Cationization of Corncob Holocellulose as a Paper Strengthening Agent

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In this paper, milled corncob powder was treated with sodium chlorite to remove lignin, and the resulting holocellulose was optionally modified with cationic agent. The derivative product was investigated using elemental analysis, Fourier transform infrared (FTIR) spectroscopy, and scanning electron microscopy (SEM). The influences of the dosage of the cationic agent, reaction temperature, particle size, dosage of paper strengthening agent, and pH value of the pulp on the paper physical properties were studied. The results indicated that cationic corncob holocellulose can improve the tensile index, burst index, and folding endurance of paper. When the dosage of cationic agent was 25% and the reaction temperature was set to 70 °C, the resulting tensile index, burst index, and folding endurance increased by 7.15%, 13.74%, and 55.95%, respectively, when compared with the control paper. The particle size of the raw material and the dosage of strengthening agent also greatly influenced the paper's properties. The SEM analysis showed that the combination of fibers improved the strength properties of the paper after adding the strengthening agent. These results provide a method for value-added use of corncob waste.

Keywords: Corncob; Cationization; Holocellulose; Fiber; Paper properties

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INTRODUCTION

Paper is made from fibrous raw materials, and its properties depend on the quality of the raw materials and the bonding strength between fibers (Page 1969; Clark 1978; Howard and Jowsey 1989). Because pulping can cause environmental pollution and there is an increase in environmental awareness, wood pulp production has been gradually reduced. However, with the increase in paper consumption, a serious supply and demand shortage ensues. Papermaking companies are increasing the use of secondary fibers and straw pulp, resulting in a significant reduction in paper properties (China Technical Association of Paper Industry 2013). One possible solution is to use paper strengthening agents to improve the bonding strength between fibers (Helle 1963; Hubbe 2006). Paper strengthening agents include polyacrylamide, chitosan, and nanocellulose. Other strength additives, such as natural polysaccharides, starch, and cellulose do not enhance paper properties well until a chemical modification is made (Fatehi *et al.* 2009; Ren *et al.* 2009; Bai *et al.* 2012; Khorasani *et al.* 2013; Vega *et al.* 2013; Wang and Song 2013; Deutschle *et al.* 2014; Song and Hubbe 2014a,b; Yang *et al.* 2014; He *et al.* 2015).

There are several methods used in polysaccharide modification, such as oxidation, carboxymethylation, cationization, sulfonation, and esterification. Cationization is a method that uses a cationic agent to modify polysaccharides, supplying the surface of fibers with a positive charge through the addition of quaternary ammonium groups. With this modification, the modified fibers are attracted to the negative charge of the pulp fiber surface, which can be integrated closely between fibers and improve the physical properties of the paper. Cationic starch and polysaccharides are widely used in the industry; however, few studies report on the cationization of holocellulose. Corn is a major food source and is largely cultivated in China, producing a copious amounts of agricultural waste in the form of corncobs. Corncob is one of the waste products from the corn production process. Corncobs are known to contain a high quantity of holocellulose (65% to 76%), and they are primarily used in the production of furfural or disposed of in landfills. In this work, corncob was used as a raw material and treated with sodium chlorite for the removal of lignin. The resulting holocellulose was cationized with 3-chloro-2-hydroxypropyl-trimethyl-ammonium chloride (cationic agent). The influences of the cationic agent, reaction temperature, corncob particle size, dosage of strengthening agent, and pH value of pulp on the paper properties were studied. The results will provide a method for value-added use of corncob.

EXPERIMENTAL

Materials

Corncob was obtained from a village of Liaocheng, Shandong province, China. Each cob was split into pieces with dimensions of 20 mm × 15 mm × 10 mm. Then, the pieces were ground into a fine powder of 80-, 100-, 150-, and 200-mesh sizes. Aspen kraft pulp was provided by the Yueyang paper group from Hunan province. The pulping conditions included a sulfidity of 21.8%, active alkali content of 15.2 g/L, maximum temperature of 168 °C, heating rate of 1.8 to 2 h, yield of 45%, and beating degree of 40 °SR.

Methods

Preparation of holocellulose

Four grams of corncob powder (dry matter basis) were put into a 250-mL conical flask. Next, 130 mL of distilled water, 20 drops of glacial acetic acid, and 1.0 g of sodium chlorite were added to the conical flask and mixed thoroughly. A small conical flask (25 mL) was buckled to the mouth of the larger conical flask, and the 250-mL conical flask was submerged in a water bath at 75 °C for 1 h. The flask assembly was shaken often over the course of the reaction process. Then, another 20 drops of glacial acetic acid and 1.0 g of sodium chlorite were added to the reaction flask, and the mixture was reacted for 1 h at 75 °C. The reaction was stopped by resting the mixture in an ice bath. The mixture was filtered using filter paper, washed four times with ice water, and the corncob holocellulose was collected.

Cationization of holocellulose

Twelve grams of corncob holocellulose were put into a 100-mL conical flask. Then, 0.13 g of NaOH was added to the flask and stirred at room temperature for 10 min. Next, 15% to 45% of 3-chloro-2-hydroxypropyl trimethyl ammonium chloride (cationic agent) based on dry corncob holocellulose weight was added to the reaction, stirred for 10 min, and placed in the oven at 70 °C for 4 h. When the reaction had ended, the mixture was filtered using filter paper, washed several times with water, and the cationic holocellulose was collected.

Papermaking and paper physical properties

Standard kraft pulp handsheets (60 g/m^2) were prepared in accordance with the China GB standard methods (GB/T 451.2-2002 2002). The papermaking conditions were as follows: 1.0% cationic corncob holocellulose, 0.5% $Al_2(SO_4)_3$, and the pH set to 5, 6, 7, or 8, respectively. A portion of the paper samples was immersed in water at room temperature for 24 h, and then it was dispersed and recycled following TAPPI methods. The physical properties of the resultant handsheets were tested after spending 24 h under the conditions of constant temperature and humidity, in accordance with the China GB standard method (GB/T 454-2002 (2002); GB/T 12914-2008 (2008); GB/T 457-2008 (2008)).

Elemental analysis

The elemental analysis was carried out using a Vario EL III Elemental Analyzer (Elementar, Germany). The carbon, hydrogen, and nitrogen contents were tested. The oxygen content was calculated using the total content of all the elements, subtracting that of carbon, hydrogen, and nitrogen.

Fourier transform infrared (FTIR) spectroscopy

The cationic corncob holocellulose, the corncob holocellulose, and approximately 0.12 g of KBr were first oven-dried and then ground into powder to obtain a sample suitable for FTIR analysis. The absorbance between 4000 and 400 cm^{-1} was studied, using a Spectrum-100D instrument (PerkinElmer, USA).

Scanning electron microscopy analysis

An S-3000N SEM device (Hitachi Ltd., Japan) was used for the observation of handsheet surfaces, cross-sections of handsheets (after cutting with a paper cutter), and paper specimens observed after breakage during tensile testing. An EM ACE200 Ion sputter device (Leica Microsystems, Germany) was used for gold sputtering to avoid charging effects.

RESULTS AND DISCUSSION

Elemental Analysis

Table 1. Elemental Analysis of Corncob Holocellulose and Cationic Corncob Holocellulose

	N (%)	C (%)	H (%)	O (%)
Corncob holocellulose	0.086	42.46	6.149	51.31
Cationic corncob holocellulose (dosage of cationic agent 15%)	0.165	43.03	6.277	50.53
Cationic corncob holocellulose (dosage of cationic agent 35%)	0.219	42.47	6.270	51.04

Note: The standard deviation was < 4%.

The elemental contents of the corncob holocellulose and cationic corncob holocellulose are presented in Table 1. The carbon and hydrogen contents increased initially and then decreased. The oxygen contents decreased initially and then increased. The nitrogen contents increased after cationization, indicating that the nitrogen groups were successfully linked to the corncob holocellulose post-reaction.

FTIR Analysis

Figure 1 shows the FTIR spectra of the cationic and unmodified corncob holocelluloses. The 3435 cm^{-1} peak represents hydroxyl groups, the 2925 cm^{-1} peak corresponds to methylene groups, and the 1633 cm^{-1} peak represents carbonyl (C=O) groups.

According to the literature, C-N stretching in a quaternary system with methyl groups should give rise to a small peak at 1491 cm^{-1} (Zhang *et al.* 2007; Kahlil-Abad *et al.* 2009; Pažitný *et al.* 2011). As shown in Fig. 1, the peak at 1491 cm^{-1} for cationic corncob holocellulose was small, which indicates that the cationic reaction was weak.

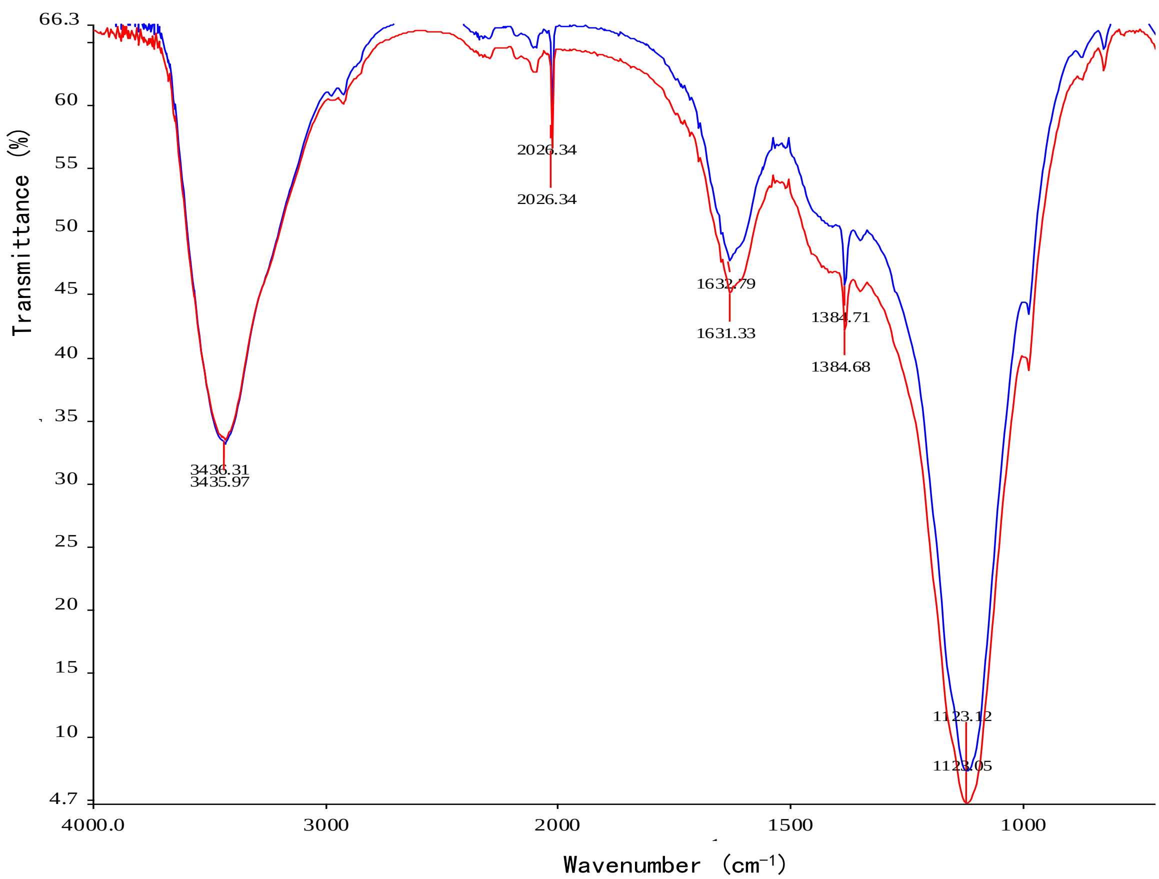

Fig. 1. FTIR spectra of corncob holocellulose (blue) and cationic corncob holocellulose (red)

Table 2. Effects of Dosage of Cationic Agent on Paper Properties

Dosage of cationic agent (%)	Density (g/cm^3)	Tensile index (N·m/g)	Tear index ($mN·m^2/g$)	Burst index ($kPa·m^2/g$)	Folding endurance (double folds)
Control	0.58	93.7	12.4	6.84	420
15	0.54	98.6	12.0	7.70	565
25	0.54	100.4	11.6	7.78	655
35	0.52	97.9	12.0	7.65	579
45	0.58	95.7	12.1	7.46	545

Note: Cationic reaction temperature was 70 °C and the corncob particle size was < 80 mesh. The control sample was prepared without chemical additives, and the other samples were prepared with 1.0% cationic corncob holocellulose and 0.5% $Al_2(SO_4)_3$•$18H_2O$ (on a dry matter basis). After the addition of $Al_2(SO_4)_3$, the pH was adjusted to 5. The standard deviation was < 5%.

Effects of Dosage of Cationic Agent on Paper Properties

The effects of the dosage of cationic agent on the density, tensile strength, tear index, burst index, and folding endurance, are shown in Table 2. Compared with the control sample, the tensile index, burst index, and folding endurance of the handsheets notably increased with the addition of 1.0% cationic corncob holocellulose. As the dosage of the cationic agent increased, the tensile index, burst index, and folding endurance increased initially and then decreased thereafter. This was opposite for the tear index, which decreased initially and then increased thereafter. When the dosage of cationic agent was increased to 25%, the effect of cationic corncob holocellulose on the paper properties was optimal. When compared with the control sample, the tensile index, burst index, and folding endurance increased by 7.15%, 13.74%, and 55.95%, respectively.

Effects of Cationic Reaction Temperature on Paper Properties

Table 3. Effects of Cationic Reaction Temperature on Paper Properties

Cationic reaction temperature (°C)	Density (g/cm^3)	Tensile index (N·m/g)	Tear index ($mN·m^2/g$)	Burst index ($kPa·m^2/g$)	Folding endurance (double folds)
Control	0.58	93.7	12.4	6.84	420
50	0.55	97.9	11.8	7.38	587
60	0.56	98.1	11.3	7.47	688
70	0.54	100.4	11.6	7.78	655
80	0.56	95.4	12.1	7.57	546

Note: The cationic agent consumption was 25% and the corncob particle size was < 80 mesh. The control sample was prepared without chemical additives, and the other samples were prepared with 1.0% cationic corncob holocellulose and 0.5% $Al_2(SO_4)_3$•$18H_2O$ (on a dry fiber basis). After the addition of $Al_2(SO_4)_3$, the pH was adjusted to 5. The standard deviation was < 5%.

Table 3 shows the effect of the cationic reaction temperature on the density, tensile index, tear index, burst index, and folding endurance. The tensile index, burst index, and folding endurance of paper increased with the addition of 1.0% cationic corncob holocellulose. This occurred *via* an increase in the inter-fiber bonding strength when cationic corncob holocellulose was added. The tensile index, burst index, and folding endurance increased initially, and then decreased with increasing reaction temperature. However, the tearing index first decreased and then increased slightly. The tensile index, burst index, and folding endurance increased by 7.15%, 13.74%, and 55.95%, respectively, compared with the control paper, with the addition of 1.0% cationic corncob holocellulose, at a reaction temperature of 70 °C.

Effects of Corncob Particle Size on Paper Properties

The effects of corncob particle size on the density, tensile index, tear index, burst index, and folding endurance are shown in Table 4. The density and the tear index increased gradually as the corncob particle size was reduced; however, the tensile index, burst index, and folding endurance first increased and then decreased as the particle size increased. However, when the particle size was smaller than 150 mesh, the tensile index, burst index, and folding endurance decreased. The tensile index, burst index, and folding endurance of paper increased by 9.39%, 16.52%, and 85.24%, respectively, compared with the control when the corncob particle size was 150 mesh. These results can be explained by the nanomaterial effect, which increases the bonding strength due to an increase in the specific surface area of the fibers during the process of cationization.

Table 4. Effects of Corncob Particle Size on Paper Properties

Corncob particle size (mesh)	Density (g/cm^3)	Tensile index (N·m/g)	Tear index ($mN·m^2/g$)	Burst index ($kPa·m^2/g$)	Folding endurance (double folds)
Control	0.58	93.7	12.4	6.84	420
80	0.54	100.4	11.6	7.78	655
100	0.56	101.6	11.7	7.89	732
150	0.59	102.5	12.2	7.97	778
200	0.60	97.7	12.2	7.78	658

Note: Cationic agent consumption was 25% and the cationic reaction temperature was 70 °C. The control sample was prepared without chemical additives, and the other samples were prepared with 1.0% cationic corncob holocellulose in addition to 0.5% $Al_2(SO_4)_3$•$18H_2O$ (on a dry fiber basis). After the addition of $Al_2(SO_4)_3$, the pH was adjusted to 5. The standard deviation was < 5%.

Effects of Dosage of Cationic Corncob Holocellulose on Paper Properties

The influences of dosage of cationic corncob holocellulose on the density, tensile index, tear index, burst index, and folding endurance are shown in Table 5. The tensile index, burst index, and folding endurance of paper increased initially and then decreased with increasing dosage of cationic corncob holocellulose, while the tear index decreased initially and then increased thereafter. These results can be explained by an increase in the inter-fiber bonding strength with increasing dosage of cationic corncob holocellulose. The tensile index, burst index, and folding endurance of the paper increased by 7.15%, 13.74%, and 55.95%, respectively, compared with the control paper, when the dosage of cationic corncob holocellulose was 1.0%. Paper strengthening agents can have a favorable effect on paper properties at a specific dosage.

Table 5. Effects of Dosage of Cationic Corncob Holocellulose on Paper Properties

Dosage of cationic corncob holocellulose (%)	Density (g/cm^3)	Tensile index (N·m/g)	Tear index ($mN·m^2/g$)	Burst index ($kPa·m^2/g$)	Folding endurance (double folds)
Control	0.58	93.7	12.4	6.84	420
0.5	0.58	95.7	12.0	7.04	502
1.0	0.54	100.4	11.6	7.78	655
1.5	0.55	96.6	12.6	7.50	588
2.0	0.53	94.3	13.0	7.34	536

Note: Cationic agent consumption was 25%, the cationic reaction temperature was 70 °C, and the corncob particle size was < 80 mesh. The control sample was prepared without chemical additives, and the other samples were prepared with the above dosage of cationic corncob holocellulose and 0.5% $Al_2(SO_4)_3$•$18H_2O$ (on a dry fiber basis). After the addition of $Al_2(SO_4)_3$, the pH was adjusted to 5. The standard deviation was < 5%.

Effects of pH on Paper Properties

Table 6 shows the effect of the pH on the density, tensile index, tear index, burst index, and folding endurance. The tensile index, burst index, and folding endurance of the paper increased initially and then decreased as the pH of papermaking increased, while the tear index increased initially and then decreased as the pH increased.

Table 6. Effects of pH on Paper Properties

pH	Density (g/cm^3)	Tensile index (N·m/g)	Tear index ($mN·m^2/g$)	Burst index ($kPa·m^2/g$)	Folding endurance (double folds)
Control	0.58	93.7	12.4	6.84	420
5	0.54	100.4	11.6	7.78	655
6	0.56	97.1	12.0	7.73	638
7	0.55	95.7	12.0	7.66	612
8	0.55	94.4	11.7	7.43	582

Note: Cationic agent consumption was 25%, the cationic reaction temperature was 70 °C, and the corncob particle size was < 80 mesh. The control sample was prepared without chemical additives and using tap water, and the other samples were prepared with 1.0% cationic corncob holocellulose and 0.5% $Al_2(SO_4)_3$•$18H_2O$ (on a dry fiber basis). After the addition of $Al_2(SO_4)_3$, the pH was adjusted to 5, 6, 7, or 8, respectively. The standard deviation was < 5%.

The paper strength properties were optimal at a pH of 5, where the tensile index, burst index, and folding endurance increased by 7.15%, 13.74%, and 55.95%, respectively, compared with the control paper (without cationic corncob holocellulose). The reason may be explained by Al^{3+} and related cationic species of aluminum; these exist in the water at pH of 5. Although the corncob holocellulose is cationic material, the cationic charge is weak. The cationic species of aluminum can help the corncob holocellulose to contact with the surfaces of fines and fibers. Thus the bond strength between the fibers will be improved.

Effects of pH on Recycled Paper Properties

Table 7 shows the effects of pH on the density, tensile index, tear index, burst index, and folding endurance of recycled paper. The tensile index, burst index, and folding endurance of recycled paper decreased as the pH increased; however, the tear index mirrored the increase in pH. The paper strength properties were optimal at a pH of 5. The tensile index, burst index, and folding endurance increased by 9.19%, 0.45%, and 30.40%, respectively, compared with the control paper without cationic corncob holocellulose.

Table 7. Effects of pH on Recycled Paper Properties

pH	Density (g/cm^3)	Tensile index (N·m/g)	Tear index ($mN·m^2/g$)	Burst index ($kPa·m^2/g$)	Folding endurance (double folds)
Control	0.51	57.7	19.9	4.45	227
5	0.51	63.0	17.3	4.47	296
6	0.51	61.2	17.5	7.43	281
7	0.51	60.4	17.7	7.40	250
8	0.51	59.4	18.1	7.37	233

Note: Cationic agent consumption was 25%, the cationic reaction temperature was 70 °C, and the corncob particle size was < 80 mesh. The control sample was prepared without chemical additives, and the other samples were prepared with 1.0% cationic corncob holocellulose and 0.5% $Al_2(SO_4)_3•18H_2O$ (on a dry fiber basis). After the addition of $Al_2(SO_4)_3$, the pH was adjusted to 5, 6, 7, or 8, , respectively. The paper samples were immersed in a water bath at room temperature for 24 h and then disintegrated. The recycled handsheets were made without any chemical additives. The standard deviation was < 5%.

SEM Analysis

Figure 2 shows SEM images of the paper samples, with or without the addition of cationic corncob holocellulose. As shown in Fig. 2a, most fibers on the edges of the control paper were intact when the paper was broken during tensile testing, while some of the edge fibers in Fig. 2d were fractured in the case of addition of 1.0% cationic corncob holocellulose. These results indicated that the inter-fiber bonding strength increased with the addition of cationic corncob holocellulose, which caused some of the fibers to fracture rather than pull away during inter-fiber bonding.

As shown in Figs. 2b and e, the fibers visible in the sliced control paper had a loose appearance, while the fibers of the sliced paper that had been prepared with cationic corncob holocellulose were more compact and thinner. This indicates that the cationic corncob holocellulose integrates with the fiber layers more completely. Figures 2c and f demonstrate that the control paper contained more pores than the paper prepared with cationic corncob holocellulose. The reduction in pore number of the paper containing cationic corncob holocellulose means that the agent may have filled in the pores of the fibers, so that the fibers could be integrated more tightly.

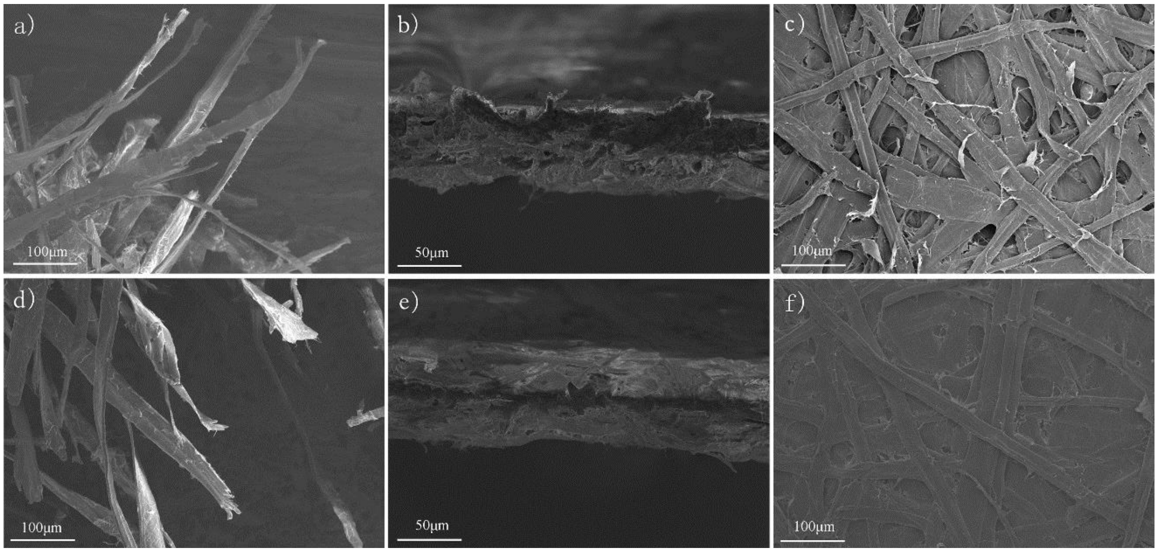

Fig. 2. SEM microscopy images of control paper and paper with 1.0% cationic corncob holocellulose (a, d are fracture images, a: original, d: with CCH. b, e are section images, b: original, e: with CCH. c, f are surface images, c: original, f: with CCH) f)

CONCLUSIONS

1. The tensile index, burst index, and folding endurance of paper can be improved with the addition of 1.0% cationic corncob holocellulose, resulting in an increase in the bonding strength of the fibers. As the dosage of cationic agent or the cationic reaction temperature increased, the above-mentioned indexes tended to first increase and then decrease. Compared with the control paper, the tensile index, burst index, and folding endurance of paper increased by 7.15%, 13.74%, and 55.95%, respectively, when the dosage of cationic agent was 25% and the cationic reaction temperature was 70 °C.
2. Cationic corncob holocellulose can improve paper's physical properties. The tensile index, burst index, and folding endurance increased by 9.19%, 0.45%, and 30.40%, respectively, when 1.0% cationic corncob holocellulose was used under the conditions of 25% cationic agent, 70 °C, and pH of 5.
3. The corncob particle size exhibited the greatest influence on the paper properties of tensile index, burst index, and folding endurance.
4. The SEM micrographs showed that the cationic corncob holocellulose could be integrated with the fibers more completely, improving the integration of fibers in the handsheets.

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