH (%)	61.73	73.19	76.52
	Brightness (%ISO)	82.87	86.42	87.53
	Fl. Comp. (%ISO)	0	3.41	4.44
	OBA Retention (%)	—	71.09	47.80

Conditions: 100% HYP (Grade 325/83); 1% pulp consistency; 0–1.0% OBA; 100 ppm Ca^{2+}; magnetic stirring for 20 minutes.

Effect of PCC Filler on the OBA Retention and its Brightening Efficiency

In the manufacture of printing and writing paper grades, mineral fillers such as precipitated calcium carbonate (PCC) are commonly used to improve the optical properties of paper products, as well as to reduce the production cost. It is important to know how the OBA retention and its brightening efficiency can be affected by the addition of PCC to a HYP-containing furnish.

As shown in Table 3, PCC filler improved the optical properties of HYP-containing paper sheets. With 0.6% OBA and 30% PCC, the brightness and CIE whiteness increased from 90.5% ISO and 87.0% to 91.1% ISO and 89.4%, respectively, with 0.6% OBA and 30% PCC. However, the fluorescent composition (brightness gain due to fluorescence) decreased from 6.24% ISO to 4.64% ISO, indicating that the addition of PCC filler had a negative effect on the brightening efficiency of OBA. This was not caused by poorer OBA retention, as the results in Table 3 show that the addition of PCC filler had negligible effect on the OBA retention. Rather, the effectiveness of OBA decreased when PCC was present in the paper. It is known that fillers can increase the light scattering coefficient and opacity of paper sheets as filler particles have much larger specific surface areas. For the same reason, PCC particles in paper sheets can scatter UV light and shorten the distance through which UV light can travel to reach OBA that was distributed across the sheets.

Fluorescence Microscopy Evidence

Based on the discussed results, we drew the conclusion that the characteristics of HYP fibers are mainly responsible for the observed difference in OBA retention and its brightening efficiency between the HYP and bleached kraft pulp. Lignin content is the main difference between the two types of fibers.

Table 3. Effect of PCC filler on OBA retention

PCC (%)	OBA dosage (%)	L*	a*	b*	CIE whiteness (%)	Brightness (%ISO)	Brightness gain from OBA (%ISO)	OBA retention (%)
0	0.2	96.4	0.33	2.31	80.5	88.2	3.82	93.6
	0.6	96.5	0.92	0.90	87.0	90.5	6.24	88.4
30	0.2	96.8	0.34	1.79	83.7	89.5	2.42	94.2
	0.6	96.6	0.98	0.44	89.4	91.1	4.64	87.8

Note: 30% SW BKP + 40% HW BKP + 30% HYP (Grade 325/83); 0% or 30% PCC filler; 1% pulp consistency; 0.2–0.6% OBA; 100 ppm Ca^{2+}; magnetic stirring for 20 minutes; 0.05% CPAM was added when PCC filler was applied.

Almost all lignin has been removed from BKP fibers during the pulping and bleaching processes. In contrast, HYP fibers retain almost all the lignin from wood. Lignin is hydrophobic in nature, and it hinders the adsorption and diffusion of hydrophilic OBA molecules on fibers. HYP fibers are less porous than BKP because lignin fills up the gaps between micro-fibrils in the cell wall. Therefore, OBA molecules have greater difficulty to diffuse through a compact cell wall of HYP fibers than a porous one of BKP fibers. To confirm the earlier hypothesis, fluorescence microscopy was used to study the distribution of OBA in the cross-section of fiber cell wall.

As shown in Figure 9, both HYP and BKP fibers dyed with OBA under the same conditions gave very strong fluorescence under UV illumination. The fluorescence appeared to be mainly due to OBA as lignin and other wood material generate only a small amount of fluorescent light with a wavelength larger than 425 nm under the conditions (Figure 10). From the intensity of fluorescent light in Figure 9, one can find that for the BKP fibers, OBA was distributed uniformly across the fiber cell wall, whereas for the HYP pulp fibers, the heterogeneous distribution of OBA was evident. A comparison of Figures 11 and 12 further showed that the fluorescence intensity is less in the middle and inner layers of the cell wall of the HYP fibers, indicating that the diffusion of OBA molecules across the cell wall of HYP fibers is

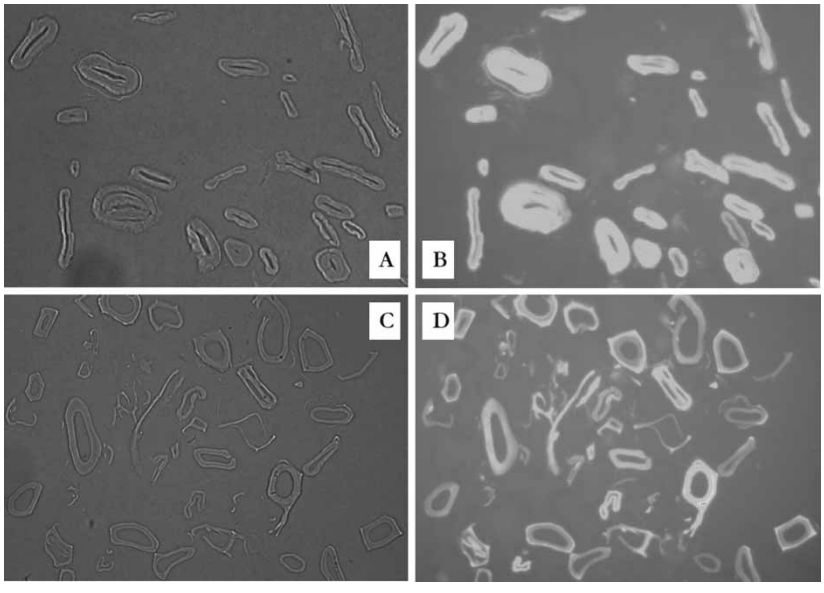

Figure 9. Comparison of the fluorescent images of the cross-section of HYP and kraft pulp fibers treated with OBA (×40). A. Bleached kraft pulp fibers without UV illumination; B. Bleached kraft pulp fibers with UV illumination; C. HYP fibers without UV illumination; D. HYP fibers with UV illumination.

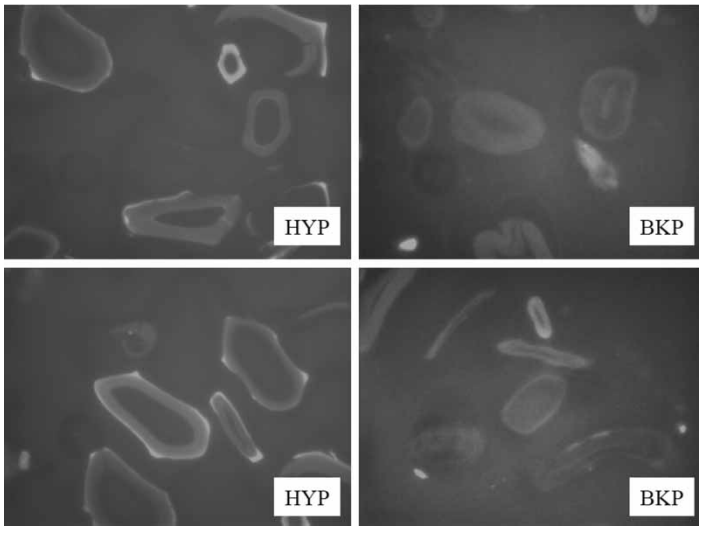

Figure 10. Fluorescent images of the cross-section of the bleached kraft and HYP fibers without OBA addition (×160, with UV illumination).

not as extensive as that for the BKP fibers. This is likely due to the hindrance of lignin and less porosity of HYP fibers.

Therefore, we concluded that the lower OBA retention on HYP fibers is mainly caused by the properties of HYP fibers.

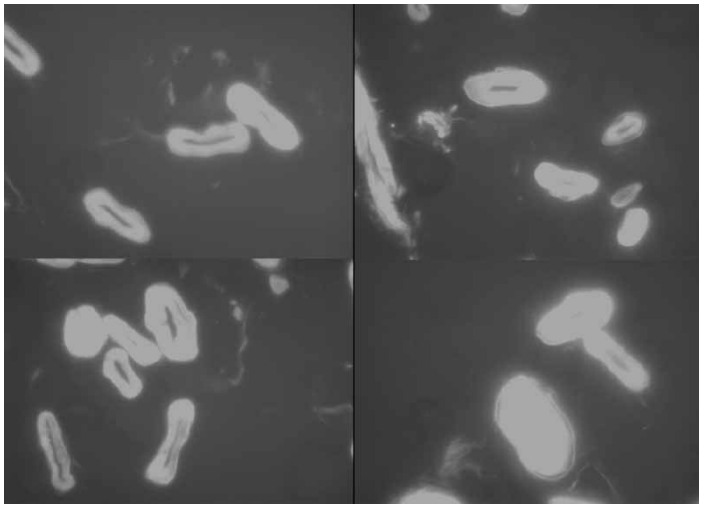

Figure 11. Fluorescent images of the cross-section of the bleached kraft pulp fibers dyed with OBA under UV illumination (×160).

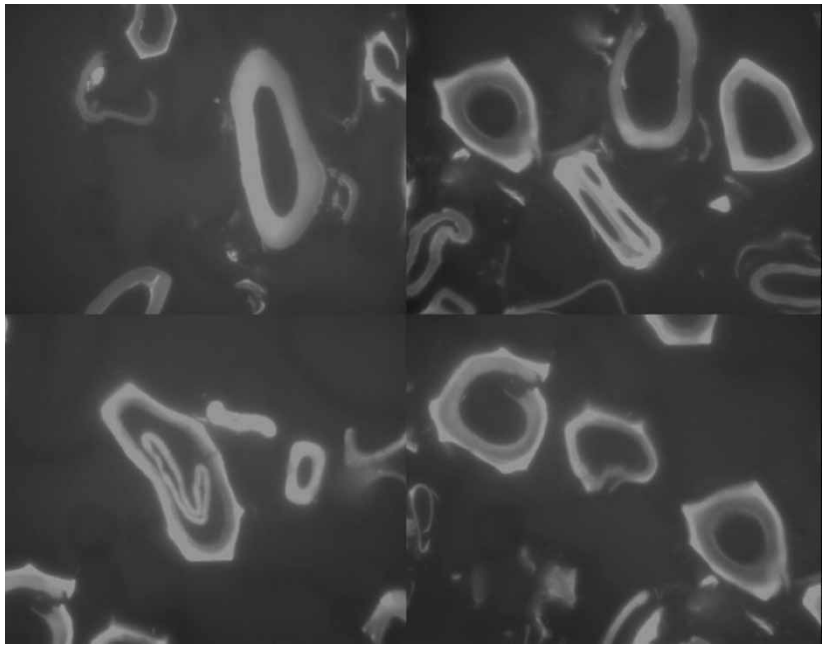

Figure 12. Fluorescent images of the cross-section of the HYP fibers dyed with OBA under UV illumination (×160).

CONCLUSIONS

The brightening efficiency of OBA depends on its retention and effectiveness in the paper sheets. The retention of OBA on fibers is affected by fiber composition, OBA dosage, contact time, and the hardness of the process water. A minimal of 5-min contact time and 25 ppm Ca^{2+} concentration are recommended for good performance of OBA. The OBA retention on HYP pulp was lower than that on BKP pulp mainly due to the physical and chemical properties of HYP fibers such as porosity and lignin content, although the anionic trash and fines in HYP also affect the OBA retention to some extent. The OBA retention was not affected by the PCC addition, but its effectiveness decreased when PCC filler particles were located in the paper sheets.

Results of fluorescence microscopy show that OBA is located mainly in the outer layer of the cell wall of HYP fibers, due to the fact that lignin hinders the diffusion of OBA. In contrast, a uniform distribution of OBA was observed for the BKP fibers across the cell wall thickness.

However, our results have shown that under the practical conditions of using HYP for the production of printing and writing paper (HYP substitution rate of less than 30%, OBA dosage of less than 0.5%), the OBA retention is

expected to be very high. For example, at a 30% substitution rate of HYP for HW BKP, the OBA retention was higher than 90% if the dosage of OBA was 0.5% or lower.

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