Photo-induced Yellowing of Mg(OH)$_2$-based Peroxide Bleached Deinked Pulp

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The effects of ultraviolet (UV) irradiation on the changes in color and chemical structure of the surfaces of unbleached, P_{NaOH}-, and $P_{Mg(OH)2}$-bleached deinked pulp (DIP) were studied by attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy. The analysis of color changes in pulp surfaces during the photo yellowing was performed by measuring the brightness loss, post color (PC) number, and CIELAB parameters (*L**, *a**, *b**, and Δ*E**). The results showed that the pulp brightness loss, PC number, and chromatic aberration had a linear relationship with *b**. During the UV irradiation, the pulp brightness loss, PC number, and chromatic aberration (Δ*E**) increased quickly, and then the changes slowed down. After being irradiated with UV for 360 min, the band intensity of the pulps at 1729 cm^{-1} increased distinctively and a new band at 1674 cm^{-1} appeared. This indicated that *p*-quinone groups were produced during the irradiation process, thus resulting in paper yellowing. The band intensity at 1674 cm^{-1} of $P_{Mg(OH)2}$-bleached pulp was lower than that of P_{NaOH}-bleached pulp, which indicated that the brightness stability of $P_{Mg(OH)2}$ pulp was better than that of P_{NaOH} pulp.

Keywords: Deinked pulp; Hydrogen peroxide bleaching; Mg(OH)$_2$; Photo yellowing; FTIR

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INTRODUCTION

Color reversion is caused by the effects of the storage environment, including temperature, relative humidity, UV irradiation, *etc*. This process can be divided into two categories: heat-reduced yellowing and light-reduced yellowing. The use of mechanical pulps in a number of applications is restricted due to their rapid loss of brightness upon exposure to light. Similarly, deinked pulps, which contain mechanical pulps, exhibit the same problem. Lignin is known to be responsible for yellowing. Over the last several decades, the color reversion of mechanical pulps has been studied by many researchers (Kimura *et al.* 1994; Leary 1994; Müller *et al.* 2003; Zou 2004; Pandey 2005; Rosu *et al.* 2010), but the underlying mechanism of photo yellowing has yet to be discovered. Moreover, the studies on the yellowing of deinked pulp, especially on the comparison of Mg(OH)$_2$-based with NaOH-based hydrogen peroxide bleached pulp, were much fewer (Savoye *et al.* 2011). Leduc *et al.* (2010) determined the surface lignin concentration of softwood thermo-mechanical pulp (TMP), hardwood chemi-thermo-mechanical pulp (CTMP), and deinked pulp using the x-ray photoelectron spectroscopy (XPS) technique and found that the $P_{Mg(OH)2}$-bleached deinked pulp (DIP) had lower surface lignin concentration than that of the P_{NaOH}-bleached DIP. Therefore, they inferred that the former was not as easy as the latter to become yellow. However, this assumption has not been confirmed by other researchers yet.

In this investigation, UV irradiation was used to accelerate color reversion of the unbleached, P_{NaOH}-bleached, and $P_{Mg(OH)2}$-bleached deinked pulp. The brightness loss, post color (PC) number, and CIELAB parameters (L^*, a^*, b^*, and ΔE^*) were measured to explore the yellowing properties of these pulps and paper. The changes in chemical structure of the surfaces of these pulps were also studied by attenuated total reflection Fourier transform infrared (ATR-FTIR) spectroscopy.

EXPERIMENTAL

Materials

The deinked pulp was obtained from the Guangzhou Paper Mill located in Guangzhou, Guangdong Province, China. The brightness value of the unbleached DIP was 47.2% ISO. Other chemicals used in bleaching and analyses were of analytical grade and supplied by the Guangzhou Chemical Reagent Factory, Guangzhou, China. Magnesium hydroxide powder was used in this experiment, containing $Mg(OH)_2$ 98%, Fe 31.5 ppm, Mn 1.7 ppm, and Cu less than 1 ppm; the median size (D50) was 10.62 μm and the size distribution (D10 through D90) was from 5.60 μm to 24.59 μm. Chemical dosing of the additive was calculated on an oven dry (o.d.) pulp mass basis.

Methods

Hydrogen peroxide bleaching

Hydrogen peroxide bleaching experiments were conducted in heat-sealed plastic bags. Before the bleaching liquor was added, the pulp slurry was pre-heated to 80 ºC. To make the bleaching liquor for peroxide bleaching, the chemicals were mixed in a beaker in the following order: distilled water, sodium silicate, ethylene diamine tetraacetic acid (EDTA), and NaOH or $Mg(OH)_2$. Then hydrogen peroxide was added directly to the pulp after other chemicals were added and mixed thoroughly. The plastic bag was then sealed and fully immersed in a pre-heated thermostatically controlled water bath for the desired retention time. The pulp was mixed every 15 min for the duration of the experiment. The experimental conditions for hydrogen peroxide bleaching were as follows: an H_2O_2 dose of 3.00%, 0.50% EDTA, 2.00% Na_2SiO_3, a temperature of 80 ºC, reaction time 120 min, and 10% pulp consistency; for P_{NaOH} bleaching the NaOH dose was 2.00% and for $P_{Mg(OH)2}$ bleaching the $Mg(OH)_2$ dose was 1.45% (Yun and He 2013). After the required retention time, the pulp slurry was washed thoroughly with distilled water for further analysis.

Formation of handsheets

Pulps were transformed to handsheets following the TAPPI T205 sp-02 standard (2002) in a sheet former (RK3AKWT, Frank-PTI, Laakirchen, Austria). Then these handsheets were put into standard conditioning and testing atmospheres, according to the TAPPI T402 sp-03 standard (2003), for further analysis.

UV light-induced yellowing measurement

The UV light-induced yellowing experiments were conducted in a photo-aging accelerator. The wave length was set to 340 nm, at 50 ºC, 50% relative humidity, 0.85 W/m^2 incident light intensity, and the size of each paper sample was 110 mm × 80 mm.

The paper samples were removed after the desired time, and they were stored in darkness for 30 min for further optical analysis.

Optical analysis

The brightness was measured according to the TAPPI T452 om-02 standard (2002) using a brightness apparatus (Elrepho 070, L&W, Kista, Sweden). The brightness loss was calculated according to Eq. 1 (Fang *et al.* 2000),

$$\text{Brightness Loss} = (\text{Brightness}_{\text{before photo aging}} - \text{Brightness}_{\text{after photo aging}}) \times 100 \qquad (1)$$

PC number analysis

The post color number was often used to evaluate the yellowing degree of paper: the higher the PC number, the higher the degree of yellowing. The PC number was calculated with Eqs. 2 and 3 (Fang *et al.* 2000; McGarry *et al.* 2002),

$$\text{PC Number} = [(k / s)_{\text{before photo aging}} - (k / s)_{\text{after photo aging}}] \times 100 \qquad (2)$$

$$K / s = (1 - R_\infty)^2 / 2R_\infty \qquad (3)$$

where k is the light absorption coefficient, s is the light scattering coefficient, and R_∞ is the ISO-brightness (%) under R457.

Chromatic aberration analysis

The photo yellowing was measured by the CIE $L^*a^*b^*$ method. The lightness (L^*), redness (a^*), and yellowness (b^*) values were recorded. The overall chromatic change (ΔE^*) was calculated by Eq. 4 (Müller *et al.* 2003; Pandey 2005),

$$\Delta E^* = [(L^*_2 - L^*_1)^2 + (a^*_2 - a^*_1)^2 + (b^*_2 - b^*_1)^2]^{1/2} \qquad (4)$$

where $_1$ denotes the values before UV light exposure, $_2$ denotes the values after UV light exposure, L^* represents the gray value that varies between 0 (black) and 100 (white), positive values of (a^*_2 - a^*_1) describe a red shift, negative values of (a^*_2 - a^*_1) describe a green shift, positive values of (b^*_2 - b^*_1) describe a yellow shift, and negative values of (b^*_2 - b^*_1) describe a blue shift.

ATR-FTIR analysis

The infrared spectra of the handsheets were recorded on a FT-IR spectrophotometer (Vector 33, Bruker, Billerica, MA, USA). The spectra were measured in ATR mode at 4 cm^{-1} resolution and wavenumber scale 4000 cm^{-1} to 400 cm^{-1}.

RESULTS AND DISCUSSION

Evaluation Indicators of Photo Yellowing of Deinked Pulps

The photo yellowing of deinked pulps was evaluated by brightness loss, PC number, and overall chromatic change (ΔE^*). According to the CIELAB system, the positive and negative values of b^* represent yellow and blue, respectively. The greater the positive values of b^*, the more yellow the samples. To understand the relationships between brightness loss, PC number, Δ*E**, and photo yellowing of deinked pulps, the relationships between brightness loss, PC number, Δ*E**, and *b** of the unbleached, P_{NaOH}-, and $P_{Mg(OH)2}$-bleached pulps were studied, and the curving fits are shown in Figs. 1 through 3. The results showed that the brightness loss, PC number, and Δ*E** had a linear relationship with *b**, as the correlation coefficient *r* was larger than 0.99. Thus, the brightness loss, PC number, and Δ*E** could be used to evaluate the photo yellowing degree of the unbleached, the P_{NaOH}-bleached, and the $P_{Mg(OH)2}$-bleached DIP.

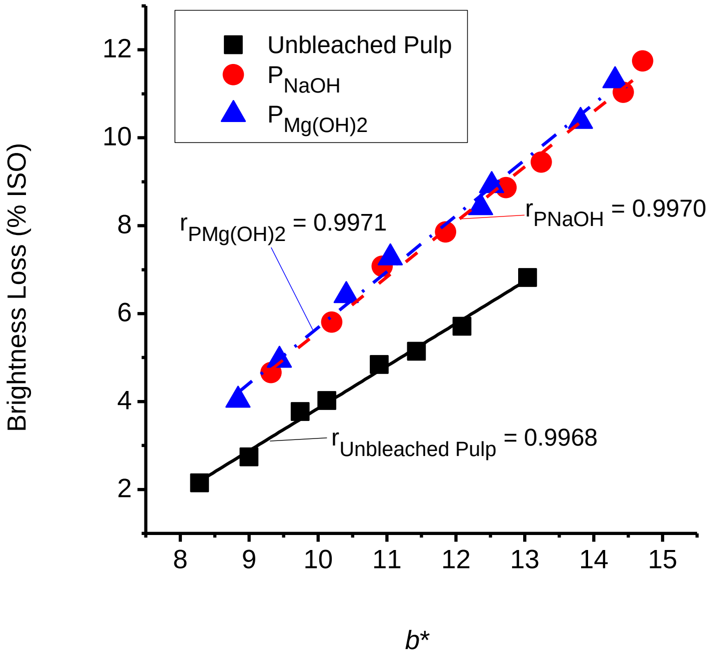

Fig. 1. Curving fit of brightness loss *vs.* *b**

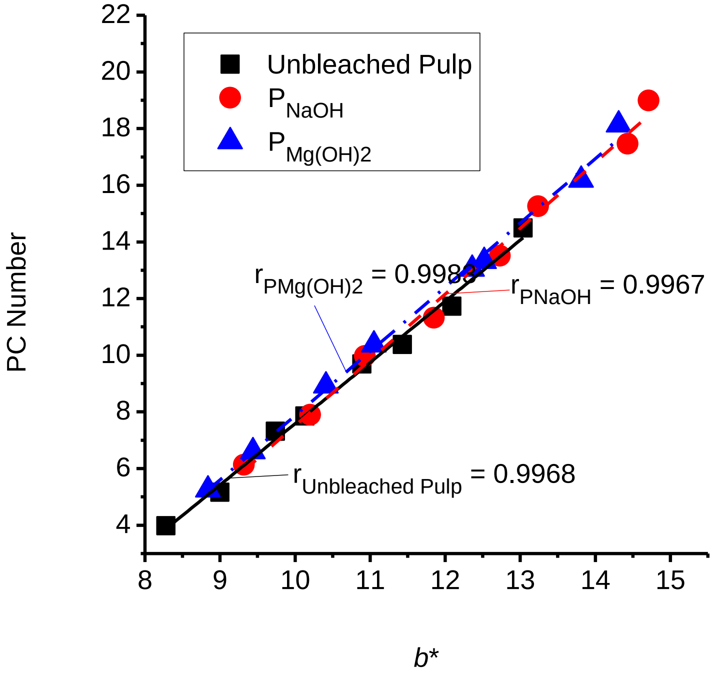

Fig. 2. Curving fit of PC number *vs.* *b**

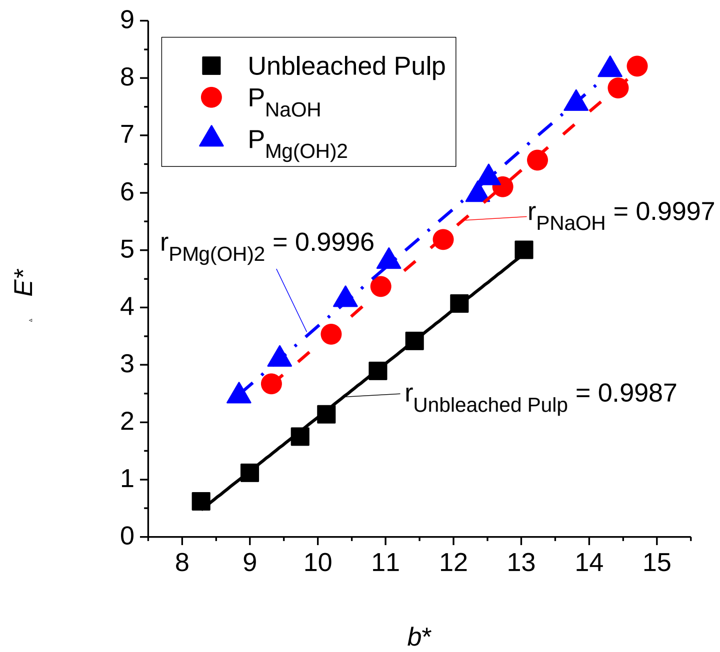

Fig. 3. Curving fit of Δ*E** *vs. b**

Effect of Irradiation Time on Brightness Loss, PC number, and Chromatic Aberration

In this investigation, with the extension of irradiation time, the changes of brightness loss, PC number, and Δ*E** were studied. The set irradiation times were as follows: 0 min, 5 min, 10 min, 15 min, 20 min, 25 min, 30 min, 60 min, 90 min, 120 min, 180 min, 240 min, 300 min, and 360 min. The changes in brightness loss, PC number, and Δ*E** of the unbleached, P_{NaOH}- bleached, and $P_{Mg(OH)2}$-bleached handsheets with increased irradiation time are shown in Figs. 4 through 6.

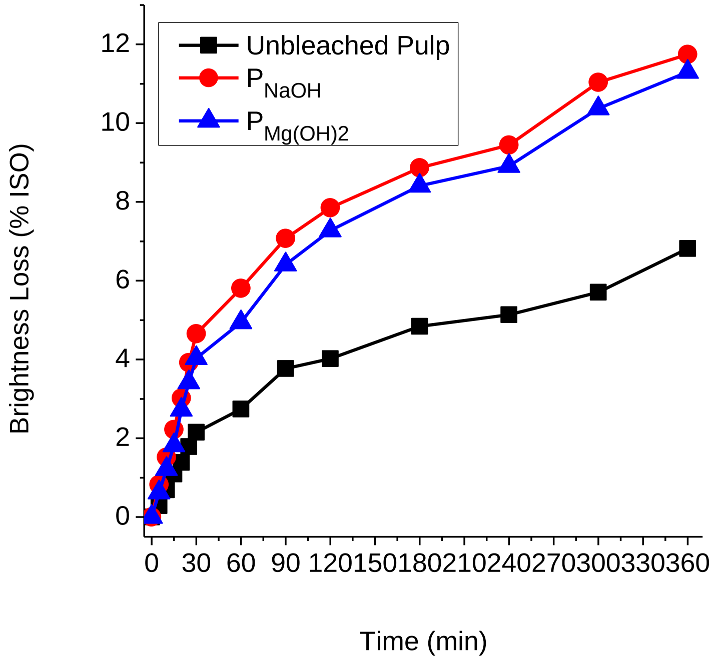

Fig. 4. Pulp brightness loss *vs.* time

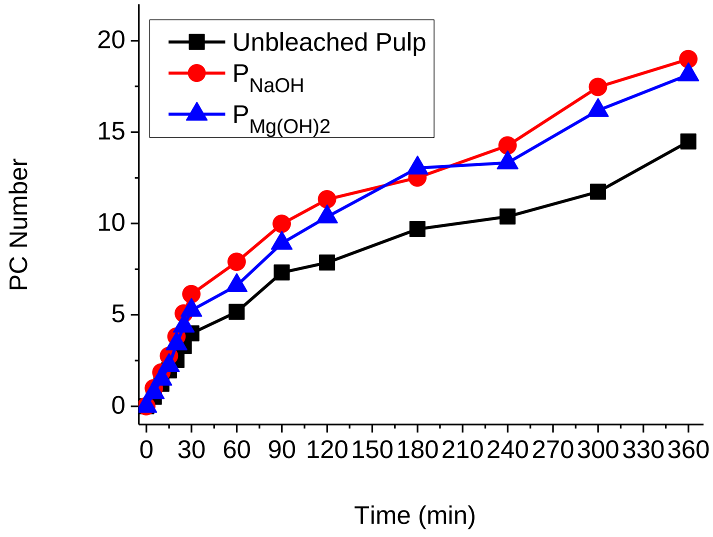

Fig. 5. PC number *vs.* time

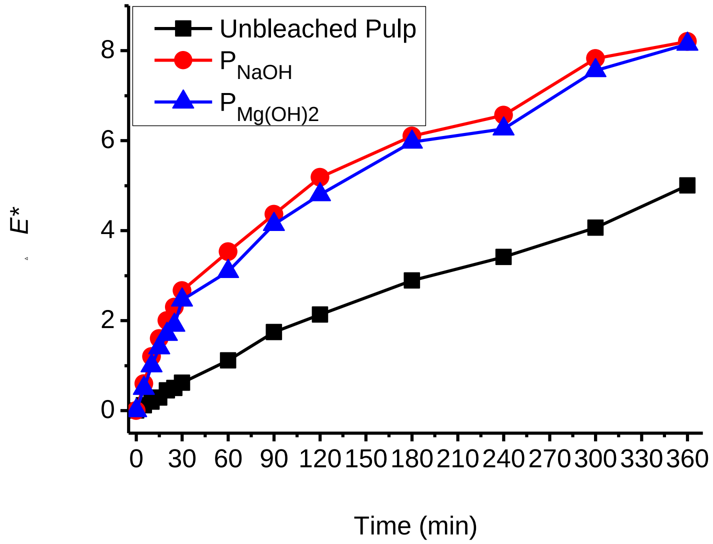

Fig. 6. ΔE^* *vs.* time

As shown, with the increase in irradiation time, the brightness loss, PC number, and ΔE^* also increased. When the irradiation time was 30 min, the brightness loss of the unbleached, P_{NaOH}-bleached, and $P_{Mg(OH)2}$-bleached pulps was 2.2% ISO, 4.6% ISO, and 4.0% ISO, respectively. During the following 150 min the brightness loss was almost the same as that of the first 30 min, and during the last 180 min the brightness loss was less than that of the first 30 min. This was the same for the PC number and ΔE^*. When the irradiation time was 30 min, the PC number and ΔE^* of the unbleached, P_{NaOH}-bleached, and $P_{Mg(OH)2}$-bleached pulps were 4.0, 6.1, 5.2, and 0.6, 2.7, 2.4, respectively. Moreover, the changes in the first 30 min of the PC number and ΔE^* were the greatest, which indicated that it was an accelerated yellowing stage. With increased irradiation time, the changes of brightness loss, PC number, and ΔE^* slowed down, which indicated that it was a slow yellowing stage. When the irradiation time was 360 min, the brightness loss, PC number, and ΔE^* of the unbleached, P_{NaOH}-bleached, and $P_{Mg(OH)2}$-bleached pulps were 6.8% ISO, 11.7% ISO, 11.3% ISO, 14.5, 19.0, 18.1, and 5.0, 8.2, 8.1, respectively. These data also indicated that the brightness stability of $P_{Mg(OH)2}$-bleached pulp was better than that of P_{NaOH}-bleached pulp.

ATR-FTIR Analysis

Spectra analysis before and after bleaching

The IR spectra of the unbleached pulp are shown in Fig. 7, and the assignment of bands in the ATR-FTIR spectra is listed in Table 1 (Kimura *et al.* 1994; Kimura *et al.* 1995; Pandey 1999; Tshabalala *et al.* 2005; Rosu *et al.* 2010).

Table 1. Assignment of Bands in ATR-FTIR Spectra of Pulp

Wavenumber (cm^{-1})	Band Assignment
3348 to 3331	O-H stretching vibration
2928 to 2924	C-H stretching vibration in methyl and methylene groups
2897 to 2872	C-H stretching vibration in methyl and methylene groups
1734 to 1725	C=O stretching vibration (non-conjugated)
1645 to 1655	Conjugated C=O stretching vibration
1640 to 1641	Water H-O-H bending vibration, conjugated carbonyl group
1605 to 1600	Aromatic ring stretching vibration
1510 to 1505	Aromatic ring stretching vibration
1430 to 1425	C-H deformation vibration (asymmetrical)
1372 to 1366	C-H deformation vibration (symmetrical)
1337 to 1336	C-H deformation vibration
1316 to 1315	Carbohydrates O-H bending vibration, CH_2 vibrations
1280 to 1276	C-O of guaiacyl unit in lignin
1242 to 1238	Stretching vibration of C-O-C in lignin structure
1160 to 1157	Carbohydrates C-O-C asymmetrical stretching vibration
1030 to 1028	Carbohydrates C-O symmetrical stretching vibration
901 to 896	C1–H deformation vibration, glucose ring stretching

As shown in Fig. 7, a key aspect of the IR spectra of a piece of paper was a strong hydrogen bonded O-H stretching band in 3700 cm^{-1} to 3000 cm^{-1} and a prominent C-H stretching band in 3000 cm^{-1} to 2800 cm^{-1}; and there are many sharp and discrete bands in the fingerprint region in 1750 cm^{-1} to 1000 cm^{-1}. These bands were mainly from the presence of lignin and carbohydrates (cellulose and hemicellulose) (Pandey 1999).

Figure 8 shows the changes of the IR spectra of unbleached pulp after it was bleached using NaOH-based and $Mg(OH)_2$-based hydrogen peroxide. After the deinked pulp was bleached, the intensity of the band at 1733 cm^{-1} assigned to non-conjugated C=O stretching decreased; the intensity of the band at 1655 cm^{-1} assigned to conjugated C=O stretching also decreased and was shifted down to 1641 cm^{-1}; both of which indicated that peroxide bleaching could deconstruct both non-conjugated and conjugated carbonyl groups. At the same time, the band at 1238 cm^{-1} assigned to the C-O-C in lignin structure vanished after bleaching, which indicated that peroxide bleaching could break the ether bond in the lignin structure.

As shown in Fig. 8, the shape of the IR spectra of the P_{NaOH}- and $P_{Mg(OH)2}$-bleached pulps was similar. However, according to the intensity of the bands related to lignin (1600 cm^{-1}, 1505 cm^{-1}), the $P_{Mg(OH)2}$-bleached pulp was lower than P_{NaOH}-bleached pulp, which indicated that the surface lignin concentration of $P_{Mg(OH)2}$-bleached pulp was lower than that of P_{NaOH}-bleached pulp. The intensity of the bands related to carbohydrates, located at 1372 cm^{-1}, 1316 cm^{-1}, and 1029 cm^{-1} in $P_{Mg(OH)2}$-bleached pulp, was higher than that of P_{NaOH}-bleached pulp, which indicated that the degradation of carbohydrates caused by $P_{Mg(OH)2}$ bleaching was less than that caused by P_{NaOH} bleaching.

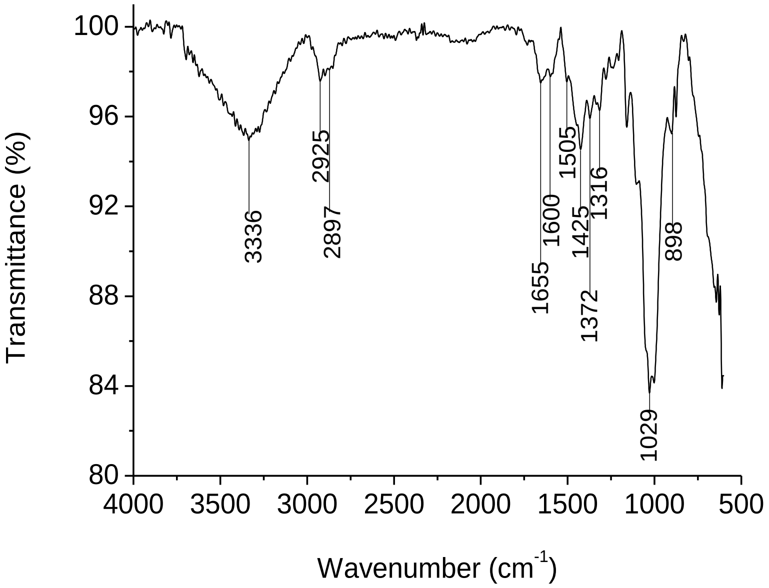

Fig. 7. IR spectra of unbleached pulp

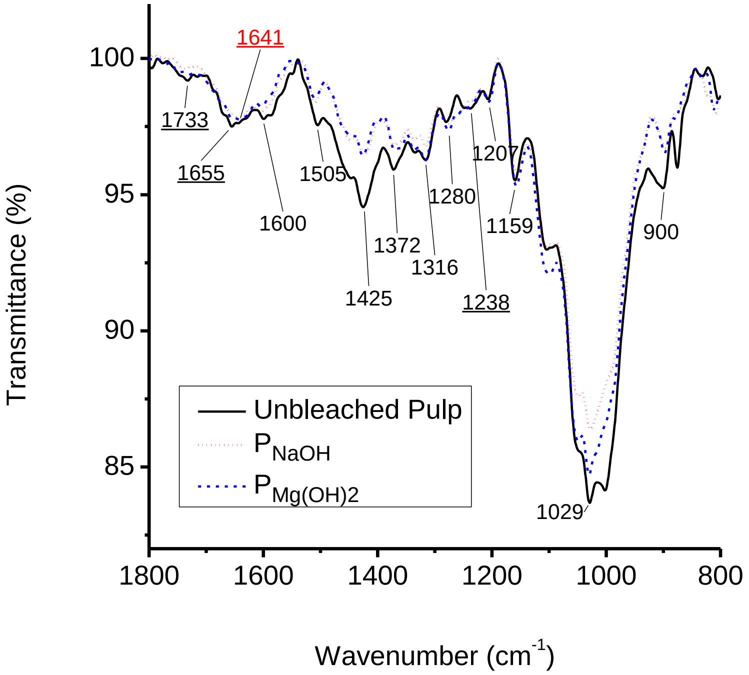

Fig. 8. IR spectra of the three pulps before irradiation

Spectra analysis before and after irradiation

The IR spectra of the unbleached, P_{NaOH}-bleached and $P_{Mg(OH)2}$-bleached pulps after UV irradiation for 360 min are shown in Fig. 9. Some researchers (Leary 1980; Lebo *et al.* 1990; Gellerstedt and Zhang 1992; Argyropoulos *et al.* 1995; Agarwal 1998) believed that the color changes were mostly caused by the conjugated double bond, carbonyl groups, and quinones, while other researchers believed it was *p*-quinones but not α-carbonyl or aromatic conjugated ethylenic bonds that caused photo yellowing of pulps (Agarwal and McSweeny 1997; Agarwal 1998). Thus, to reveal the photo yellowing mechanism of the deinked pulp, the existence and changes of functional groups in lignin before and after UV irradiation were investigated in this study.

The previous studies showed that the bands at 1600 cm^{-1} and 1505 cm^{-1} assigned to the aromatic ring stretching vibration were caused by lignin (Owen and Thomas 1989); the band at 1729 cm^{-1} was caused by a carbonyl stretching vibration (Hergert 1971; Owen and Thomas 1989); and the band at 1674 cm^{-1} was assigned to *p*-quinones (Agarwal 1998). As for the bands at 1600 cm^{-1} and 1505 cm^{-1}, the latter could better reflect the changes of lignin (Marton and Sparks 1967). The IR spectra of the unbleached, P_{NaOH}-bleached, and $P_{Mg(OH)2}$-bleached pulps before and after 360 min of UV irradiation are shown in Figs. 8 and 9.

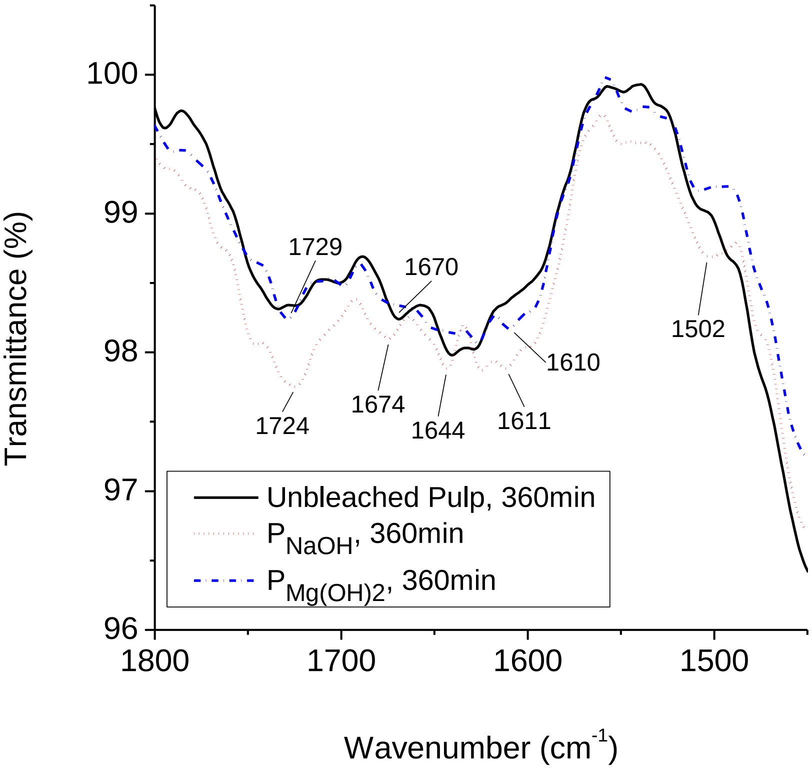

Fig. 9. IR spectra of three pulps after UV irradiation for 360 min

After 360 min of UV irradiation, the intensity of these pulps in the IR spectra changed drastically. The band near 1505 cm^{-1} decreased quickly and even disappeared, which indicated that the aromatic rings in the lignin were degraded; and this observation has been verified by other researchers (Kimura *et al.* 1992, 1995). The intensity of the band at 1655 cm^{-1} in the unbleached pulp and at 1641 cm^{-1} in the bleached pulps that were assigned to the conjugated carbonyl stretching vibration decreased. Meanwhile the intensity of the band around 1729 cm^{-1} assigned to non-conjugated carbonyl stretching increased, and a new band around 1674 cm^{-1} assigned to *p*-quinones appeared, which indicated that the lignin or hemicelluloses on the pulp surface was photooxidized, and the new bands had a relationship with photo-yellowing. This finding was in accordance with Agarwal and McSweeny's research (1997), as they discovered that the IR spectra of photo-yellowed TMP had new bands at 1727 cm^{-1} and 1673 cm^{-1}. The scheme of the formation of the band at 1727 cm^{-1} is as follows: upon UV light exposure, the hydroquinone in the pulp is converted first to a semi-quinone radical, then to a quinone. The quinone absorbs UV light to produce a triplet quinone or a di-radical one. In the presence of hydrogen provided by alcohol from carbohydrates or lignin, the semi-quinone radical and hydroxyl radicals are produced; and the hydroxyl radical reacts with a quinone to generate a keto group. The formation of the latter functionality can account for the IR band at 1727 cm^{-1} in the photo-yellowed pulps (Agarwal 1998).

As shown in Fig. 9, the IR spectra of P_{NaOH}- and $P_{Mg(OH)2}$-bleached pulps after UV irradiation for 360 min were compared. The authors found that the intensity of the band at 1727 cm^{-1} and 1673 cm^{-1} of P_{NaOH}-bleached pulp was stronger than that of $P_{Mg(OH)2}$-bleached pulp, which indicated that the non-conjugated carbonyl and *p*-quinone on P_{NaOH}-bleached pulp surface was more than that on $P_{Mg(OH)2}$-bleached pulp's surface. As during peroxide bleaching, lignin reacted with hydrogen peroxide and HOO^- was produced, and the hydroquinone and quinone were formed due to HOO^-. Less HOO^- was produced in $P_{Mg(OH)2}$ bleaching than that in P_{NaOH} bleaching (Yun and He 2013). Therefore, $P_{Mg(OH)2}$ bleaching produced less hydroquinone and quinone than that of P_{NaOH} bleaching (Gierer *et al.* 1994). Furthermore, $P_{Mg(OH)2}$ bleaching produced less oxygen and hydroxyl radicals than that of P_{NaOH} bleaching, which meant that less new quinones had been produced (Agnemo and Gellerstedt 1979). Before UV irradiation, the band at 1673 cm^{-1} assigned to *p*-quinone was too weak to be detected (Agarwal 1998). After 360 min irradiation, compared with P_{NaOH}-bleached pulp, less *p*-quinone was produced in the $P_{Mg(OH)2}$-bleached pulp due to lower amounts of hydroquinone and *p*-quinone. This was in accordance with the result that the PC number of $P_{Mg(OH)2}$-bleached pulp was less than that of P_{NaOH}-bleached pulp.

CONCLUSIONS

1. The brightness loss, PC number, and overall chromatic change had a linear relationship with *b**, and could be used to evaluate the photo yellowing degree of the unbleached and bleached DIP.
2. With the increase in UV irradiation time, the brightness loss, PC number, and overall chromatic change at first increased quickly, and then slowed down. After UV irradiation for 360 min, the brightness loss of P_{NaOH}- and $P_{Mg(OH)2}$-bleached pulps was 11.7% ISO and 11.3% ISO; the PC number was 19.0 and 18.1; and the overall chromatic change was 8.2 and 8.1, respectively.
3. After UV irradiation for 360 min, the band intensity at 1729 cm^{-1} increased distinctively, and a new band at 1674 cm^{-1} appeared. This indicated that *p*-quinone groups were produced during the irradiation process, thus resulting in paper yellowing. The band intensity at 1674 cm^{-1} of $P_{Mg(OH)2}$-bleached pulp was lower than that of P_{NaOH}-bleached pulp, which indicated that the brightness stability of $P_{Mg(OH)2}$ pulp was better than that of P_{NaOH} pulp.

ACKNOWLEDGMENTS

This work was supported by the Research Award Fund for Outstanding Young Teachers in Higher Education Institutions of the Guangdong Province (YQ2014179) and State Key Laboratory of Pulp and Paper Engineering (201739).

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Article submitted: February 7, 2017; Peer review completed: April 15, 2017; Revised version received: April 21, 2017; Accepted: May 27, 2017; Published: June 6, 2017.
DOI: 10.15376/biores.12.3.5236-5248