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Alkaline peroxide treatment of ECF bleached softwood kraft pulps: Part 2. Effect of increased fiber charge on refining, wet-end application, and hornification

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Abstract

The effect of increased fiber charge on refining, cationic starch adsorption, and hornification was examined. Two pulps were investigated: (1) a softwood (SW) kraft pulp (KP) which was bleached elementally chlorine-free (ECF) and served as control; and (2) a control pulp treated with alkaline peroxide, which had a higher fiber charge. It was shown that increased fiber charge can improve the efficiency of the refining treatment, as indicated by differences in tensile index refined from 0 to 1000 revolutions. When the control pulp was refined from 4000 to 8000 revolutions, the tensile index decreased. In contrast, the tensile index of the higher fiber charge pulp (HCP) was higher under the same refining conditions. Upon addition of 2% cationic starch to both pulps, the tensile index of the control pulp increased by 13.7% and that of HCP by 23.7%. Atomic force microscopy did not reveal differences in the surface morphology of the two pulps with and without cationic starch adsorption. Peroxide treatment enhanced the fiber charge of the never-dried pulp. This was beneficial in reducing hornification when pulp was dried at 105°C. However, if the once dried pulp at 105°C was treated with peroxide, this resulted in a drastic decrease in intrinsic viscosity of the pulp and lower tensile and burst indices of the test sheets.

Keywords: atomic force microscopy; carboxyl; cationic starch; drying; fiber charge; hornification; peroxide; refining; tensile; wet-end.

Introduction

The environmental challenges of pulp manufacturing have been addressed by employing elementally chlorine-free (ECF) and totally chlorine-free (TCF) bleaching technologies. A key future task is the development of new fiber properties by modification of fibers using enzymatic, chemical, and physical methods. The fiber surface has been modified by means of carboxymethylation (Barzyk et al. 1997; Laine et al. 2000, 2003a,b), xyloglucan adsorption (Zhou et al. 2006), cationic polymer adsorption, including cationic poly(amideamine) epichlorohydrin condensate, polyacrylamide, chitosan, and cationic starch as additives (Bobacka et al. 1999; Liu et al. 2001; Gardlund et al. 2003; Nanko 2003; Gaiolas et al. 2004, 2005; Li et al. 2004; Eriksson et al. 2005; Krupin 2005; Stanciu 2005). A number of chemical modification studies involving catalyzed oxidation of fibers have also been reported, including TEMPO-mediated oxidation (TEMPO =2,2,6,6-tetrametyl-1-piperidinyloxy radical) (Kitaoka et al. 1999; Le Roux et al. 2005) and periodate oxidation (Schmidt et al. 1995; Liu et al. 2001). The objective of the former oxidation is to enrich carboxyl groups of pulp fibers by converting primary alcohol groups to carboxylates. The latter can introduce aldehyde groups by cleavage of the C2–C3 bond of cellulose.

It has been well established that mechanical treatment of fibers can modify their morphology and structure (Jordan and Page 1979; Page et al. 1983; Miller 1989; Page 1989; Batchelor et al. 1999; Fardim and Duran 2003; Sjoberg and Hoglund 2005). The objective of refining of chemical pulps is to improve the properties of the end products (Hietanen and Ebeling 1990; Wang et al. 2006). It has been reported that unbleached softwood kraft pulp (SW-KP) with high charge suffers less damage during refining than low-charge pulp, and the high-charged pulp (HCP) yields higher tensile strength than the low-charged pulp when analyzed at constant sheet density (Hiltunen et al. 1999).

The term hornification refers to stiffening of the polymer structure in pulps that occurs upon drying or water removal (Fernandes Diniz et al. 2004). Hornification leads to lower fiber bonding and loss of swelling (Lindström and Carlsson 1982; Kato and Cameron 1999; Maloney and Paulapuro 2000; Wang et al. 2003). Carboxyl groups can influence the hornification of kraft fibers. For example, Lindström and Carlsson (1982) carboxymethylated fibers and found that the degree of hornification decreased if carboxyl groups were in their ionized form (instead of proton form) during drying.

Carboxyl groups of fibers also play a key role in papermaking, as many of the interactions between soluble and particulate fractions of the furnish are charge-based (Sanders and Schaefer 1995; Isogai et al. 1997). Cationic starch (CS), which is generally a starch ether having a

quaternary ammonium group, is one of the most popular dry-strength additives and retention aids in the papermaking industry (Bobacka et al. 1999; Eriksson et al. 2005; Gaiolas et al. 2005; Maximova et al. 2005; Stanciu 2005; Yan et al. 2005).

The aim of this study was to investigate how improved fiber charge obtained from a peroxide-stage treatment affects refining, wet-end application, and hornification. On the basis of Part I of this study (Dang et al. 2007), an ECF bleached SW-KP was treated with 2% NaOH and 1% H_2O_2 at 60°C, resulting in a 12.8% increase in fiber charge. This study examines the effect of the change in fiber charge with respect to refining, CS adsorption, and drying.

Materials and methods

Materials

The ECF bleached pulp described by Dang et al. (2007) was studied. HCP was generated under the following conditions: 2% NaOH, 1% H_2O_2, 60°C, 10% consistency, 2 h. A cationic corn starch (CATO 31) with 0.35% quaternary nitrogen substitution was obtained from National Starch and Chemical Company. All other chemicals were purchased from J.T. Baker and Aldrich-Sigma as analytical grade and were used as received.

Carboxyl group content and surface charge of pulp fibers

Conductometric titration was employed to measure carboxyl group content (Katz et al. 1984; Lloyd and Horne 1993), while the surface charge of the fibers was determined by polyelectrolyte adsorption (Wågberg et al. 1989). Detailed procedures for determination of fiber charge and surface charge were presented in a previous study (Dang et al. 2006). Data for fiber charge had an error of less than ±3% and the surface charge data were evaluated as expressed by a reliable standard deviation at 95% confidence level.

Testing of physical properties

Physical testing of the test sheets included tensile strength (Tappi standard T494 om-88) and burst strength (Tappi standard T403 om-91) determination. The experimental error was less than ±5%. The fiber length, curl, and kink were characterized using a fiber quality analyzer (FQA) and the results are presented in Table 1. Freeness of pulp was measured according to Tappi standard T227 om-92.

Table 1 Properties of the control pulp and higher fiber charge pulp (HCP).

Pulp properties	Pulp sample	
	Control pulp	HCP
Brightness (Tappi standard)	84.5	87.3
Intrinsic viscosity (ml g^{-1})	672	628
Carboxyl group content (mmol 100 g^{-1} o.d. pulp)	3.98	4.49
Freeness (ml)	696±5	690±5
FQA of fibers		
Fiber length (mm)	1.090±0.034	0.995±0.032
Curl	0.144±0.006	0.128±0.006
Kink index (l mm^{-1})	1.48	1.38

PFI refining of pulp fibers

PFI refining was based on Tappi standard T248 cm-85. The various beating degrees of the pulps are defined by the number of revolutions (r), namely: 500, 1000, 2000, 4000, and 8000 r.

CS adsorption onto fibers

A CS solution was prepared according to Yan et al. (2005): 1% starch solution was cooked at 95°C for 30 min. The pulp slurry was prepared at a consistency of 0.40%. CS was added at concentrations of 0.25%, 0.50%, 1.0%, and 2.0% to the pulp (based on o.d. fibers) and allowed to adsorb for 30 min. Subsequently, handsheets were prepared based on Tappi standard T205 om-88.

Surface morphology by atomic force microscopy (AFM)

A silicon nitrile cantilever tip was applied using a Digital Instruments 3100 scanning probe microscope. The following samples were analyzed: (1) control pulp; (2) control pulp treated with 2% CS; (3) HCP; and (4) HCP treated with 2% CS.

Drying and hornification

Six pulp samples were prepared as summarized in Table 2. Sample 1 was a handsheet of the control pulp air dried at 23°C (Tappi standard conditioning room temperature). Sample 2 was control pulp dried at 105°C on a hot plate. Samples 3 and 4 were handsheets of HCP dried at 23°C and 105°C, respectively. Sample 5 was rewetted sample 2 in water, which was disintegrated, filtered, alkaline peroxide-treated, and dried at 23°C. Sample 6 was the same as sample 5, but dried at 105°C. The samples were dried on a hotplate at a constant temperature of 105°C for

Table 2 Properties of the control pulp and higher fiber charge pulp (HCP) dried at 23°C and 105°C.

Pulp sample	Drying temperature (°C)	Intrinsic viscosity (ml g^{-1})	Total carboxyl group content (mmol 100 g^{-1})	Tensile index (Nm g^{-1})	Burst index (kPa m^2 g^{-1})
1. Control pulp	23	672	3.98	32.8	2.6
2. Control pulp	105	667	3.97	29.0	2.1
3. Peroxide-treated pulp[a]	23	628	4.49	36.0	2.7
4. Peroxide-treated pulp[a]	105	616	4.45	31.5	2.4
5. Control pulp[b]	105 and 23	544	4.12	23.2	1.6
6. Control pulp[c]	105 and 105	542	4.11	19.2	1.0

[a] Peroxide treatment conditions: 2% NaOH, 1% H_2O_2, 60°C, 10% consistency, and 2-h treatment.
[b] First dried at 105°C, peroxide-treated[a], and finally dried at 23°C.
[c] First dried at 105°C, peroxide-treated[a], and finally dried at 105°C.

10 min. The intrinsic viscosity, carboxyl group content, tensile index, and burst index of each sample were determined (Table 2).

Results and discussion

Based on the results from Part I of our studies (Dang et al. 2007), it has been concluded that the bulk fiber charge of a fully bleached SW-KP can be increased by 12.8% with the treatment parameters 2% NaOH, 1% H_2O_2, 60°C, 10% consistency, and 2-h treatment time. The properties of the control pulp and the HCP are summarized in Table 1. Freeness values do not show an obvious difference between the two pulps. Given these results, the effects of enhanced fiber charge were investigated on refinability, CS adsorption, and hornification.

Influence of elevated fiber charge on refining

HCP and control pulp were PFI refined at 500, 1000, 2000, 4000, and 8000 r. Fiber charge values of 3.98–4.00 mmol 100 g^{-1} (control pulp) and 4.45–4.51 mmol 100 g^{-1} (HCP) were observed. Total fiber charge properties of both pulps remained relatively constant throughout the refining. Thus, PFI refining does not appear to alter total fiber charge properties, a finding that is in agreement with the results of Bhardwaj et al. (2004).

The surface charge, on the other hand, increased as a function of PFI revolutions (Figure 1a). As expected, refined pulp adsorbs more cationic polymers onto the fiber surface than unrefined pulp. The surface charges of the two pulps without refining were statistically the same (Figure 1a). We can safely conclude that peroxide treatment of ECF bleached pulp does not significantly affect the surface charge.

Figure 1b shows the tensile indices plotted against the level of PFI refining. The initial tensile strength of the HCP was 10% higher than the control pulp. The differences in tensile indices between the two pulps were increased to 15.4% when refining took place at 1000 r. On the basis of Figure 1b, the tensile indices of the two pulps are identical if refined at 2000–4000 r. The control pulp exhibits a reduction in tensile strength for an increase from 4000 to 8000 r.

It is well known that refining enhances the surface area of fibers (Khalandovskii et al. 1971; Seo et al. 2003;

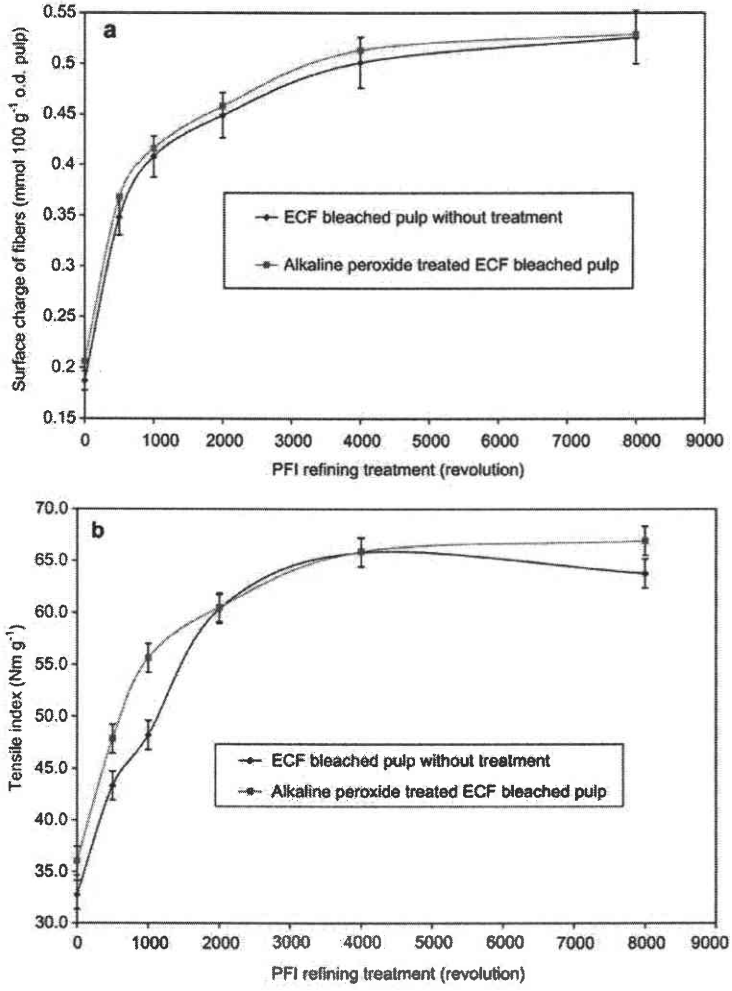

Figure 1 Effect of increased fiber charge on PFI refining: (a) surface charge and (b) tensile strength against refining treatment of the control pulp and the alkaline peroxide-treated pulp.

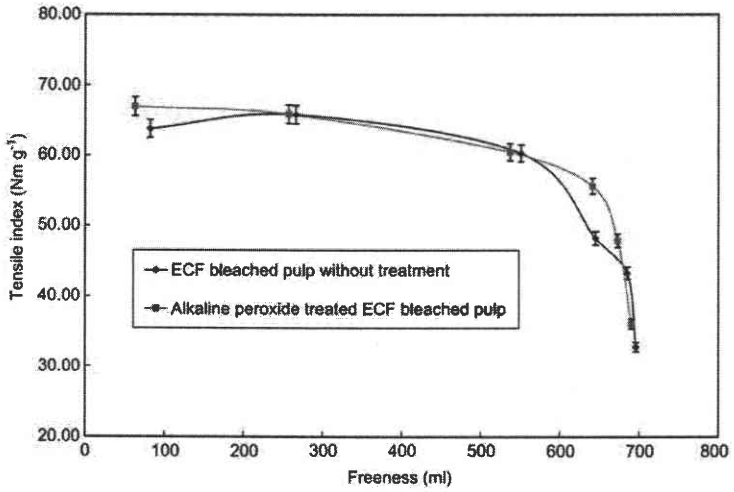

Figure 2 Comparison between tensile strength and freeness of the control pulp and the alkaline peroxide-treated pulp after refining.

Bhardwaj et al. 2007). Also, Carrasco et al. (1996) found that freeness is related to the total surface area of fibers after refining. The tensile strength is plotted against the freeness in Figure 2. It is obvious that HCP has better tensile indices than for the control pulp in the freeness range between 540 and 670 ml and below 260 ml. This observation is in agreement with the results illustrated in Figure 1b.

The increased surface area of fibers after milling also leads to better bonding (Paavilainen 1994), but may damage the fiber quality, which results in lower single fiber strength and lower final paper strength (Robertsen and Joutsimo 2005). In this study the tensile strength of the control pulp increased under the mild refining treatment, but decreased at higher levels of refinement. It is well established that the strength of the final paper is primarily controlled by fiber-fiber bonding at low levels of mechanical treatment, but the single fiber strength may become more important at high levels of refining. However, this is not the case for the HCP; it is more beneficially responsive to mechanical refining from the very beginning.

Influence of increased fiber charge on CS adsorption onto fibers

The wet-end behavior of the pulps was evaluated in the context of CS as additive. Usually, papermakers add approximately 4.54 kg ton^{-1} (10 lb ton^{-1}), i.e., 0.454%, of CS into the system and then gradually optimize it from that point (Hubbe 2007). In this study, dosages of 0.25, 0.50, 1.0, and 2.0% (based on o.d. pulp) were applied to both pulps in focus. Figures 3 and 4 show the results for tensile and burst strength. It is apparent (Figure 3) that the tensile index increased 23.7% with 2% CS application to the HCP in comparison to a 13.7% increment for the control pulp. The tensile index did not change very

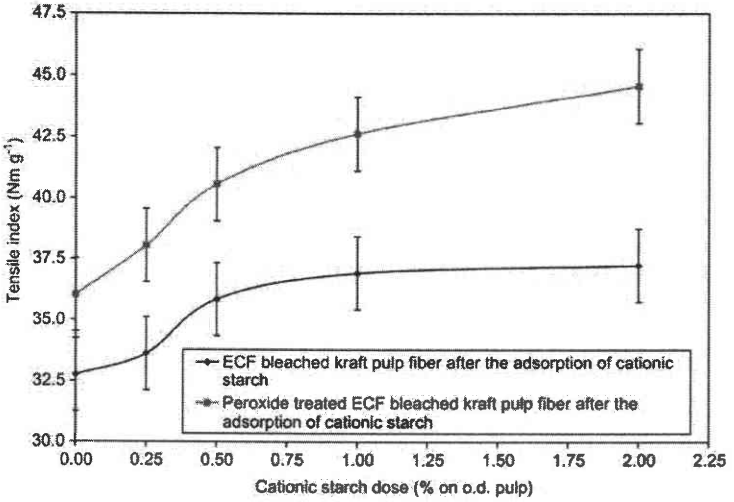

Figure 3 Comparison of tensile index between the control pulp and the alkaline peroxide-treated pulp after adsorption of cationic starch onto the fibers.

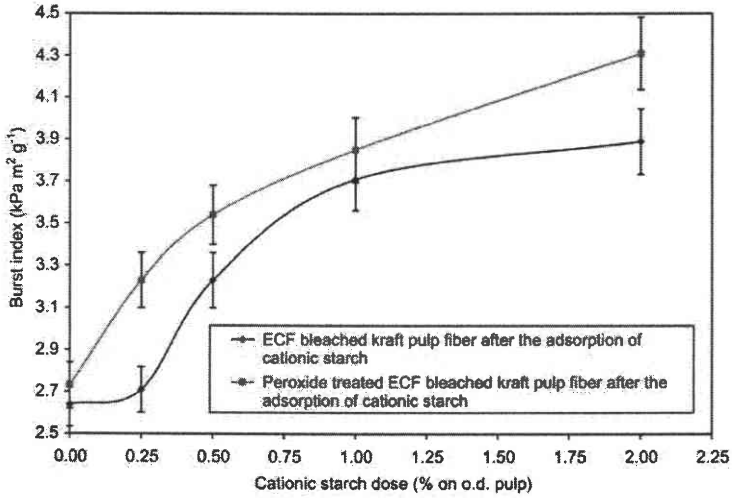

Figure 4 Comparison of burst index between the control pulp and the alkaline peroxide-treated pulp after adsorption of cationic starch onto the fibers.

much for 1% CS addition to the control pulp. However, the tensile index of the peroxide-treated pulp exhibited a continuing increase, even at higher application levels. As shown in Figure 4, the greatest increase in burst indices was 47.4% (control) and 57.9% (HCP) when 2% CS was applied. Clearly, the efficiency of CS adsorption is better in the case of HCP.

Tensile index has a linear relationship with burst index (Figure 5). The slope of the control sample trend line is lower than that of the enhanced fiber charge pulp.

Surface morphology and roughness of CS-treated fibers measured by AFM

AFM is an effective tool for evaluating surface morphology and properties of wood, fibers, and cellulose. In our previous study on holocellulose fibers, we reported no apparent relationship between roughness and surface charge of fibers (Dang et al. 2006). Figure 6 shows AFM phase images of the control, the control treated with 2% CS, HCP, and HCP treated with 2% CS. No striking differences in the surface topography among the samples were found.

Influence of increased fiber charge on hornification

Six samples were prepared and the data are summarized in Table 2. By comparing the results of pulps dried at 23°C (samples 1, 3, and 5) with those dried at 105°C (samples 2, 4, and 6), it can be observed that drying at elevated temperature does not have an obvious effect on intrinsic viscosity and fiber charge. However, high-temperature drying does decrease physical strength properties, including tensile and burst strength for both control pulp and HCP.

The tensile index of sample 2 decreased by 11.6% compared to that for sample 1, while the decrement was 12.5% between samples 4 and 3. In terms of the effect

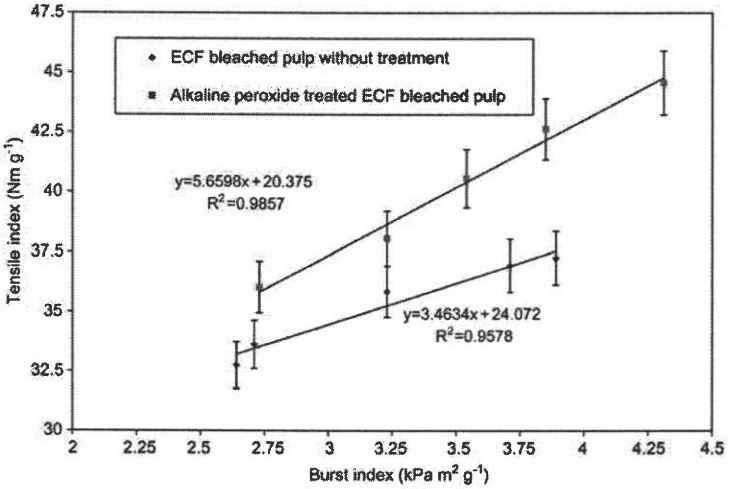

Figure 5 Comparison of burst index against tensile index of the control pulp and the alkaline peroxide-treated pulp after adsorption of cationic starch onto the fibers.

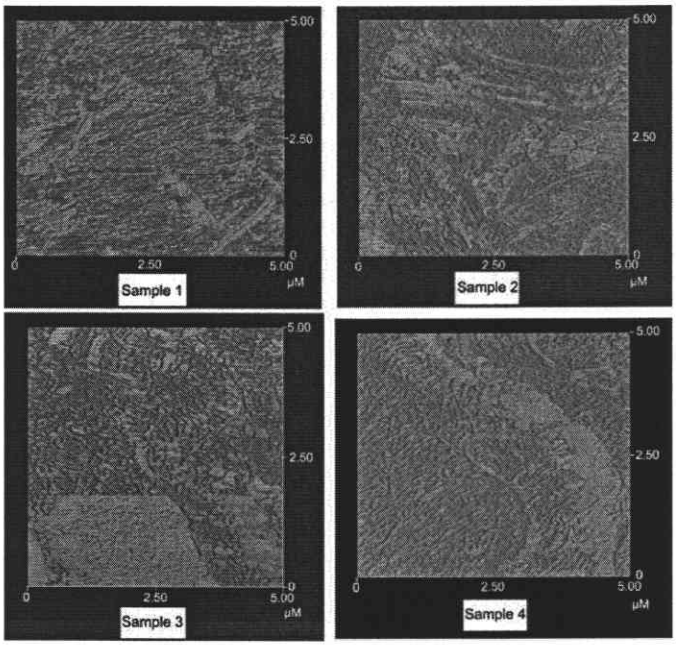

Figure 6 Atomic force microscopy phase images of pulp fibers: 1, control pulp; 2, control pulp treated with 2% cationic starch; 3, higher fiber charge pulp (HCP); and 4, HCP treated with 2% cationic starch.

of peroxide treatment on hornification, the tensile and burst indices of sample 4 were 8.6% and 14.3% higher, respectively, than the values for sample 2. Accordingly, increased fiber charge can reduce the effect of hornification for never-dried fibers to some extent. On the contrary, the tensile and burst indices of sample 6 showed a drastic decrease compared to those of samples 2 and 4. Although the carboxyl group content of sample 6 was 3.5% greater compared to sample 2, the tensile index of sample 6 decreased by 33.8% (vs. sample 2) and 39.0% (vs. sample 4). Similar to the results for the tensile index, the burst index decreased by 52.4% and 58.3%. These results indicate that peroxide treatment of oven-dried pulps cannot be used to recover physical strength properties. This treatment is in some cases detrimental.

Conclusions

Increased fiber charge can enhance the efficiency of refining between freeness values of 540 and 670 ml and lower than 260 ml. In these ranges, the tensile index of HCP is greater than that of the control pulp. The tensile index increased 23.7% with 2% starch application to HCP, while the tensile index of the control pulp was improved by 13.7%. The burst index of the control pulp and HCP increased by 47.4% and 57.9% after 2% cationic starch addition, respectively. When the never-dried pulp was treated with peroxide, the increased fiber charge reduced hornification at 105°C in comparison to the control pulp. Peroxide treatment of the once-dried pulp at 105°C increased hornification, manifest as very low intrinsic viscosity, tensile and burst indices.

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