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# Influence of hemicellulosic derivatives on the sulfate kraft pulp strength

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## ABSTRACT

In this work the application of different hemicellulosic derivatives including different degrees of substitution (DS) cationic hemicelluloses and carboxymethyl hemicelluloses in sulfate kraft pulp were investigated in detail. It was found that cationic hemicelluloses and carboxymethyl hemicelluloses could improve the physical properties of hand sheets, while the cooperation of cationic hemicelluloses and carboxymethyl hemicelluloses could enhance sharply the physical properties of hand sheets. When the dosages of cationic hemicelluloses with DS of 0.37 and carboxymethyl hemicelluloses with DS of 0.35 were 1.0% and 1.0% (based on dry pulp weight), respectively, the physical properties of hand sheets was significantly improved, breaking lengthen and tear index of hand sheets increased by 21.1% and 54.6% compared with the control hand sheets without any additives added. The fiber intertexture of the hand sheets was observed by SEM. The results indicated that hemicelluloses could be used as the source for the production of wet-end additives in papermaking, which might represent an important strategy for sustainable use of the agricultural residue.

## 1. Introduction

Hemicelluloses are plant cell wall polysaccharides closely associated to cellulose. Unlike the latter, hemicelluloses are composed of different sugar units, have more short side chains, being non-crystalline heteropolysaccharides whereas cellulose is formed by only β-(1,4) glucosyl linkage in a linear backbone. Hemicelluloses are, nevertheless, the most complex components in the cell wall of woods, straws and grasses, forming hydrogen bonds with cellulose, covalent bonds (mainly α-benzyl ether linkages) with lignins and ester linkages with acetyl units and hydroxycinnamic acids. Heimicelluloses consist of various different sugar units, arranged in different proportions and with different substituents (Aspinall, 1959; Ehrenthal, Montgomery, & Smith, 1954; Glaudemans & Timell, 1958; Timell, 1965; Whistler, 1950). The main sugars are D-xylose, L-arabinose, D-glucose, D-galactose, D-mannose, D-glucuronic acid, 4-*O*-methyl-D-glucuronic acid, D-galacturonic acid and, to a lesser extent, L-rhamnose, L-fucose, and various *O*-methylated neutral sugars.

Functionalization creates novel opportunities to exploit the various valuable properties of hemicelluloses for previously unconceived applications (Ebringerova & Heinze, 2000). Carboxymethylation is one of the most versatile functionalization procedures as it provides access to bio-based materials with valuable properties. Furthermore, quaternization is also one of the effective functionalization procedures. In particular, quaternization of the hydroxyl groups of hemicelluloses is to increase their hydrophilicity. Guar gum and starch are polysaccharides currently used as wet-end additives in papermaking, whereas hemicelluloses and their derivatives have been reported to improve paper quality. Based on our previous work of chemical modification of hemicelluloses (Ren, Sun, & Liu, 2007; Ren, Sun, Liu, Chao, & Luo, 2006), the influence of quaternized hemicelluloses and carboxymethyl hemicelluloses as wet-end additives on the physical properties of hand sheets were studied in this work. Scan Electron Microscope (SEM) was used to observe the intertexture of fibers. Hand sheets were characterized by FT-IR.

## 2. Experimental

### 2.1. Materials

Hemicelluloses were isolated using 10% KOH at 25 °C for 10 h with a solid to liquor ratio of 1:20 (g $\text{ml}^{-1}$) from the holocellulose obtained by delignification of sugarcane bagasse (SCB) with sodium chlorite in acidic solution (pH 3.7–4.0 adjusted by 10% acetic acid) at 75 °C for 2 h. 2,3-Epoxypropyltrimethylammonium chloride (ETA) were purchased from Dongying fine chemicals Ltd., Shandong, China. Other chemicals were of analytical-reagent grade and purchased from Guangzhou Chemical Reagent Factory, Guangdong, China.

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### 2.2. Synthesis of cationic hemicelluloses

Modification was carried out in three-necked flask fitted with mechanical stirrer and a reflux condenser. The procedure of synthesis of cationic hemicelluloses is as follow. The dried hemicelluloses (0.66 g, 0.01 mol hydroxyl functionality in hemicelluloses) were dispersed in 20 ml distilled water and heated to 60 °C for 20 min, and then sodium hydroxide (the molar ratio of sodium hydroxide to etherifying agent was varied from 0.1 to 2.0) was added at 60 °C for 20 min. Subsequently, ETA (the molar ratio of etherifying agent to anhydroxylose units in hemicelluloses was varied from 0.5 to 3.0) was added. The reaction mixture was additionally stirred for 5 h at 60 °C. After cooling down, the solution was neutralized with 0.1 M HCI and precipitated with 80% ethanol. The resulting hemicellulosic derivative precipitate was filtered off and washed thoroughly with 95% ethanol to remove residual reagents. The purified product was first air-dried for 12 h and then further dried in an oven at 45 °C for 24 h. This procedure was depicted in a previous paper (Ren et al., 2006). The products with degree of substituents ($DS_N$) of 0.09–0.55 could be obtained and their characterizations were detected as the method described in a previous paper (Ren, Liu, Sun, She & Liu, 2007).

### 2.3. Synthesis of carboxymethyl hemicelluloses

The typical procedure of the synthesis of carboxymethyl hemicelluloses was below: About 0.66 g of hemicelluloses was weighed and added to 50 ml three-necked flask and followed by 40% NaOH (accounting for all amount sodium hydroxide), then the mixture was stirred at 30 °C for 20 min. The slurry medium in the bottle was added up to 15 ml of ethanol:water in appropriate ratios. Followed by the addition of sodium monochloroacetate (the molar ratio of SMCA to anhydroxylose units in hemicelluloses varied from 0.5 to 3.0 mol), the temperature was raised to 65 °C for 75 min. During the etherification reaction, the remaining sodium hydroxide was added after the addition of SMCA. The reaction mixture was neutralized with diluted acetic acid, and then the resulting polymer was filtered off and washed one time with 50 ml 65% aqueous ethanol (v/v), and four times with 50 ml 95% ethanol. The product was first air-dried for 12 h and then further dried at 45 °C in a vacuum oven for 24 h. The products with the degree of substituents (DS) of 0.10–0.56 were obtained, and their characterization was described as the method mentioned in a previous paper (Ren, Sun, & Peng, 2008).

### 2.4. Determination of degree of substitution (DS) of cationic hemicelluloses

The nitrogen and carbon contents of cationic hemicellulsic derivatives were minored by a Vario EL II Elemental Analyzer (Elementar, Germany). The DS of hemicellulosic derivatives was calculated from the ratio of the nitrogen to the carbon content according to the equation (Schwikal, Heinze, Ebringerova, & Petzold, 2006):

$$\mathrm{DS} = (60 \times \%\mathrm{N})/(14 \times \%\mathrm{C} - 72 \times \%\mathrm{N})$$

### 2.5. Determination of degree of substitution of ionic hemicelluloses

To determine the purity of the resulting carboxymethyl hemicelluloses derivatives, exact 0.5 g of carboxymethylated product was dissolved with 10 ml of water and stirred. Followed by adding 10 ml of 1 M hydrochloric acid, and then the mixture were agitated to dissolve completely. Five drops of phenolphthalein indicator was added into the mixture, and then 1 M sodium hydroxide was added dropwise with stirring until red color of the solution didnot disappears. Fifty milliliters of 95% ethanol was slowly added in the mixture with stirring. Then, 100 ml of 95% ethanol was added and the mixture was left to settle for 15 min. After the solution had settled, the supernatant liquid was filtered by G3 type glass cullender and discarded. The precipitate was washed four times with 80% ethanol. The precipitate was then washed again with 50 ml of 95% ethanol. The precipitate was dried in an oven at 105 °C for 4 h.

Absolute values of degree of substitution (DS) were determined by acidimetric titration. Exact 0.2 g of carboxymethyl hemicelluloses was weighed in 250 ml flask, and then 50 ml distilled water was added and stirred for 10 min. pH value of the solution was adjusted up to 8 by acid or alkali. Then the solution was titrated with 0.05 M $H_2SO_4$ until pH value of solution was 3.74 by acidometer. The degree of substitution was calculated based on the equations below.

$$a = (\mathrm{m}')/\mathrm{m}$$
$$B = 2 \times M \times V/(a \times \mathrm{m})$$
$$\mathrm{DS} = 0.132 \times B/(1 - 0.08 \times B)$$

Where $a$ is the purity of carboxymethyl hemicelluloses. $m'$ and $m$ = g carboxymethylated products purified after and before, $M$ = normality of $H_2SO_4$ used, $V$ = mL $H_2SO_4$ used to titrate sample, $B$ = m mol/g of $H_2SO_4$ consumed per gram of carboxymethylated products (Fan & Xia, 1997).

### 2.6. Preparation of hand sheet formation

Five sheets were formed for each treatment according to GB 2828-81 norms (Chinese Technical Association of Pulp and Paper). These sheets were made to have a grammage (weight per unit area) of ~60 g/m$^2$ and a thickness of 0.110 mm. The pulp (BKP) was obtained from wood of spruce by kraft process being subsequently delignified and bleached. The refining process was performed to achieve ~50°SR (Schopper-Riegler degree). Aqueous solutions of hemicellulosic derivatives were added to the pulp fiber (0.5% consistency) during homogenization for limited time before sheet formation.

### 2.7. Mechanical properties of sheets

The mechanical properties of the hand sheets were tested by China norm (Chinese Technical Association of Pulp and Paper). At least 12 tests strips were evaluated for each pulp sample, and the standard deviation was always less than 3 U.

### 2.8. FT-IR spectra

FT-IR spectra of hand sheets with additives or not were obtained on an FT-IR spectrophotometer (Nicolet 510) using a KBr disc containing 1% finely ground samples. Thirty-two scans were taken of each sample recorded from 4000 to 400 cm$^{-1}$ at a resolution of 2 cm$^{-1}$ in the transmission mode.

### 2.9. SEM test of hand sheets

The condition of fiber intertexture of hand sheets was detected by Scanning Electron Microscope (SEM, LEO/530V, and Germany). The samples tested were prepared by spraying gold on sheets to form film so as to enhance the conductivity of hand sheet samples.23

## 3. Results and discussion

### 3.1. Influence of cationic hemicelluloses with different DS on the physical properties of hand sheets of BKP pulp

In general, an increase in the dry tensile strength of paper products can be achieved either by mechanical processes to insure adequate formation of hydrogen bonding between the hydroxyl groups of adjacent papermaking fibers, or by the inclusion of certain dry strength additives (Hartmans et al., 2004). Strength additives that are added prior to the papermaking (wet-end addition) should preferably have cationic functionalities so that they are easily retained by the cellulose fibers, which are naturally anionic. The chemical structure of cationic hemicellulosic derivatives synthesized is shown (Fig. 1). The cationic products with DS values of 0.28, 0.37, and 0.53 were selected as wet-end additives in the application of papermaking. A first set of experiments was carried out in order to determine the effect of cationic hemicelluloses with different DS and isolated hemicelluloses on the physical properties of hand sheets of BKP pulp. The results are summarized in Table 1. As shown clearly, with increasing DS value of cationic hemicelluloses the physical properties of hand sheets improved. Breaking length of hand sheets increased from 4.35 to 4.50 km, and burst index of hand sheets increased from 6.23 to 6.75 kPa $m^2$/g, corresponding to the increment of DS values of cationic hemicelluloses from 0.28 to 0.53. The increment in physical properties of hand sheets was explained that cationic groups on the backbone of hemicellulosic derivatives could adsorb ionic fibers to form the static sorption force (Ma & Qiu, 2004), which could improve the combined forces among fibers so that the physical properties of hand sheets obviously improved. In addition, the hydroxyl groups in cationic hemicelluloses could form hydrogen bonds with fibers, which could improve the physical properties of hand sheets. While tear index of hand sheets decreased. Because the fiber length was the best important factor on tear index. The physical properties of the sample H of hand sheets with the addition of hemicelluloses were improved. Breaking length and burst index increased by 2.5% and 10.9%, compared with control sample without any additives. Therefore, it can be concluded that the cationic hemicelluloses could more highly improve the physical properties of hand sheets than unmodified hemicelluloses.

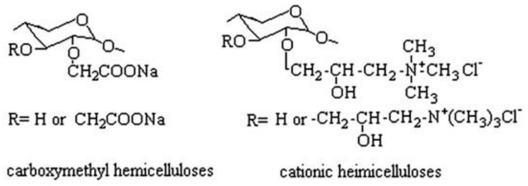

**Fig. 1.** The chemical structure of cationic hemicelluloses and carboxymethyl hemicelluloses.

**Table 1**
Influence of different degrees of substitution (DS) of cationic hemicelluloses on the physical properties of hand sheets of BKP pulp

| Sheet sample | $DS_N$ | Breaking length (km) | Burst index (kPa $m^2$/g) | Tear index (mN $m^2$/g) |
|---|---|---|---|---|
| YH-1 | 0.28 | 4.35 | 6.23 | 11.22 |
| YH-2 | 0.37 | 4.42 | 6.51 | 9.86 |
| YH-3 | 0.53 | 4.50 | 6.75 | 9.19 |
| H | 0.00 | 4.43 | 6.84 | 9.02 |
| Control | | 4.32 | 6.17 | 7.9 |

*Note:* the dosage of cationic hemicelluloses was 1.0% (base on weight dry pulp), and the stirring time of pulp after addition of cationic hemicelluloses was 5 min. Sheet samples from YH-1 to YH-3 were the hand sheet with addition of cationic hemicelluloses, and sample of H was the hand sheet with addition of the unmodified hemicelluloses isolated from sugarcane bagasse.

### *3.2. Influence of carboxymethyl hemicelluloses with different DS on the physical properties of sheets of BKP pulp*

If ionic chemicals as strengthening agents in papermaking, it need to cooperate with aluminium sulfate (Zhang, Luo, Feng, & Wang, 2008). Aluminium sulfate absorbs fibers and fine fibers to form absorption points with positive charges, and then act with ionic groups by coordination bond, which improve the physical properties of hand sheets. Thus in this work a certain amount of aluminium sulfate must be put into pulp before the addition of carboxymethyl hemicelluloses obtained with DS of 0.15, 0.26, 0.35, and 0.45 (Fig. 1). Influence of carboxymethyl hemicelluloses with different DS on the physical properties of hand sheets of BKP pulp is given in Table 2. As can be seen, carboxymethyl hemicelluloses could obviously improve the physical properties compared with the control sample without any additive. When carboxymethyl hemicelluloses with DS value of 0.15 was added into BKP, breaking lengthen and burst index was maximum, and increase by 15.0% and 11.3% compared with that of the control sheets. The increase in the physical properties of hand sheets was interpreted that carboxyl groups acted by coordination bond with the complex with positive charges formed by absorption of $Al^+$ and fibers, so that the combined force between fibers increased, and bonds energy enhanced, the physical properties of hand sheets augmented. In addition, carboxymethyl hemicelluloses with hydroxyl groups acted with hydroxyl groups of fibers to form hydrogen bonds, which could improve the physical properties of hand sheets. As shown also in Table 2, with increasing in DS values of carboxymethyl hemicelluloses, the physical properties of hand sheets didnot increase. It maybe be ascribed that there was not enough aluminium sulfate to act with carboxyl groups of hemicellulosic derivatives. If the amount of aluminium sulfate was not enough, it would induce ionic fibers to exclude carboxymethyl hemicelluloses, which did not result in the increasing physical properties of the hand sheets.

**Table 2**
Influence of different degrees of substitution (DS) of carboxymethy hemicelluloses (CMH) on the physical properties of hand sheets of BKP pulp

| Sample | DS | Breaking length (km) | Burst index (kPa $m^2$/g) | Tear index (mN $m^2$/g) |
|---|---|---|---|---|
| CMH-1 | 0.15 | 4.97 | 6.87 | 9.66 |
| CMH-2 | 0.26 | 4.60 | 6.40 | 9.46 |
| CMH-3 | 0.35 | 4.32 | 6.86 | 11.30 |
| CMH-4 | 0.45 | 4.56 | 6.21 | 9.85 |
| Control | | 4.32 | 6.17 | 7.9 |

*Note:* pH value of pulp was adjusted to 6.5, the dosage of $Al_2(SO_4)_3$ was 2.0% (based on dry pulp), the dosage of carboxymethyl hemicelluloses was 1.0%, the stirring time of pulp after addition of carboxymethyl hemicelluloses was 5 min.

**Table 3**
Influence of the cooperation of cationic hemicelluloses (YH) and carboxymethyl hemicelluloses (CMH) on the physical properties of hand sheets of BKP pulp

| Sample | Dosage of YH and CMH | Breaking length (km) | Burst index (kPa $m^2$/g) | Tear index (mN $m^2$/g) |
|---|---|---|---|---|
| 1 | 1.0%+0.3% | 5.22 | 7.47 | 8.00 |
| 2 | 1.0%+0.5% | 4.92 | 7.35 | 8.25 |
| 3 | 1.0%+0.8% | 4.98 | 6.36 | 10.87 |
| 4 | 1.0%+1.0% | 5.23 | 7.35 | 12.21 |
| 5 | 0.8%+1.0% | 4.93 | 7.73 | 10.70 |
| 6 | 0.5%+1.0% | 5.22 | 6.99 | 11.10 |
| Control | 0% | 4.32 | 6.17 | 7.90 |

*Note:* the dosage of $Al_2(SO_4)_3$ was 1.5%, DS values of CMH was 0.35, and DS values of YH was 0.37, the stirring time of pulp after addition of carboxymethyl hemicelluloses was 5 min.

### 3.3. Influence of the cooperation of cationic hemicelluloses (YH) and carboxymethyl hemicelluloses (CMH) on the physical properties of hand sheets of BKP pulp

Usually carboxymethyl polymers can be cooperatively used as wet-end additives with cationic polymers, which could bring the effect of the good cooperation (Zhang et al., 2008). Table 3 indicates the influence of the cooperation of cationic hemicelluloses and carboxymethyl hemicelluloses on the physical properties of hand sheets of BKP pulp. As seen obviously, the cooperation effect significantly improved the physical properties of hands sheets. When the dosages of cationic hemicelluloses with DS of 0.37 and carboxymethyl hemicelluloses with DS of 0.35 was 1.0% and 1.0%, respectively, breaking lengthen and tear index of hand sheets were maximum, and increased by 21.1% and 54.6% compared with that of control hand sheets without any additives. This indicated that cationic hemicelluloses had high compatibility with carboxymethyl hemicelluloses, suggesting that the cooperation of two agents had better effect on physical properties of hand sheets than agents used alone.

### 3.4. Influence of the reaction time of cationic hemicelluloses (YH) and pulp on the physical properties of hand sheets of BKP pulp

Based on the study above, different agents showed the different effect on the physical properties. The addition of cationic hemicelluloses and carboxymethyl hemicelluloses could make the sheet get better strength properties than modified hemicelluloses alone added. The time necessary for complete homogenization of cationic hemicelluloses and pulp fibers was also tested. There was significant difference in the physical properties when the solutions were kept under homogenization either for 5, 10, 15, or 20 min at the addition of cationic hemicelluloses with DS of 0.55 and dosage of 1.0% (w/w, cationic hemicelluloses/pulp fibre) and pulp consistence of 0.5% (w/w, pulp fibre/water) in Table 4. In comparison, we clearly observed that the physical properties of hand sheets increased with an increment of time. When the time reached for 15 min, breaking lengthen and burst index was maximum, and increase by 16.3% and 23.9% compared with that of the control sheets. This suggested that more static sorption force and hydrogen bonding occurred during enough duration after the mixture of polysaccharides.

### 3.5. SEM analysis

To increase the physical properties of sheets, it mainly needs to enhance the combined forces between the fiber layers. The combined forces of fiber layers could improve by the chemical and physical combination between strengthening agent and fibers (Zhu, 2004). Figs. 2 and 3 show the fiber intertexture with and without additives by SEM. As shown clearly, there are many interspaces among fibers on control sample without addition of additives (Fig. 2). The combined force mainly was hydrogen bonds. However, the combination between the fibers was very compact (Fig. 3), and there were polymers on surface of fibers, which resulted that fibers were conglutinated, which was explained that hemicellulosic derivatives adsorbed fibers so that enhanced combined force and resulted in the increment in the physical properties of hand sheets. Moreover, strengthening agent added enhanced the retention of fine fibers, and improved the uniformity of surface of hand sheets.

**Table 4**
The mixed time of cationic hemicelluloses and pulp on the physical properties of hand sheets of BKP pulp

| Time (min) | Breaking length (km) | Burst index (kPa m$^2$/g) | Tear index (mN m$^2$/g) |
|---|---|---|---|
| 5 | 5.22 | 7.6 | 9.19 |
| 10 | 5.39 | 7.28 | 8.56 |
| 15 | 5.64 | 8.1 | 9.76 |
| 20 | 5.23 | 7.48 | 10.21 |
| Control | 4.85 | 6.54 | 8.05 |

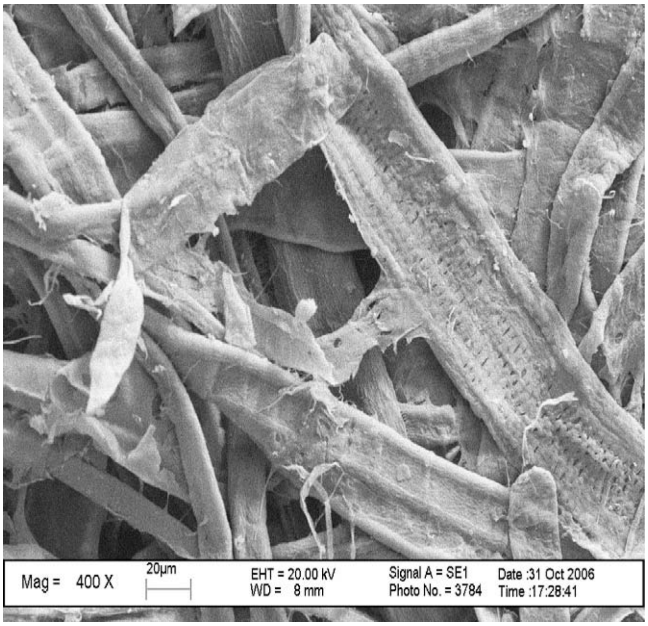

**Fig. 2.** SEM of hand sheets of BKP pulp without additives.

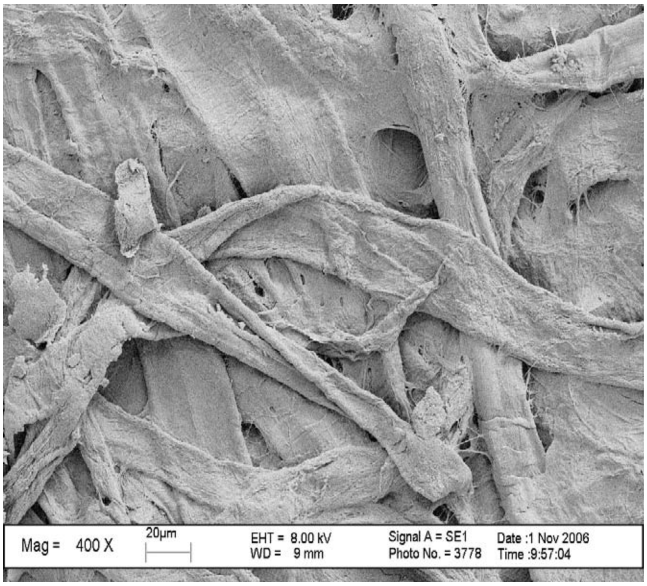

**Fig. 3.** SEM of hand sheets of BKP pulp with addition of cationic hemicelluloses.

### 3.6. FT-IR spectra

In this work FT-IR spectra of hand sheets with or without addition of additives is detected (Fig. 4). As shown obviously, there was the same profile in spectra a and b. but a little difference exited. The new band at 1457 cm$^{-1}$ in spectrum b is attributed to CH$_2$ bending mode and methyl groups of cationic hemicelluloses (Kacu-

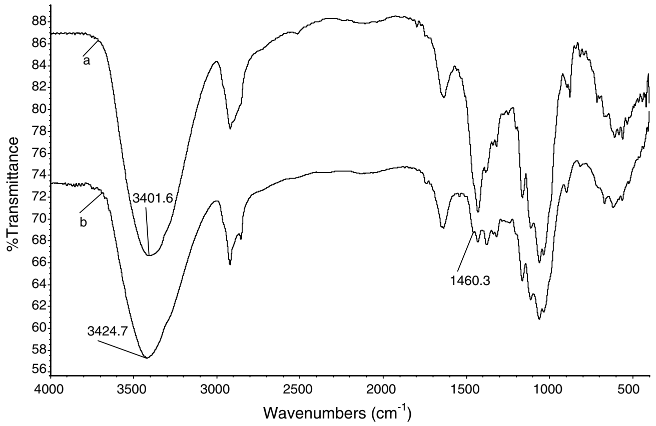

**Fig. 4.** FT-IR spectra of hand sheets without any additives (spectrum a) and with cationic hemicelluloses (spectrum b).

rakova, Ebringerova, Hirsch, & Hromadkova, 1994), which indicated that cationic hemicelluloses adhered to fibers in pulp. Moreover, the band at 3424.7 cm$^{-1}$ (–OH groups bending) in spectrum b became wider than that in spectrum a, and its peak shift to high wave, which meant that there were hydroxyl groups on the backbone of cationic hemicelluloses.

## 4. Conclusion

This study is included in a wide research project and it gives an insight in understanding that the hemicelluloses derivatives synthesized could improve the physical properties of BKP hand sheets. Cationic groups and carboxyl groups, which could have positive effect on physical properties of hand sheets, have been grafted onto hemicelluloses. The results of the applications indicated that cationic hemicelluloses and carboxymethyl hemicelluloses as wet-end additives could improve the physical properties of hand sheets. In particular, the cooperation of cationic hemicelluloses and carboxymethyl hemicelluloses could sharply enhance the physical properties of hand sheets than agent alone used. When the dosages of cationic hemicelluloses and carboxymethyl hemicelluloses was 1.0% and 1.0%, respectively, breaking lengthen and tear index of hand sheets increased by 21.1% and 54.6% compared with that of control hand sheets without any additives. Moreover, it was demonstrated that increasing the mixed time was more effective on paper strength. Therefore, the improvement observed on the physical properties of paper sheets showed that modified hemicelluloses in this work will have some promise as wet-end additives and might be used widely in papermaking. Even if the utilization of hemicellulosic derivatives will not cost less than starch or guar gum, it might represent an important strategy for sustainable use of hemicelluloses from agricultural residue.

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