

Effects of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ Treatment on the Filter Paper Performance

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Abstract: Bleached softwood kraft pulp (BSKP) fibers were pretreated in a 5 mol/L NaOH aqueous solution at 35°C for 90 min. The $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ was used to treat the pretreated BSKP fibers, filter papers were then prepared. In the course of processing, the effects of various molar ratios of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ on the filter paper performance were evaluated. SEM-EDX was conducted to analyze the physical configuration of the filter papers. Moreover, FT-IR and XPS were performed to further characterize the BSKP, the pretreated BSKP and the filter paper fibers. The results indicated that the fibers treated by $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ were swollen and exhibited slight fibrillation, leading to a looser fiber structure and therefore, enhanced filter paper performance. Air permeability increased from 161.7 L/(m²·s) to 1450.4 L/(m²·s) for the filter papers when the molar ratio of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ was 1:14:1. The optimum molar ratio of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ was found to be 1:14:1, and no significant effect of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ on fibers' functional groups was detected. These results showed that filtration performance of filter papers was improved when treated with the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ solution.

Keywords: BSKP fiber; $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$; filtration performance; filter papers

1 Introduction

Filter paper is a filtering material which can be used to intercept and separate impurities in liquids or gases. It is an essential material in the medical, auto industry^[1], construction, and food industries, as well as in scientific research^[2-4], etc. Filtration performance of filter paper depends mainly upon its structural characteristics, such as air permeability (low air resistance), pore size, and tightness (bulkiness)^[5]. Generally, cellulose fibers are the basic materials used for production of various paper-based products^[6], and modifying fibers allows considerable cost reduction and value-added



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use of pulp fibers by the pulp and paper industry^[7].

Cellulose is the most abundant renewable and biodegradable material available in nature^[8]. Cellulose is a linear polymer composed of β -(1,4)-linked *D*-glucopyranosyl units in C1 chair conformation. Hydroxyl groups in the glucopyranose rings can form abundant inter-molecular, intra-molecular, interchain, and intersheet hydrogen bond networks^[9-13]. The existence of hydrogen bonds ensures the self-assembly, crystallinity, and stability of cellulose. Therefore, cellulose cannot be melted, and cannot be dissolved in common solvents, water or typical organic solvents^[8]. In the conventional filter paper industry, mercerization is used to enhance filter paper performance^[14-15]. Hemicellulose and resin in the cellulose crystal range are easily dissolved and removed during mercerization, leading to fiber swelling, which facilitates production of high-permeability filter paper^[16-17]. The degree of swelling is finite, therefore, limits the improvement of filter paper performance.

Intensive studies have been conducted on the swelling, reaction, and dissolution of cellulose in the ZnCl_2 solution^[18-20]. The dissolution ability of the concentrated salt solution toward cellulose was discussed by Lv^[21], while Letters also researched cellulose swelling and dissolution in ZnCl_2 solution, and found that cellulose would swell and dissolve in 63% (w/w) and 55% (w/w) ZnCl_2 solutions, respectively^[22]. Three aqueous solution systems including alkaline aqueous solution, inorganic acid solution and inorganic salt solution have been used to swell or dissolve cellulose^[23]. Xu et al also explored cellulose dissolution and fiber formation in ZnCl_2 solution, and noted that regeneration of cellulose could be facilitated by precipitation of a zinc-cellulose complex with alcohol^[24]. Xiong et al discussed the dissolving ability of ZnCl_2 solution toward cellulose with different degrees of polymerization^[25], and found that the ZnCl_2 solution could not dissolve cellulose unless the salt mass fraction was >65.0%, and that the intramolecular hydrogen bond of regenerated cellulose was weakened after dissolution. Ethanol could accelerate inorganic

salt ion penetrating into fibers due to its stronger penetrability than water, while the penetrability of inorganic salt has been shown to be related to surface tension and the higher the surface tension, the lesser the penetrability of the solution. Surface tension decreases with the addition of ethanol, which is beneficial to improving penetrability of the solution in order to assist the swelling and dissolving of cellulose. Little is known, however, about the effects of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treatment on filter paper performance.

In this study, the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ was used to treat BSKP fibers which had been pretreated in a 5 mol/L NaOH aqueous solution at 35°C for 90 min. The molar ratio of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ was varied to determine its effects on filter paper performance. The BSKP fibers, mercerized BSKP fibers and the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fibers were characterized using scanning electron microscopy-energy dispersive spectrometer (SEM-EDX), Fourier transform-infrared spectroscopy (FT-IR), and X-ray photoelectron spectroscopy (XPS). The air permeability, tightness, and the maximum and average pore sizes of filter papers were determined. The mercerized BSKP fibers were fully swollen, and exhibited slight fibrillation without clogging after treating with $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$. Filter paper prepared from the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fibers not only maintained high air permeability but also exhibited fine filtration performance. The mercerized BSKP fibers could also be used to prepare advanced filtration, separation, and adsorption materials.

2 Materials and methods

2.1 Materials

Bleached softwood kraft pulp (BSKP) fibers, imported from Canada, were provided by Hengfeng Paper Co., Ltd., (Mudanjiang, China). Analytical reagent grade NaOH, ZnCl, ethanol, and isopropanol were purchased from Tianjin Zhiyuan Chemical Reagent Co., Ltd. (China), and were used as received without further purification.

2.2 Fiber treatment

BSKP board were soaked in water for 12 h, then torn

into grain-sized pieces with approximately 1-cm length. The moisture was then measured, after the pieces were stored in plastic sealing bags in a freezer at 5°C for 24 h. 50 g of oven-dried BSKP fibers (obtained from the above process), and 500 g of 5 mol/L NaOH solution were added to a 1000-mL beaker and the mixture was then heated to 35°C and stirred at around 500 r/min for 90 min. After this process, the fibers were firstly washed with tap water to remove excess NaOH, and then washed with distilled water until the filtrate reached pH value of 7. In order to avoid moisture changes and the impact of excessive ambient temperature, the resulting mercerized BSKP fibers which were agglomerated after treating with NaOH were again separated into grain-size pieces, sealed in plastic bags and kept at 5°C until required for further use.

A parallel series of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ solutions with different $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ molar ratio of 1:10:1, 1:12:1, 1:14:1, 1:16:1, 1:18:1 and 1:20:1 were prepared. 10 g of mercerized BSKP fibers and 400 g of the selected $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ were preheated in a beaker to 65°C. The mixture was then continuously stirred using a screw rotating at 200 r/min for 25 min. The obtained treated fibers were washed with tap water several times and then stored in a freezer at 5°C.

2.3 Hand-sheet preparation

A ZCX-200 hand sheet mold (China) was employed to prepare hand-sheet with a target basis weight of 108 g/m². The BSKP fibers, mercerized BSKP fibers and the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fibers were separately dispersed in water with a concentration of 0.03 wt% and the resultant wet filter paper sheets were dried at 105°C for 20 min without pressing (to preserve high permeability) in an electro-thermostatic blast oven. The filter paper sheets were then stored at constant temperature and humidity until needed for testing.

2.4 Detection and analysis of filter paper performance

2.4.1 Air permeability

Air permeability measurements were performed with a YG461E Numerical Air Permeability Instrument,

supplied by Ningbo Textile Instrument Factory (China). The air permeability is to measure the amount of air passing through the filter paper sheet or paper board per unit area (50 mm diameter) at a certain pressure gradient, ΔP (20.0 mm water column) as mandated by ISO 9237:1995 (E) Standard^[26]. For many filter products, air permeability is an important feature as it helps to evaluate the filtration performance of industrial filter materials^[27].

2.4.2 Pore size

An MP-10K Hole Size Reflect Scope Reflector (Xinxiang Dongfeng Filtration Technology Co., Ltd., China) was employed to measure maximum and average pore sizes of the filter papers using the bubble method, with isopropanol as the test medium—in compliance with ISO 4003. Filter paper pore sizes were calculated using Eq.(1).

$$D=1.0 \times 10^{-6} \times 4S/P \quad (1)$$

Where D indicates the pore size (μm), S represents the test medium surface tension (taken as 21.35×10^{-3} N/m), and P denotes pressure (Pa).

2.4.3 Tightness

In compliance with National Standards GB/T451.2—2002, and GB/T451.3—2002, a ZUS-4 paper thickness tester supplied by Changchun Yueming Mini Testing Machine Co., Ltd., (China) was used to measure filter paper thickness, while the tightness of the filter paper was calculated using Eq.(2).

$$G=q/(d_a \times 1000) \quad (2)$$

Where G is the tightness (g/cm³) of the filter paper, d_a is thickness (mm), and q is the basis weight (g/m²).

2.5 Characterization

An FEI Quante-200 (USA) SEM was used to observe and analyze fiber surfaces and cross-sections morphology. FT-IR (Nicolet 6700, Thermo Fisher Scientific, USA) was employed to determine whether or not the treated fibers had generated new functional groups. For each sample, the spectrum was measured at a resolution of 4 cm⁻¹ with an accumulation of 40 scans in the range of 700~4000 cm⁻¹. To further investigate

the elemental composition, the treated fibers were characterized using XPS (K-Alpha, USA).

2.6 Statistical analysis

Results were analyzed using Excel 2010 and SPSS software. Analysis of variance (ANOVA) was carried out, and Duncan's multiple range tests were used to compute significant differences ($p < 0.05$) among filter paper samples' data using mean $\pm$ SD (for thickness, $n=10$; for air permeability and pore size, $n=6$). Means of pore sizes for different series samples (Fig.1) are significantly different ($p < 0.05$).

3 Results and discussion

3.1 Effect of mercerization on filter paper performance

The properties of BSKP filter papers and mercerized BSKP filter papers (pretreated by NaOH) are listed in Table 1. The air permeability of mercerized BSKP filter papers was 671.80% greater than the BSKP filter papers, the maximum pore size and the average pore size of mercerized BSKP filter papers increased by 44.8%, and 57.4%, respectively, compared with the BSKP filter paper. The results may be due to the pulp fibers becoming partially swollen and rounded in the mercerized BSKP filter papers, resulting in a decrease in the tightness and a corresponding increase in air permeability after mercerization.

Table 1 Properties of BSKP filter papers and mercerized BSKP filter papers

Pulp fiber type	Air permeability $/(L \cdot m^{-2} \cdot s^{-1})$	Tightness $/(g \cdot cm^{-3})$	Maximum pore size $/\mu m$	Average pore size $/\mu m$
BSKP	161.7	0.245	37.5	31.2
Mercerized BSKP	1248.0	0.170	54.3	49.1

3.2 Effect of various molar ratio of $ZnCl_2/H_2O/C_2H_5OH$ on filter paper performance

The effects of various molar ratio of $ZnCl_2/H_2O/C_2H_5OH$ on filter paper properties are shown in Fig.1. As can be seen from Fig.1(a), with increased concentration of $ZnCl_2$ in each $ZnCl_2/H_2O/C_2H_5OH$ solution, air permeability of the filter papers initially

increased, and then showed a decreasing trend. The maximum air permeability was $1450.4 L/(m^2 \cdot s)$ when the molar ratio of $ZnCl_2/H_2O/C_2H_5OH$ solution was 1:14:1, which was much higher than the previously reported result^[28-29], in the same time, the tightness reached $0.159 g/cm^3$ which was much lower than that of the mercerized BSKP fibers of $0.170 g/cm^3$. The maximum pore size of $58.0 \mu m$ and an average pore size of $55.4 \mu m$ achieved with the molar ratio of $ZnCl_2/H_2O/C_2H_5OH$ was 1:14:1. When the mercerized BSKP fibers were treated in the $ZnCl_2/H_2O/C_2H_5OH$ with different molar ratio, they became further with increased $ZnCl_2$ concentration in $ZnCl_2/H_2O/C_2H_5OH$. The degree of fiber swelling gradually increased, therefore, the fibers become more rounded resulting in increased pore sizes and reduced tightness. At the same time, Zn^{2+} reacted with the hydroxyl groups on the cellulose fibers, destroying the hydroxyl groups of the cellulose fiber chains, resulting in improved air permeability of filter papers. However, when the molar ratio of $ZnCl_2/$

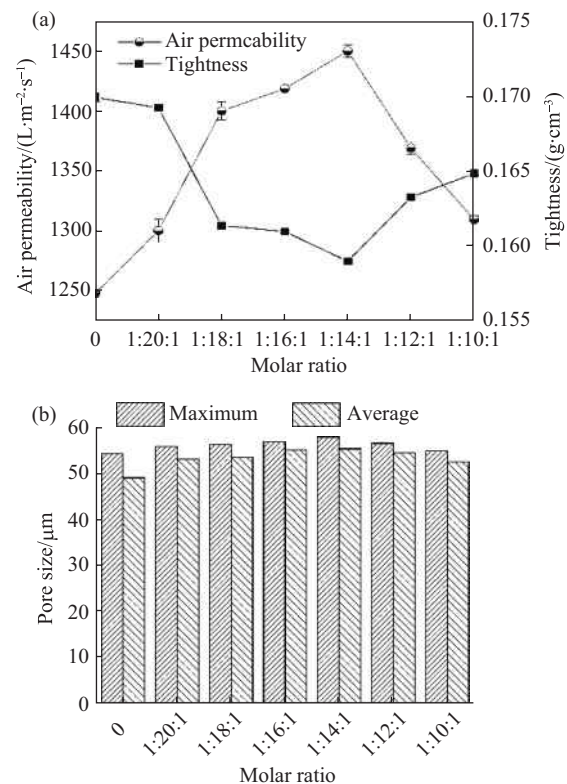


Fig.1 (a) Air permeability and tightness of filter papers and (b) maximum and average pore sizes of filter papers against different molar ratio of $ZnCl_2/H_2O/C_2H_5OH$

$\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ was higher than 1:14:1, it may be that the dissolution of the pulp fibers in the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ was stronger than its swelling effect, resulting in the structure of pulp fibers being broken, which in turn decreased filter paper performance.

3.3 Fiber morphological and quality analysis

3.3.1 SEM-EDX analysis

SEM images of different fibers are shown in Fig.2. Comparing images Fig.2(a)~Fig.2(c), it can be seen that the BSKP fibers were ribbon-like and the surfaces of the fibers were very smooth, while the mercerized BSKP fibers were more swollen, and the surfaces were relatively smooth compared with the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fibers which had a rough surface, round and loose structure.

Fig.2(d)~Fig.2(f) show the cross-sections of different fibers. The BSKP fibers were flat with thin cell walls, while the mercerized BSKP fibers were tumid with thick cell walls, and the cell walls of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fibers were fibrillated and became looser. The SEM images show that the diameter of

the mercerized BSKP fiber was larger than that of the BSKP fiber, while the diameter of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fiber was larger than that of the mercerized BSKP fiber. The results indicated that the treatment of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ decreased the tightness and therefore, enhanced filtration performance of the filter papers.

3.3.2 FT-IR analysis

The FT-IR spectra of BSKP fibers, mercerized BSKP fibers, and $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fibers are shown in Fig.3, and the peaks were assigned by reference to the literature [30-31]. In Fig.3, the peak observed at $\sim 3300\text{ cm}^{-1}$ was attributed to O—H stretching, while the peak at $\sim 2890\text{ cm}^{-1}$ resulted from the stretching of C—H and —CH₂— bonds. The peak at $\sim 1320\text{ cm}^{-1}$ was assigned to the stretching vibration of C—C and C—O bonds, while peaks at $\sim 1010\text{ cm}^{-1}$ and $\sim 889\text{ cm}^{-1}$ corresponded with C—OH and C—O—C bond stretching in β -(1,4)-glycosidic linkages, respectively. There were no new peaks in all the

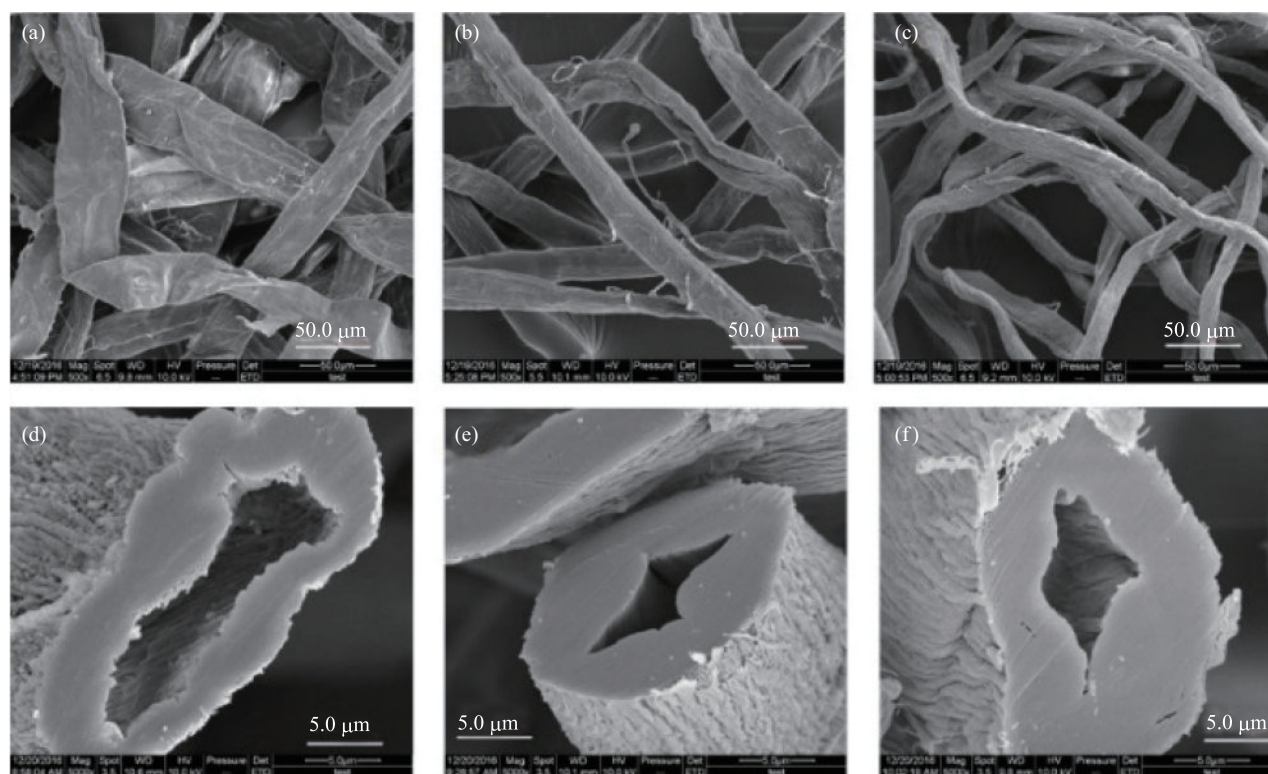


Fig.2 SEM images of fiber surfaces and cross-sections: (a) and (d) BSKP fibers; (b) and (e) mercerized BSKP fibers; (c) and (f) $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fibers

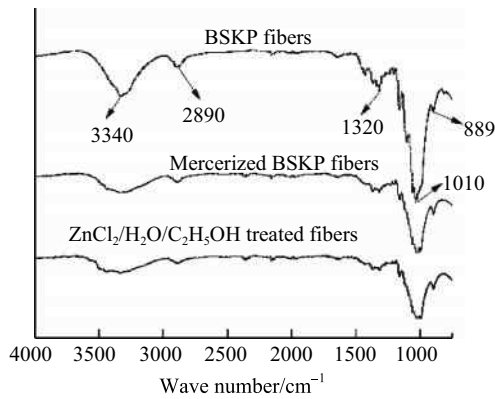


Fig.3 FT-IR spectra of different pulp fibers

three spectra and the three samples exhibited similar spectra, indicating that the fiber structures had not been changed by the mercerization and $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treatment.

There were, however, some differences in peak wave numbers of the BSKP, mercerized BSKP and the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fibers. The intensity of the $-\text{OH}$ groups of the three fibers decreased (BSKP fibers > mercerized BSKP fibers > $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fibers), and the red shift phenomenon occurred, indicating that the hydrogen bonds were partly broken^[32-33]. It is quite conceivable that some hydrogen bonds were partially destroyed, causing fibers to become looser, as a result of the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$

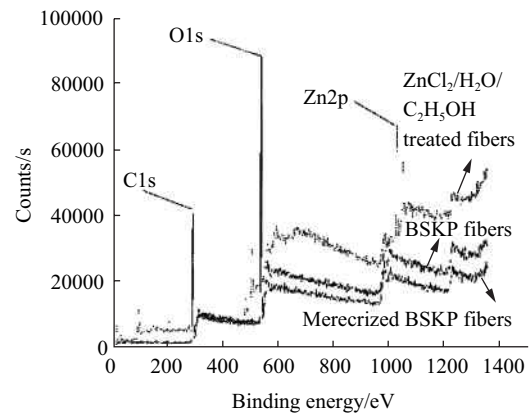


Fig.4 XPS spectra of different pulp fibers

solution treatment.

3.3.3 XPS analysis

The XPS spectra of BSKP fiber, mercerized BSKP fiber, and $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fiber are shown in Fig.4. The presence of Zn2p in the XPS spectrum of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ treated fiber was attributed to Zn^{2+} forming a chelate with hydroxyl groups in the cellulose, leading to a decreased number of hydrogen bonds and weakened binding strength and the mechanism for this has been illustrated in Fig.5^[25,34-37]. This effect is beneficial to improving filtration performance such as air permeability and tightness of filter papers.

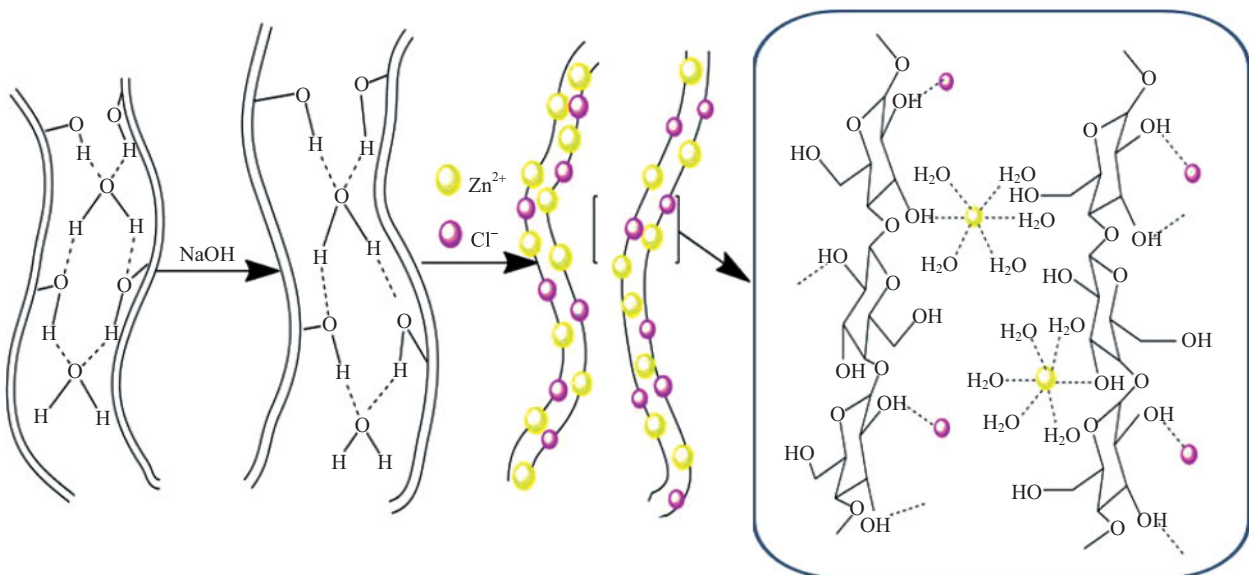


Fig.5 Schematic of chelate formation and changes in hydrogen bonding in cellulose after $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ solution treatment

4 Conclusions

BSKP fibers pretreated in a NaOH aqueous solution were then treated with $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ solution, and the effects of various molar ratio of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ on filter paper properties were investigated. Results revealed that the air permeability and pore size of the filter paper were improved after treatment with the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ solution. SEM observation indicated that fibers treated by the $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ solution became swollen and exhibited slight fibrillation, leading to improved filter paper properties. In addition, the XPS spectra demonstrated the existence of Zn^{2+} . The optimal molar ratio (1:14:1) of $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ for improving filter paper performance was found by experiment. And under this condition, the optimum air permeability of $1450.4 \text{ L}/(\text{m}^2 \cdot \text{s})$, tightness of $0.159 \text{ g}/\text{cm}^3$ and maximum pore size of $58.0 \mu\text{m}$ were obtained. Briefly, this study has provided a feasible and recommended method for obtaining high permeability filter paper by treating the BSKP fiber in NaOH aqueous and $\text{ZnCl}_2/\text{H}_2\text{O}/\text{C}_2\text{H}_5\text{OH}$ solutions.

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