INFLUENCES OF INTEGRATED TEMPO-MEDIATED OXIDATION AND RECYCLING ON THE PROPERTIES OF TMP FIBERS

Pu Ma,[a] Huamin Zhai,[a,*] Kweinam Law,[b,*] and Claude Daneault [b]

In order to improve the properties of thermomechanical pulp (TMP), the influences of the TEMPO (2,2,6,6-tetramethylpiperidyl-1-oxyl radical)-mediated oxidation on recycled TMP properties were investigated, and the impacts of recycling process on TEMPO-mediated oxidized TMP properties were studied as well. The results showed that TEMPO-mediated oxidation is an effective way to enhance the recycled TMP inter-fiber bonding dependent properties due to the introduction of carboxylic acid groups onto pulp, while the oxidation had some negative impacts on the tear index, zero span tensile index, and brightness. The oxidation-recycling (O-R) process had remarkable adverse impacts on TMP compared with the recycling-oxidation (R-O) process. The tensile, burst, tear strengths, as well as the zero-span tensile strength dropped sharply when oxidized TMP was recycled, and the physical strength properties decreased with the increasing recycling times. The opacity was improved after the O-R treatment, although the O-R treatment had an adverse impact on the pulp brightness.

Keyword: Thermomechanical pulp; Recycling; TEMPO-mediated oxidation; Carboxylic acid group

*Contact information: a: Jiangsu Provincial Key Lab of Pulp and Paper Science and Technology, Nanjing Forestry University, Nanjing 210037, China; b: Centre intégré en Pâtes et Papiers, Université du Québec à Trois-Rivières, G9A 5H7, Canada; * Corresponding author: hzhai@njfu.edu.cn; kwei-nam law@uqtr.ca*

INTRODUCTION

Recycled fibers have been used more and more in recent years due to their low price and the shortage of fiber raw materials. High-yield pulp, due to its high bulk and high light scattering coefficient, is widely used in various kinds of paper and paperboard. Therefore, inevitably, these high yield pulps become reused as a component in recycled paper. However, the poor strength of high yield pulp has restricted its application (Gu 1999). It is very important to find a novel useful method to enhance the fiber strength performance of high-yield pulp. TEMPO (2,2,6,6-tetramethylpiperidyl-1-oxyl)-mediated oxidation, which was first used for polysaccharide oxidation in 1995 (De Nooy *et al*. 1995), is considered a very effective method for paper strength enhancement because of the introduction of the carboxyl and aldehyde groups on fiber surface (De Nooy *et al*. 1995; Saito and Isogai 2006). TEMPO-mediated oxidation had been used for kraft pulp enhancement by Dang *et al*. (2007), and for thermomechanical pulp (TMP) enhancement by Law and Mao (Law *et al*. 2007; Mao *et al*. 2008); it was also used for deinking pulp modification by Leroux *et al*. (2007). All the results indicated that TEMPO-mediated oxidation could improve the paper strength remarkably. TEMPO-mediated oxidation also has been used for recycled kraft pulp modification by Gomes *et al*. (2009). However, it is generally believed that the properties of high yield pulp are quite different from those of

kraft pulp due to its high lignin content, and their performances during recycling are quite dissimilar. For low-yield pulp, the recycling reduces inter-fiber bonding ability significantly and in turn reduces bonding-dependent strength properties (Wistara and Young 1999). For high-yield mechanical pulps, the trend is reversed (Howard and Bichard 1992). Until now, the influences of TEMPO-mediated oxidation on recycled TMP properties have remained unclear. In return, the effects of recycling on TEMPO-mediated oxidized TMP properties have not been reported yet. To better understand the potential of TEMPO-mediated oxidation, prior to and after recycling, on improvement of physical strength and optical properties of TMP, the pulp was examined using two different approaches, namely TEMPO-mediated oxidation-recycling (O-R) and recycling-TEMPO-mediated oxidation (R-O), and their influences on paper quality were investigated in this study.

EXPERIMENTAL

Materials

Unbleached eastern Canadian spruce and balsam fir (70:30) TMP was sampled after a twin-wire press at the Kruger paper mill in Trois-Rivières, Québec, Canada. The pulp consistency was 32 to 34%, and the pulp freeness was 110 mL.

Recycling-Oxidation (R-O) Procedure

The recycling of never-dried TMP was carried out on a laboratory scale, where the fibrous suspension of 0.2% consistency was prepared, and 90 g/cm^2 papersheets were made by the dynamic sheet former (DSF). The wet sheets were pressed mechanically with a roll pressing machine; the pressure was increased to 6 bars (600 kN/m). Then the pressed papersheets were dried on a cylindrical drier for 4 minutes at 105 ^{o}C. However, differently with the traditional recycling treatments, the pre-dried sheets were dried again in an oven at 105 ^{o}C for 1 hour to accelerate paper aging, simulating extended recycling times. As such, one cycle in this study stands for 4 minutes on the drying cylinder and 1 h of oven-aging. Then the dried sheets were stored at 23 ^{o}C and 50% RH for 12 hours. The dried paper was torn into small pieces and soaked in deionized water for at least 24 hours before being disintegrated. After re-slushing, one part of the first recycled pulp was ready for analysis, and other parts of the pulp were prepared for further recycling according the procedure mentioned above. We obtained the 1st, 2nd, and 3rd recycled TMP one by one, and then the recycled TMP fibers were TEMPO-mediated oxidized according to the procedure described by Law *et al*. (2008). In detail, the experiment was conducted in a 2-litre glass reactor at 21 ^{o}C. The TMP fibers of 30 g (o.d.) were first diluted to 1.5% consistency with deionized water, to which TEMPO (0.17% o.d fiber), sodium bromide (3 mmol/g o.d fiber), and sodium hypochlorite (3 mmol/g o.d fiber) were then added gradually. The pH of the system was maintained at 10.5 using NaOH or HCl solution, depending on the situation. The pulp suspension was continuously agitated by means of an electric stirrer. At the end of the predetermined reaction time (50 min), the fibers were drained, washed, and filtered, and then the pH of the fiber suspension was adjusted to 5.0. The pulp was again drained, washed, and filtered for at least four times to ensure the

removal of the residual chemicals and metallic ions prior to the measurement of carboxylic acid content. Finally we obtained the 1st recycled-oxidized, 2nd recycled-oxidized, and 3rd recycled-oxidized TMPs for handsheet making.

Oxidation-Recycling (O-R) Procedure

The oxidation-recycling treatment is the reverse process of the recycling-oxidation procedure described above. The never-dried TMP was first oxidized (TEMPO-mediated) with the same oxidation conditions described above. Then the recycling of the oxidized TMP was carried out according to the procedure described above as well. Finally, we obtained oxidized-1_{st} recycled, oxidized-2_{nd} recycled and oxidized-3_{rd} recycled TMPs for making handseets.

Pulp and Paper Testing

The carboxylic acid content of each of the pulps was determined by the conductometric titration method described by Katz *et al*. (1994). A Metrohm (Brinkmann) titrator and a conductivity meter (Thermo Orion, model 150) were used to measure the carboxyl groups. Briefly, a 3 g pulp sample (o.d. basis) was first treated with 200 mL 0.1 M HCl for 45 min and then thoroughly washed and filtered four times with 200 mL deionized water. The washed specimen was treated with 0.1 M HCl for 45 min again and washed as described previously. The filtered sample was suspended in 450 mL 0.001 M NaCl for the titration process. The measurements of carboxyl content were repeated twice and processed by a computer.

The pulp freeness was determined according to TAPPI method T227 om-99. Standard handseets of 60 g/m^2 for physical and optical properties analysis were prepared by means of a British sheet-mould according to the PAPTAC Standard Testing Methods C.4 and C.5, respectively. The major physical properties were determined according to PAPTAC Standard Testing Methods D.4, D.6H, D.8, D.9, and D.27u. The optical properties were obtained following the TAPPI Standard T452 om-08.

RESULTS AND DISCUSSION

Carboxylic Acid Group Content

As previously described, the TMP was treated either by recycling, TEMPO-mediated oxidation, R-O, or O-R treatments. As shown in Fig. 1, the letter R stands for the TMP after recycling. The pulp without any treatment (R_0) is the original untreated TMP. R-O stands for the TMP that was first recycled and then TEMPO-mediated oxidized. O-R stands for the reverse of procedure R-O, in which TMP was first subjected to TEMPO-mediated oxidization, and then recycled. O-R_0 is the TEMPO-mediated oxidized pulp, but without recycling.

The influences of recycling and TEMPO-mediated oxidation on carboxylic acid content are shown in Fig. 1. The recycling process had little impact on TMP carboxylic acid content, relative to R_0. The carboxylic acid group content introduced to the surface of never-dried TMP fibers by TEMPO-mediated oxidation was 297.2 mmol/kg (O-R_0). The R-O and O-R treatments had different impacts on carboxylic acid group content.

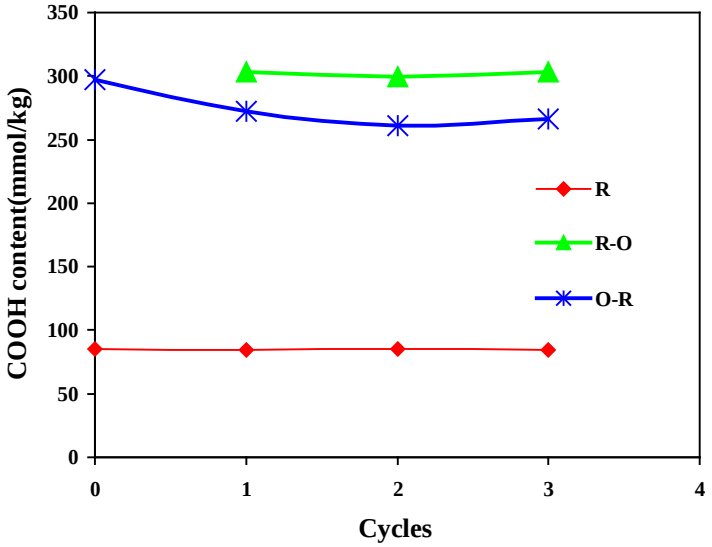

Fig. 1. Carboxylic acid content of R-O and O-R TMP

The carboxyl contents of R-O TMP samples were similar to that of O-R_0, and recycle times had no impact on pulp carboxyl content, which agrees with the findings of Gomes *et al.* (2009) for recycled kraft pulp oxidation. When TMP fibers were treated by the O-R process, they had lower carboxyl contents, 11.5% lower in contrast to that of R-O pulp. The reduction of carboxylic acid content of O-R pulp may be associated with the irreversible hornification on the fiber surface during recycling and aging process (Lee and Joo 2000).

Freeness

As shown in Fig. 2, the pulp freeness of recycled pulp was higher than that of untreated pulp (R_0, *ca*. 110 mL). Pulp freeness rose to *ca*. 300 mL after 3 recycles, which agrees with the trend found by Quan *et al*. (1996); the white water was not recirculated in the present study; thus some loss of fines may also have contributed to the observed gain of freeness during recycling. Recycling had similar effects on pulp freeness for both R-O and O-R TMP (Fig. 2). However, interestingly, we found that the freeness of R-O or O-R TMP was much lower than that of R, which should be attributed to the introduction of carboxylic acid groups by TEMPO-mediated oxidation, which also had been proved in our previous studies (Mao *et al*. 2008; Ma *et al*. 2010). On the other hand, the freeness of O-R was lower than R-O at any given cycle. The introduction of carboxylic acid groups on fiber surface first had adverse impacts on pulp freeness when these oxidized fibers were further reused. As we know, the oxidized fibers with acid groups become more brittle when dried at high temperature and moisture content (Caufield 1994), and they are more easily broken to small fragments in the following recycling process, resulting in the reduction of pulp freeness. In addition, the length-weighted fines percent of O-R_3 measured by FQA (Fiber quality analyzer), was 7.24%, compared with that of R_3-O, 6.84%, and this also indicates the impact of O-R and R-O treatments on pulp freeness.

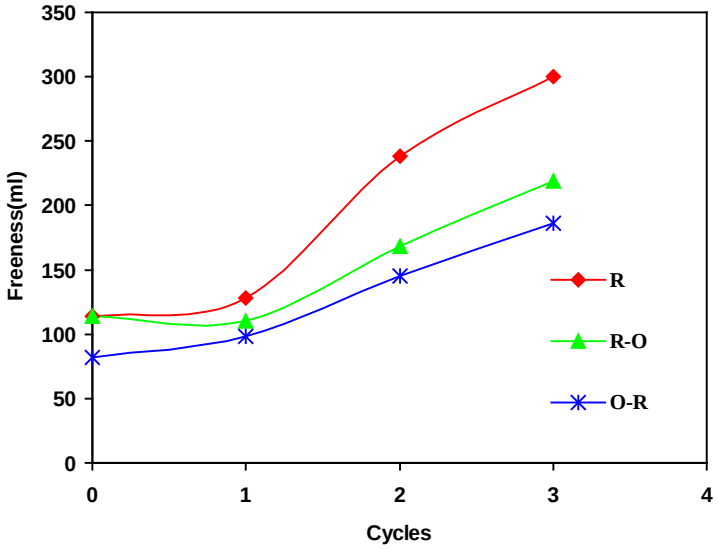

Fig. 2. Pulp freeness of R-O and O-R treatments

Strength Properties

Some recent studies (Leroux *et al*. 2006; Dang *et al*. 2007; Law *et al*. 2007; Leroux *et al*. 2007; Mao *et al*. 2008; Gomes *et al*. 2009) have revealed that carboxylic acid groups introduced on fiber surface by TEMPO-mediated oxidation can enhance the inter-fiber interaction by hydrogen bonding. The inter-fiber bonding-dependent factors, tensile and burst indexes, increased significantly after TEMPO-mediated oxidation for both kraft pulp (Saito and Isogai 2006) and TMP (Leroux *et al*. 2006; Law *et al*. 2007; Mao *et al*. 2008). The influences of TEMPO-mediated oxidation on tensile and burst indexes of recycled TMP presented in Figs. 3 and 4 reveal that TEMPO-mediated oxidation can be used for the enhancement of recycled TMP as well. The tensile index of the oxidized TMP (O-R_0) was 12.2% higher than that of R_0, and the tensile strength of R-O was higher than that of R at any given cycle (Fig. 3). R_3-O was 22.73% higher than R_3. Based on our previous studies (Ma *et al.* 2009, 2010), the carboxylic acid groups were associated with the NaClO charge, and we anticipated that we could obtain handseets with higher tensile strength in this study when a higher amount of NaClO was used for recycled TMP oxidation. TEMPO-mediated oxidation is an effective way to enhance the tensile strength of recycled TMP, even after many recycles. However, unfortunately, recycling had significant adverse impact on oxidized TMP. The tensile index of oxidized TMP was reduced by 50% after 1 cycle and 74% after 3 cycles as compared to R_0 (Fig. 3). It was much lower than that of R-O at any given cycle as well. The remarkable decline of paper tensile strength of O-R TMP might be attributed to the accelerated acid hydrolysis rate of cellulose when more acid groups were introduced (Hirosawa *et al*. 2001), resulting in the decline of fiber intrinsic strength. On the other hand, as remarked in a previous study, the factor which improved bonding between cellulosic fibers would unfortunately tend to reduce the capability to form strong inter-fiber bonds again (Pycraft *et al*. 1980), since the factors that contribute to strong bonding, such as coalescence of adjacent cellulosic surfaces, are also likely to contribute to hornification during recycling. The introduction of carboxylic acid groups by TEMPO-mediated oxidation promoted

hornification during recycling (Lindstrom and Carlsson 1982; Fernandes Diniz *et al*. 2004), resulting in the significant drop of tensile strength of O-RTMP in this study. It should be pointed that the influences of recycling on TMP tensile (Fig. 3) and burst strength (Fig. 4) were opposite to those of a previous study (Howard and Bichard 1992), which should be attributed to the severe recycling and aging conditions in this study.

Comparing Figs. 4 and 3, the TEMPO-mediated oxidation and recycling had similar impacts on burst index compared with tensile index. In contrast to R, burst strength of R-O was higher due to the introduction of carboxylic acid groups by TEMPO-mediated oxidation. But it decreased remarkably when these oxidized fibers were reused, as indicated by the comparison between O-R_0 and O-R_1, O-R_2 and O-R_3 (Fig. 3). The burst index of the O-$R_{1,2,3}$ pulp was only 50% when compared with that of $R_{1,2,3}$-O.

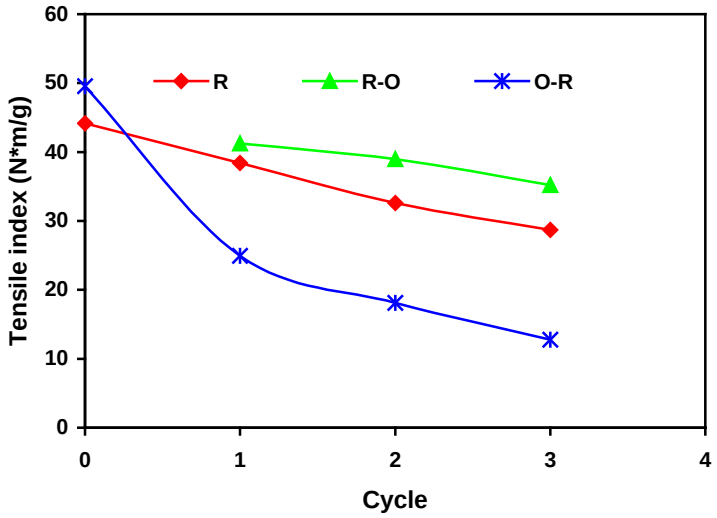

Fig. 3. Effects of R-O and O-R treatments on tensile index

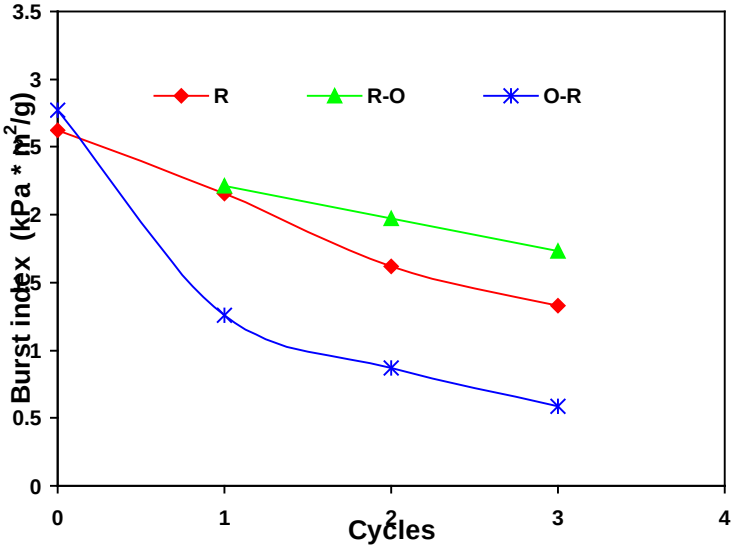

Fig. 4. Effects of R-O and O-R treatments on burst index

Our recent studies indicated that TEMPO-mediated oxidation had an adverse impact on TMP tear strength (Mao *et al*. 2008, 2010). Recycling also had a negative impact on tear index due to a weakened bonding potential of hornified fiber walls (Minor and Attalla 1992). As shown in Fig. 5, a significant reduction of tear index was observed when TMP was oxidized; the tear index of oxidized virgin TMP (O-R_0) was 19% lower than that of the untreated one (R_0). The recycling process had an adverse impact on TMP tear strength as well; the tear strength of R_3 was 14% lower than that of R_0. However, interestingly, R-O and O-R treatments had quite different impacts on pulp tear strength. The tear index of R-O TMP was quite similar regardless of the number of recycling cycles. But for O-R TMP, recycling treatment remarkably decreased the tear index; only 41% tear strength was retained when the oxidized TMP was recycled 3 times. It is known that fiber length and strength are particularly important for tearing resistance (Seth and Page 1988; Wangaard and Williams 1970). However, it was quite interesting that the O-R TMP with longer fiber length (*e.g*. 1.62 mm for O-R_3, in contrast to 1.58 mm for R_3-O), showed much lower tear strength than R-O TMP (Fig. 5). The significant decline of fiber intrinsic strength should be responsible for this phenomenon, because the energy of tear failure derives primary from the energy released when fibers fail rather than when fibers are pulled out or when bonds break (Page 1994). A 10% loss in fiber strength can lead to a 25 to 30% loss in tear strength (Page and MacLeod 1992).

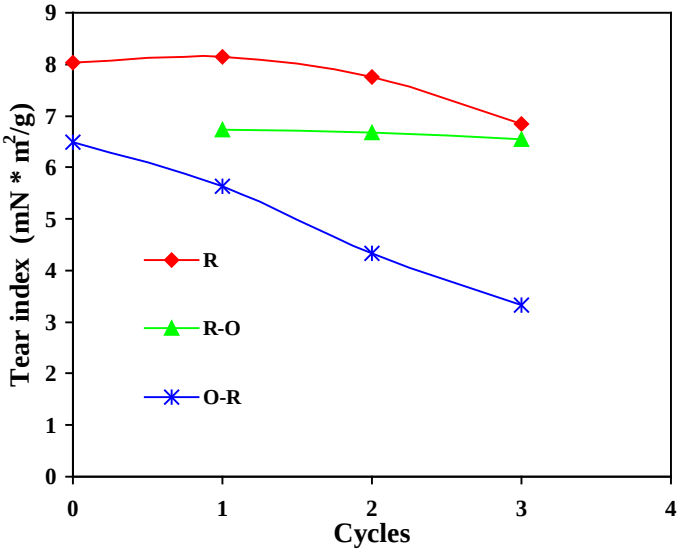

Fig. 5. Effects of R-O and O-R treatments on tear index

The degree of polymerization (DP) of TMP cellulose, which is related to the intrinsic fiber strength, was indirectly evaluated by measurement of the zero-span tensile strength; the viscosity test for DP was not used in the present study due to the high lignin content. The impact of recycling on the recycled TMP zero span strength is shown in Fig. 6. It is understood that cellulose chain scissions contribute to a loss of zero-span tensile strength that is often observed when fibers are recycled (Klungness *et al.* 1982). The TEMPO-mediated oxidation also had an adverse impact on the zero span tensile strength due to the side reactions during TEMPO-mediated oxidation (Ma *et al.* 2001), in particular the β-elimination of cellulose (De Nooy *et al*. 1996; Potthast *et al*. 2007),

which was also evident when R_0 was compared with $O\text{-}R_{0,}$ (Fig. 6). R-O and O-R treatments also showed different impacts on zero span tensile strength. Only a 5 to 10% reduction in zero span tensile index was observed when comparing zero span strength of R-O with that of R. However, as for O-R pulp, it was quite different with R-O pulp, the zero span tensile strength dropped sharply after recycling. The zero span tensile strength of $O\text{-}R_1$ was 15.8% lower than that of $O\text{-}R_0$. Hirosawa *et al*. (2001) showed that the hydrolysis rate of oxidized cellulose samples accelerated with increasing carboxyl content, decreasing the cellulose polymerization. This can explain the sharp drop of zero span tensile strength for O-R TMP.

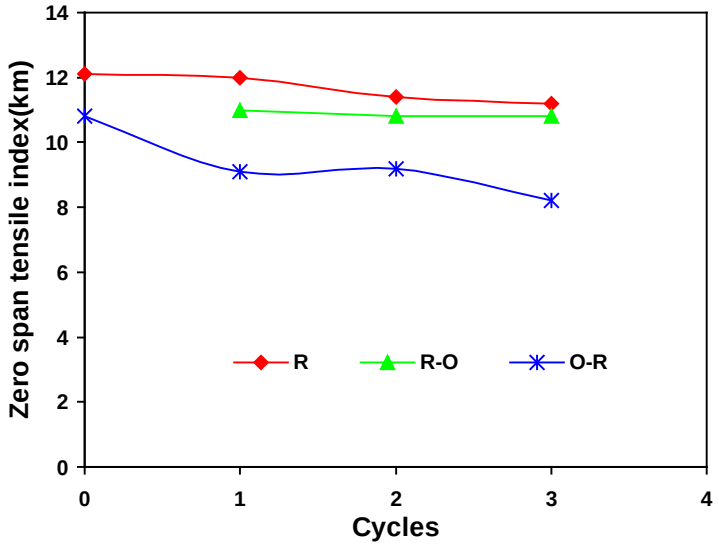

Fig. 6. Effects of R-O and O-R treatments on zero span tensile index

Optical Properties

Figure 7 shows the effects of R-O and O-R treatments on pulp brightness. The TMP brightness decreased slightly after TEMPO-mediated oxidation, as R_0 was compared with $O\text{-}R_0$, and the brightness of R was similar to that of R-O. The oxidation did not have remarkable negative impact on brightness of recycled TMP. However, the brightness of O-R was much lower than that of R-O at any given cycle. The brightness of $O\text{-}R_3$ was 31.3% ISO which was lower than that of R_3-O, 45.88% ISO.

Our previous study indicated that a portion of lignin was removed during TEMPO-mediated oxidation, which was dependent on the NaClO dosage (Ma *et al.* 2009). But the decline of lignin content did not improve brightness in this study due to the relatively low pH (10.5) environment; the dissolved lignin was easily condensed at this low pH, resulting in lower brightness for $O\text{-}R_0$ as compared to R_0. On the other hand, pulp yellowing is directly related to the oxidation, and production of carbonyl and carboxylic acid groups could lead to coloured fibers (Sjostrom and Eriksson 1968). Therefore, the brightness of TEMPO-mediated oxidized TMP in this study declined remarkably due to its severe yellowing at high temperature during recycling, as R-O was compared to O-R.

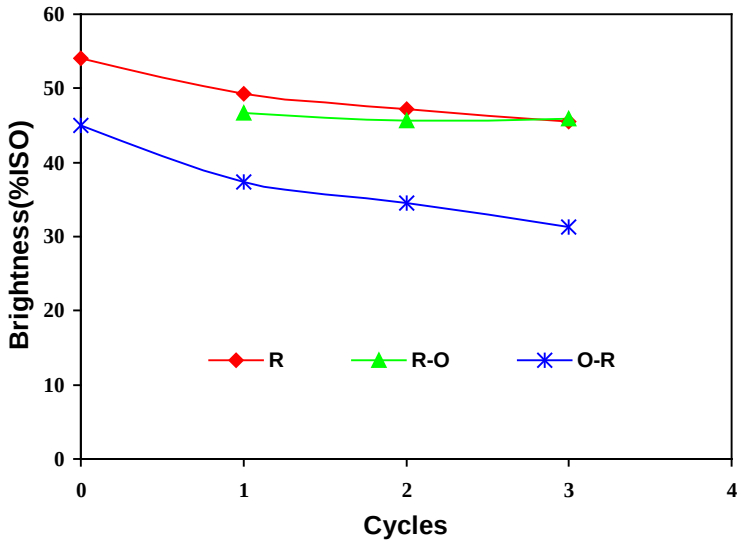

Fig. 7. Effects of R-O and O-R treatments on brightness

The opacity decreased slightly as the untreated TMP (R_0) was oxidized (R_0-O), as shown in Fig. 8. A similar trend was noted when the recycled TMP was TEMPO-mediated oxidized. These phenomena were also observed in our previous study (Ma *et al*. 2010), indicating the negative impact of TEMPO-mediated oxidation on paper opacity. A slight increase of paper density due to the introduction of carboxyl acid groups, *e.g*. 0.324 g/cm^3 for R_3 *vs*. 0.333 g/cm^3 for R_3-O, was accountable for the drop in opacity. On the other hand, recycling positively increased the opacity of both untreated (R_0 and $R_{1,2,3}$) and oxidized TMP (O-R_0 and O-$R_{1,2,3}$). The fiber hornification during recycling, resulting in the drop of inter-fiber bonding potential and paper density, should be responsible for this phenomenon.

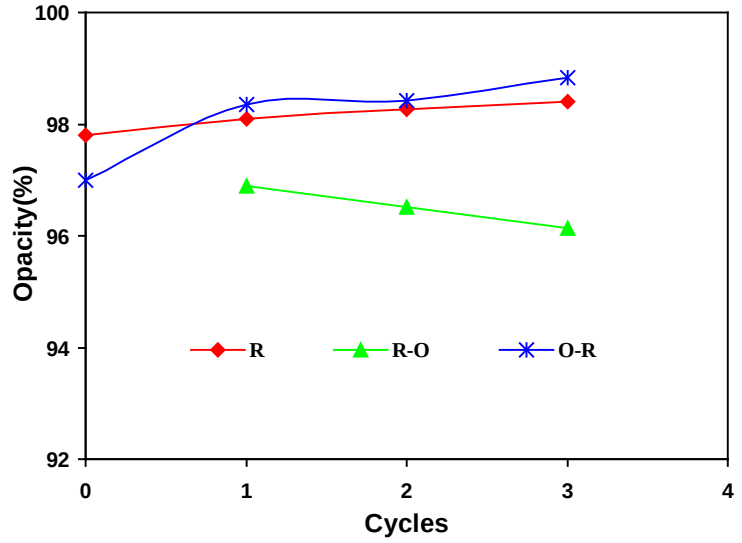

Fig. 8. Effects of R-O and O-R treatments on opacity

CONCLUSIONS

1. TEMPO-mediated oxidation has similar impact on both recycled TMP and the initial TMP, and it can be used for the enhancement of recycled TMP. High contents of carboxylic acid groups can be introduced onto the recycled fiber surface, improving the tensile and burst strengths.
2. TEMPO-mediated oxidation has adverse impacts on pulp freeness, tear, zero span tensile indexes, brightness, and the opacity of the recycled paper.
3. Oxidation-recycling treatments have different impacts on TMP properties as compared to the recycling-oxidation treatments. The R-O treatment significantly improves the tensile and burst strengths, while the O-R treatment decreases all the properties except for opacity. The adverse impacts on paper strength properties increase with increasing recycle times.
4. TEMPO-mediated oxidation of TMP pulp is not suitable for recycling, since most of the pulp properties decrease significantly when recycling is performed after the TEMPO-oxidation.

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