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Water retention and physical properties of recycled fibers treated with NaOH/urea aqueous solution

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Abstract Irreversible hydrogen bonding is the most widely recognized mechanism of fiber hornification, and NaOH/urea aqueous solution can break the inter- and intramolecular hydrogen bonds of cellulose. In order to investigate whether or not this solution system reverses the hornification of fibers that repeatedly underwent wetting and drying, we studied the degree of hornification of repaired fibers, which have been treated with NaOH/urea aqueous solution and regenerated in H_2SO_4/Na_2SO_4 aqueous solution, and we analyzed the physical properties of the resulting paper sheets. The water retention value (WRV) of the repaired fibers was equivalent to that of virgin pulp, and the hornification has been thoroughly reversed. The physical properties of the resulting paper sheets have improved, especially the tear and burst indexes, which have approximately doubled. Moreover, the dissolution/regeneration process slightly inhibited the degree of fiber hornification and increased the cycle number of the recycled fibers.

1. Introduction

Paper and paperboard remain indispensable materials in modern society, and 401 million tons of paper and paperboard were produced all over the world in 2013 [1]. Post-consumers' paper and paperboard are gathered as waste paper, which have become the main source of papermaking raw materials [2] and, thus, not only mitigate the problem of scarce wood resources but also reduce the issues that are associated with waste generation and final disposal. Unfortunately, using waste paper as recycled material for papermaking results in the loss of strength and the reduction of wet flexibility and bonding properties of the fibers, which are caused by hornification [3]. As a rough general guide, due to hornification, cellulose fibers become too short and weak to make paper sheets after 5–6 times being recycled [4, 5].

The term hornification was coined firstly by Jayme [6]. This phenomenon occurs when fibers repeatedly undergo wetting and drying, causing the loss of water retention in the pulp, which decreases the tensile strength of the resulting sheets. Multiple hypotheses [7], such as irreversible hydrogen bonding, formation of covalent bonds, fiber crystallinity, hemicellulose loss, surface inactivation, and cellulose chain cleavage, have been made to explain the exact causes and mechanisms involved in hornification, but these causes are still a matter of discussion [7]. The most widely recognized hypothesis is the formation of non-reversible, intra-fiber hydrogen bonds among the cellulose

microfibrils, which restrict the swelling of cellulose and decrease its absorption capacity [8, 9]. Various methods such as mechanical beating or refining [1, 10, 11], use of chemical additives [12–15], physical fractionation [16, 17], and blending [18, 19] are used to improve fiber flexibility and swelling ability caused by hornification. However, the methods mentioned above present physical methods in a broad sense, which failed to essentially reverse hornification and only compensated it by increasing the available bonding area. Chemical methods can effectively reverse hornification, although only few related studies have been reported. Gruber et al.[20] proposed the introduction of spacer groups, such as alpha, beta-unsaturated carbonyl compounds and amino derivatives, through derivatization, which considerably reduced the hornification tendency, but mirrored the effect of hornification by increasing the paper strength. Alkali treatment can improve the fiber swelling ability and is known to be potentially useful to partly reverse hornification [21–25]. This method effectively reverses hornification, but fails to break the irreversible hydrogen bonds. In order to fundamentally solve the issue of fiber hornification, i.e., thoroughly break the non-reversible, intra-fiber hydrogen bonds, a new method needs to be developed that does not change the chemical composition and structure of cellulose.

Recently, Zhang et al. have developed a green process for dissolving cellulose using a NaOH/urea aqueous solution pre-cooled to –12 ºC, in which cellulose is rapidly dissolved at ambient temperature (below 20 ℃) [26]. During the dissolution of cellulose in alkali solution, OH^- ions break the inter- and intramolecular hydrogen bonds of cellulose and surround the cellulose chains with negative charge, which will attract hydrated Na^+ and Li^+ ions to electrostatically bind to the hydroxyl groups of cellulose and prevent cellulose chains to aggregate with each other [27]. This solvent system effectively destroys inter- and intramolecular hydrogen bonds and presents a non-derivatizing solvent, i.e., it dissolves cellulose by intermolecular forces and does not change the fiber composition and structure. We put forward the hypothesis that the NaOH/urea aqueous solution system can fundamentally solve the issue of fiber hornification, and the mechanical properties of the resulting paper sheets should be improved by Zhang's aqueous solution system.

In order to provide quantitative data to substantiate this hypothesis and to confirm the cycle times of repaired fibers, we measured their WRV and degree of hornification. In addition, other physical properties of the resulting paper sheets, such as tensile strength, burst strength, and tear resistance, were measured according to the TAPPI Standard.

2. Materials and methods

2.1 Materials

Never-dried unbleached Eucalyptus Kraft Pulp and unbleached Eucalyptus Kraft Dry Pulp Sheets (UEKDPS, commercially available dry pulp sheets) were kindly provided by the Yunnan Yunjing forestry and the paper industry Limited by Share Ltd. (Simao, China). All chemical reagents were of analytical grade. Sodium hydroxide, urea, and anhydrous sodium sulfate were purchased from Shanghai Crystal Pure Reagents Co. (Shanghai, China), and sulfuric acid was purchased from Chengdu Institute of Chemical Reagents (Chengdu, China) and used without further purification.

2.2 Methods

2.2.1 Preparation of recycled handsheets. The UEKDPS were broken into pieces of approximately 0.3 × 1 cm^2 with a paper shredder (S152TPS3-1, Deli group Co., Ltd.) and soaked in deionized water for 24 h to a solid content of 2.0%. The resulting slurry was dispersed to a pulp consistency of 0.5% using a laboratory pulper equipped with a helical rotor for 2 min at room temperature. The dispersed unbleached Eucalyptus Kraft Pulp was used to make handsheets employing a standard handsheet making apparatus (G 8E, Gockel & Co. GmbH, München). The basis weight of the handsheets was 60 g/m^2, and the wet substrates were dried for 10 min at a temperature of 95 ℃ by laying a sheet on a heated plate of the dryer of the aforementioned standard handsheet making apparatus. This sheet was labeled as virgin paper (cycle 0). For recycling, cycle 0 handsheets were broken into approximately

0.3×1 cm^2 pieces to immerse, disintegrate, and make C1 handsheets following the method described above. The recycling procedure was repeated six times on a laboratory scale [28]. From each cycle, some of the samples were retained for WRV and physical property testing.

2.2.2 Restoration of hornified fibers. Dry pulp sheets and handsheets subjected to different cycle times were initially teared into pieces of approximately 0.3×1 cm^2 and then treated according to Zhang´s method [26]. In detail, a certain amount of an aqueous solution containing 7.0 wt% NaOH and 12.0 wt% urea was stored in a refrigerator in a 2000 mL beaker. After the solution was precooled to –13 °C, 4 wt% of the above pieces was added while vigorously stirring using a laboratory pulper equipped with a helical rotor for 2 min at ambient temperature. The resultant viscous mixtures were regenerated for 5 min in a solution containing 5 wt% H_2SO_4 and 5 wt% Na_2SO_4 and then drained, washed, and filtered with deionized water until the pH of the pulps was neutral. A certain number of restored pulps were retained for WRV testing. The remaining restored pulp was used as starting material to make handsheets according to the procedure mentioned above for the determination of the physical properties.

2.2.3 Water Retention Value. The Water Retention Values (WRV) of the pulp before and after the recycling treatment were determined according to the Chinese standard GB 29286–2012. In detail, approximately 1.5 g (o.d.) pulp was placed in a centrifuge at 3000 × g for 30 min to remove all free water. The wet pulp was weighed immediately in a pre-weighted weighing bottle and dried in an oven at 105 ± 2 °C for 24 h. Next, the dry pulp was weighed again to determine the amount of bound water. The WRV was calculated as the weight of bound water per unit of dry pulp using the following equation:

$$\mathrm{WRV},\% = \frac{\mathrm{WRV_W} - \mathrm{WRV_D}}{\mathrm{WRV_D}} \times 100\%$$

where WRV_W is the weight of the wet pulp after centrifugation and WRV_D is the weight of the dry pulp (in grams). The test was repeated four times for each sample, and average value and standard deviation were calculated.

The degree of hornification was calculated as the percentage decrease of the WRV of the dried samples (WRV_{RX}) relative to that of the never-dried samples (WRV_{R0}) using the following equation [29]:

$$\mathrm{Hornification},\% = \frac{\mathrm{WRV_{RO}} - \mathrm{WRV_{RX}}}{\mathrm{WRV_{RO}}} \times 100\%$$

2.2.4 Determination of paper sheet properties. The physical properties of the paper sheets before and after restoration were assessed by the standard method TAPPI T 220 sp-01. In detail, tensile strength, bursting strength, and tearing resistance of the paper sheets were analyzed according to TAPPI T 494 om-01, TAPPI T 403 om-02, and TAPPI T 414 om-98, respectively.

3. Results and discussion

3.1 Water Retention Value

The costs of transporting and storing dry pulp sheets are lower than for wet pulp sheets [15, 30]. Hence, the mill has transformed wet virgin pulps to dry pulp sheets, i.e., the once-dried pulp underwent wet pressing and drying. The once-dried pulp is not recycled fiber, but exhibits the highest degree of hornification [31]. For this reason, once-dried pulp was treated with NaOH/urea aqueous solution according to the procedure described above. For better verifying and comparing the repair effect of recycled fibers, we also determined the WRV of never-dried fibers. WRV and degree of hornification of never-dried fibers as well as of recycled fibers with different cycle numbers are shown in Table 1.

As shown in column 4 and 6 of Table 1, the WRV of never-dried fibers gradually declined to that of

6th-cycle fibers when the fibers underwent rewetting and drying. Such WRV decreases are accompanied by increases in the degree of hornification, especially for the first drying step. This finding is consistent with other reports [7, 25, 31–33] and is not the focus of the present work. Results in column 5 and 7 show that the WRV of repaired fibers continuously increased and is only slightly less than that of never-dried fibers. Furthermore, it mirrors a lower degree of hornification. This shows that the NaOH/urea aqueous solution can reverse the hornification of recycled fibers and greatly improve the WRV of hornified fibers. However, with an increasing cycle number, the WRV of recycled fibers treated by the dissolution/regeneration process increased drastically but was only slightly lower than that of never-dried fibers. We surmise that this is due to the loss of hemicellulose. Since hemicelluloses exhibit the highest hygroscopicity among all chemicals in the pulp, several lines of evidence suggest that loss of hemicellulose is one explanation of the decrease in the water retention ability [34–36].

Table 1. WRV and degree of hornification of primary and recycled fibers

Sources		Number of cycles	WRV/%		Degree of Hornification /%	
			Before	After	Before	After
Primary fiber	Never dried	—	139.59 (1.94)	—	0.00	—
	Once dried	—	78.42 (0.84)	146.72 (2.85)	43.82	–5.11
Recycled fiber		0	76.18 (1.03)	151.09 (2.12)	45.53	–8.24
		1	68.92 (0.85)	136.94 (1.56)	50.63	1.90
		2	66.98 (0.65)	135.47 (0.95)	52.02	2.95
		3	65.48 (0.72)	134.55 (1.08)	53.09	3.61
		4	65.07 (0.70)	133.89 (1.08)	53.38	4.08
		5	64.65 (0.63)	132.28 (0.96)	53.69	5.24
		6	62.57 (0.68)	131.01 (1.23)	55.18	6.15

Data in parentheses present the standard deviation.

3.2 Physical Properties of Paper Sheets

The water-holding capacity of repaired fibers is equivalent to that of never-dried fibers, and the physical properties of the resulting paper sheets are equivalent to those of never-dried fibers or virgin pulp (VP) fibers. We made handsheets using repaired and un-repaired fibers and measured their physical properties, such as the tensile, burst, and tear indexes according to the TAPPI standard. The test results are shown in Figure 1.

Figure 1 shows that the tensile, burst, and tear indexes of the resulting paper sheets that have been prepared from repaired fibers have increased. In particular, it should be noted that the burst and tear indexes approximately doubled compared with those of un-repaired fibers. However, although the WRV of repaired fibers (Table 1) is comparable with that of virgin pulp, the mechanical properties are much worse than those of virgin pulp handsheets. There might be two reasons for the finding that the WRV of repaired fibers is equivalent to that of virgin pulp, while the physical properties are worse than those of virgin pulp sheets. The first reason presents the breakage of the recycled paper sheets into pieces using a paper shredder before being treated with NaOH/urea aqueous solution while vigorously stirring using a laboratory pulper equipped with a helical rotor. In this series of processes, the fibers were cut off once. In consequence, the fiber length (corresponding to the amount of long fibers) reduced, and the amount of fines increased [37–39], leading to worse physical properties [1]. Another reason is the irreversible breakage of intra-fiber hydrogen bonds among the cellulose microfibrils induced by treatment with NaOH/urea aqueous solution, which reversed the fiber hornification and sharply increased the WRV of repaired fibers. This is supposed to be the main reason

for the WRV increase of repaired fibers. Consequently, the WRV of repaired fibers increased to be comparable to that of never-dried fibers, but the mechanical properties of the resulting paper sheets failed to recover to the same level as those of virgin pulp paper sheets.

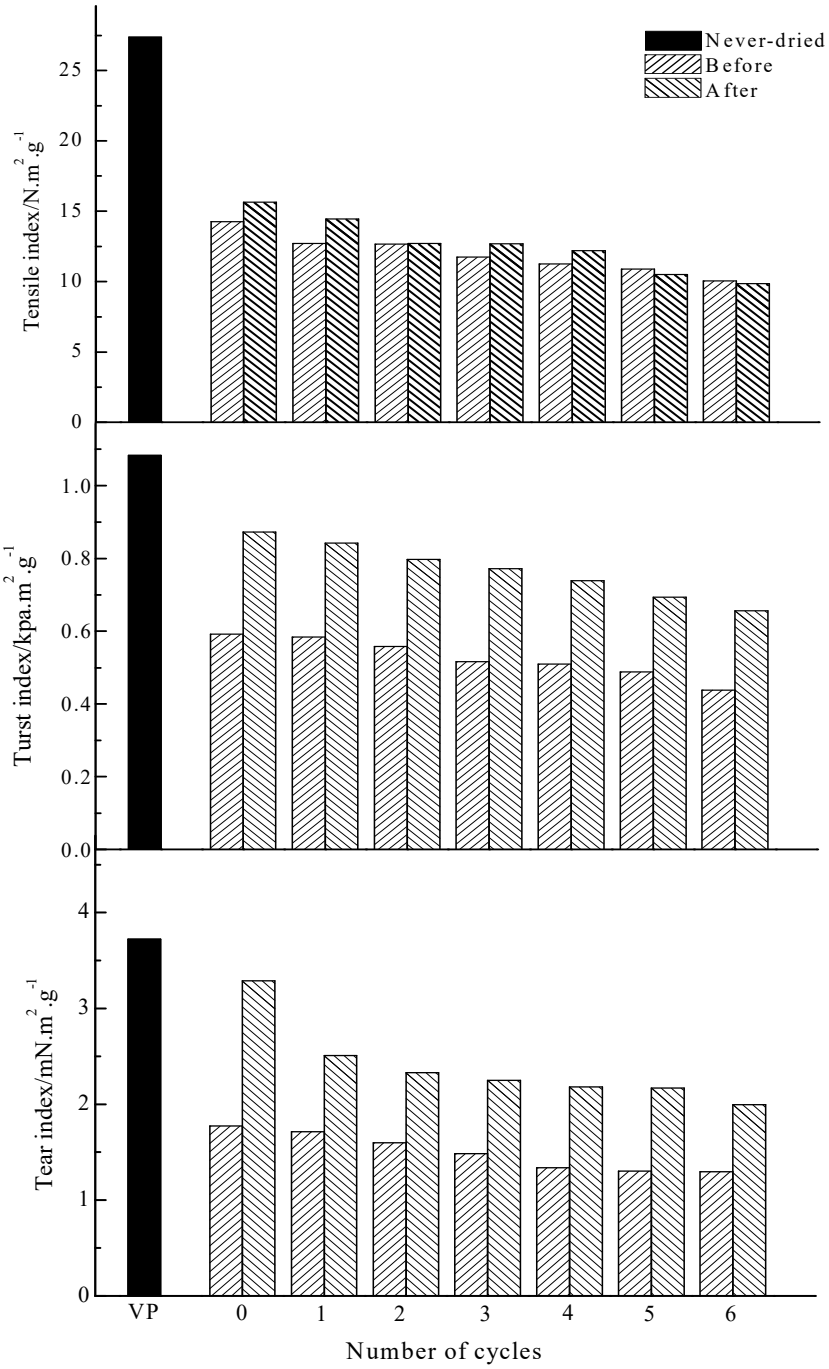

Figure 1. Effect of the dissolution/regeneration process on the paper sheet properties.

3.3 Cycle numbers of Repaired Fiber

The EPA and related experts estimates that, with current technology, the production of each metric ton recycled paper from recycled fibers saves 1.2 tons of standard coal, 600 kWh electric power, 100 m^3 water, and 300 kg chemical raw materials. Furthermore, it reduces the deforestation by 1.7×10^3 m^2 forest, landfill usage by 3 m^3, air pollution by 74%, and water pollution by 35% compared with the

production of new, virgin paper [7, 40, 41]. This means that the economic and environmental benefits for each recycling of secondary fibers are considerable, and especially for the benefit for the environment cannot be economically estimated.

In order to indicate the number of cycles of repaired fibers and repair processes required to promote or inhibit fiber hornification, we chose to repair dry pulp sheets as staring material. These dry pulp sheets have experienced drying, while the first drying step had the most dramatic effect on fiber hornification [31, 42, 43]. This effect is consistent with our experimental results obtained for the 6th recycling of once-dried fibers, which exhibited a WRV of only 62.57% of the WRV of virgin paper. These results are shown in Figure 2.

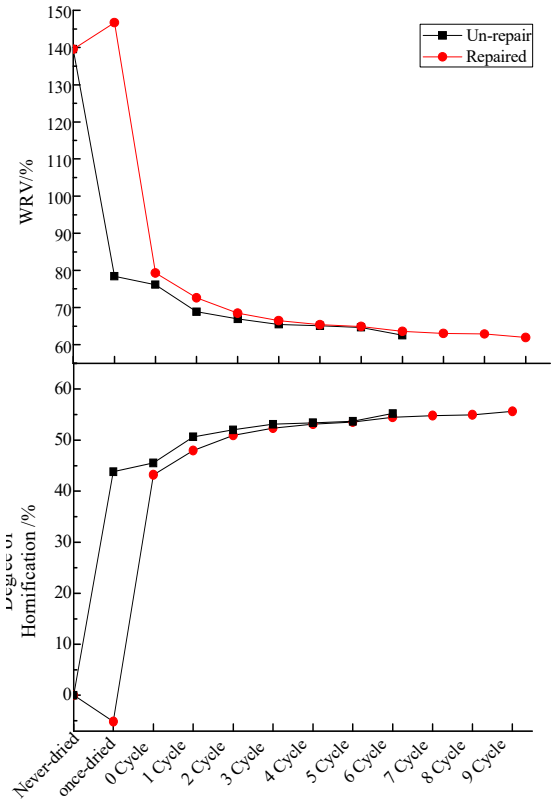

Figure 2. Effect of fiber repair on the fibers' water holding ability.

Figure 2 shows that the WRV of repaired fibers continuously decreased as the number of recycling repetitions increased, and the WRV decrease is mirrored by an increasing degree of hornification. The most rapid decrease in the WRV of repaired fibers occurs after the first recycling step, and subsequent recycling further exacerbates the WRV, which levels off after three recycling steps. This change is in principle consistent with that observed for un-repaired fibers [33, 44–47]. However, the WRV downtrend of repaired fibers is slightly less than that of un-repaired fibers, which reflects in less growth of the hornification degree, and the number of recycling repetitions doubles. This implies that the dissolution/regeneration process slightly reduces the degree of fiber hornification, which may be mainly due to the fact that natural cellulose is converted from cellulose I into cellulose II in the regeneration process [48], and cellulose II has less hydrogen bonds and is less stable [49]. Although this process does not significantly inhibit the fiber hornification, it has significant implications for the papermaking industry and especially for the environment.

4. Conclusions

The purpose of the current study was to determine the degree of hornification of recycled fibers treated

with NaOH/urea aqueous solution as well as the physical properties of the resulting paper sheets. The water retention capacity of repaired fibers has been restored to be equivalent with virgin pulp, and the hornification has been completely reversed. Moreover, the physical properties of the resulting paper sheets have improved, especially the tear and burst indexes approximately doubled. One of the most significant findings that emerged from this study is that the repair process slightly inhibited the degree of fiber hornification, and that the cycle number of recycled fibers increased.

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