Analysis of Dissolved and Colloidal Substances in Old Corrugated Containers' Whitewater and Dissolved Substances' Impact on Colloidal Substances' Stability

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The physicochemical properties of dissolved and colloidal substances (DCS) in the old corrugated containers' (OCC) whitewater were studied. Then, the colloidal substances (CS) were separated from dissolved substances (DS) and the effect of inorganic salts on the stability of CS (not DCS) was studied for the first time. The results showed that many DCS aggregated and attached to the fiber surface when pulping. The primary sources were resin, lignin, adhesives, coating fixatives, and fillers. The colloidal stability of DCS and solids of whitewater differed because the DCS contained less filler. Both Ca^{2+} and Na^+ can affect the stability of CS, but Ca^{2+} led to more CS instability and aggregated into larger flocculent precipitates. The surprising discovery in the experiment was that when Ca^{2+} and Na^+ were added together, the instability degree of the system was between the addition of Ca^{2+} and Na^+ alone. Ca^{2+} played a dominant role in affecting the stability of CS, and Na^+ competed for adsorption sites.

Keywords: OCC whitewater; Dissolved and colloidal substances; Stability; Instability

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INTRODUCTION

Wastepaper recycling is beneficial to saving resources of wood, protecting the environment, and reducing production costs. Old corrugated containers (OCC) are one crucial component of the wastepaper industry (Monte *et al.* 2009; Li *et al.* 2018). When the OCC are reused, many foreign materials are introduced to system, with some of them becoming present in whitewater as dissolved and colloidal substances (DCS) (Liang *et al.* 2018). The composition of DCS, which mainly comes from pulp, fillers, recycled water, and the chemicals added during the papermaking processes, is complex (Hubbe *et al.* 2012; Wang 2013). It has been found that DCS keeps accumulating with whitewater and might interfere in the wet-chemical environment of pulp and papermaking (Rundlof *et al.* 2000, 2002; Nurmi *et al.* 2004). Most of the colloids in DCS have carboxyl groups and are negatively charged. The DCS will preferentially react with cationic additives, reducing the effectiveness of the cationic additives and increasing the required dosage, simultaneously increasing costs, and further deteriorating the wet-end of pulp and papermaking. Second, with the environment changing suddenly, it will lead to the instability of the whitewater system from a large amount of DCS flocculates and formation of more harmful stickies, resulting in mesh clogging, eventually affecting the operation of the paper machine and the quality of the paper products (Blanco *et al.* 1996; Monte *et al.* 2004; Blanco Suárez *et al.* 2007).

Previous studies mainly have focused on the characteristics of DCS as a whole. For example, Liang *et al.* (2018) found that with the increase of water recycling times, the accumulation of DCS increased linearly and the concentration of inorganic ions also increased. Xu *et al.* (2004) concluded that in a closed system, any reduction in the water consumption of the paper machine would increase dissolved and colloidal substance. Wang (2014) reported that DCS in whitewater mainly came from agents, extraction content of fiber raw material was minimal. Few scholars have explored the effects of electrolytes on the stability of CS and the mechanism of DCS instability. Interestingly, cultural paper and old newspaper have been widely studied (Francis and Ouchi 2001; Zhang *et al.* 2007; Su *et al.* 2016). The OCC was researched less, as a critical component of recycled paper in the paper industry.

This experiment needed a large amount of wastewater, which was difficult to transport and store (Jiang *et al.* 2018). Therefore, simulated OCC whitewater was used to explore the influence of inorganic electrolyte on the stability of CS, and to determine the maximum concentration of inorganic electrolyte when DCS water was destabilized, to provide technical assistance to control the DCS of whitewater effectively.

EXPERIMENTAL

Materials

Preparation of simulated OCC whitewater

The OCC (Mei Ying Da, Shenzhen, China) was cut into 5 × 5 cm pieces and stored in a sealed bag at room temperature for 24 h to balance the moisture. The pieces were beaten at 300 rpm for 20 min at 10% pulp concentration in a high consistency pulp machine (Adirondack Machine Corporation, Hudson Falls, NY, USA) at 60 °C. The pulp concentration was diluted to 2% with tap water and stirred at 60 °C for 1 h. The pulp was then screened with a 200-mesh dynamic water filter (DDJ) under 800 rpm stirring, and the liquid was filtered as OCC simulated whitewater (Jiang 2007; Chen *et al.* 2014).

Separation of DCS and CS in simulated whitewater

The OCC whitewater was centrifuged at 2000 rpm for 20 min. The solids of the whitewater were separated to obtain the supernatant as the DCS water. The DCS water was dialyzed with deionized water by a dialysis tube for 48 h (deionized water replaced every 4 h), then obtained the CS (Johnsen *et al.* 2004; Chen 2016).

DCS stability test

Different concentrations of NaCl, $CaCl_2$, and mixed solutions of them were prepared and preheated to 45 °C. The NaCl, $CaCl_2$, and mixed solution were added to 100 mL CS water, stirred at 50 rpm at 45 °C for 30 min. The zeta potential and the particle size of the CS were then tested. The supernatant was obtained at 2000 rpm for 20 min and turbidity measured to analyze the influence of inorganic electrolyte on the stability of CS.

Analytical Methods

The cation demand (CD) was automatically titrated and measured using the PCD-03 streaming current detector (Mutek, Filderstadt, Germany). The electrical conductivity of different water samples was measured using the DDSJ-308F conductivity measurement (Leici, Beijing, China). Turbidity was measured using the 2100N Turbidimeter (Hach, Loveland, CO, USA). The zeta potential and particle size were measured using the Zetasize Nano-SZ90 Zeta potentiometer (Malvern, Westborough, MA, USA). The chemical oxygen demand (COD) was determined by the 5B-3B water quality analyzer (Lianhua, Shanghai, China). The simulated concentration of Ca^{2+} and Na^{+} in whitewater was determined using the 700 Series ICP-OES inductively coupled plasma emission spectrometry (Agilent Technologies, Santa Clara, CA, USA). After the OCC was pulped under different conditions, the DCS appearance was studied by the scanning electron microscope (SEM) (F16502) (Phenom, Eindhoven, Holland). The material composition of DCS was analyzed by pyrolysis-gas chromatography-mass spectrometry-QP2010 Plus (Py-GC-MS) (Shimadzu, Kyoto, Japan) and determination of thermal stability by the STA 449F5 thermogravimetric analyzer (TG) (Netzsch, Selb, Germany) (Li 2005; Li *et al.* 2008; Sousa *et al.* 2018).

RESULTS AND DISCUSSION

The Basic Properties of Simulated OCC Whitewater

The pH of the simulated whitewater was 7.8. The basic characteristics of simulated OCC whitewater, DCS, and CS is shown in Table 1. The solid content, the ash content, and the CD value of DCS water decreased compared with the whitewater. This trend was because the fibers and filler of whitewater were removed, and the positive charge needed for carboxyl ionization on the fiber surface was reduced after centrifugal sedimentation, resulting in decreased solid content, ash content, and CD values. The CD value and turbidity of CS water were only slightly reduced, showing that CS plays a major role in the cationic demand of DCS. The decrease of turbidity indicated that the DCS turbidity was mainly contributed by CS (Nylund *et al.* 2007).

The COD content of whitewater was higher than that of DCS and CS water, but there was no remarkable difference between DCS and CS. This was because the fibers of whitewater were removed after centrifugal sedimentation, which provides the most COD of whitewater. The COD content of CS accounts for 79.7% of DCS.

Table 1. Basic Properties of OCC Whitewater

Parameters	Measurement Indicators							
	Solid Content	Ash	Turbidity	CD	Conductivity	COD	Metal Ion Content (mg/L)	
	g/L	g/L	NTU	μeq/L	μs/cm	mg/L	Ca^{2+}	Na^{+}
Whitewater	1.75	0.50	766	132	666	1005.2	360.8	23.4
DCS	1.2	0.31	38.1	99	610	154.8	204.03	8.39
CS	0.65	0.13	25.5	79	20	123.4	10.456	2.951

The conductivity of the DCS water was slightly lower than the whitewater. The conductivity of the CS water decreased remarkably after dialysis, indicating that DCS conductivity was mainly contributed by DS. Conductivity can be used to show the content of inorganic electrolytes. The main inorganic electrolytes in whitewater were Ca^{2+} and Na^{+} (Tang 2018), and the content of Ca^{2+} and Na^{+} also decreased remarkably in CS after dialysis in Table 1. The materials removed by centrifugation were fillers and other materials, and most of the dissolved substances still existed in the DCS water. Inorganic electrolytes were almost removed after dialysis treatment, resulting in a sharp decline in the conductivity of CS.

Basic Properties of DCS

SEM analysis

The OCC was diluted with raw whitewater (Fig. 1a), DCS water (Fig. 1b), and distilled water (Fig. 1c) to make pulp. The micromorphology of the paper sheets with different water pulping was observed by SEM. Figure 1(a) shows whitewater OCC pulping. A great deal of adhesives flocculated to form large stickies on the fiber surface, and there were also some fillers that make it look messy. Figure 1(c) was relatively clean compared with Fig. 1(b), but there was some large flocculation, indicating that DCS flocculates into a larger stickie deposited on the fiber surface. Therefore, the stickies acted like the flocculant in Fig. 1(a). Thus, it can be said that DCS had a great influence on the system due to the occurrence of unstable flocculation and is a key factor in the study of whitewater stability.

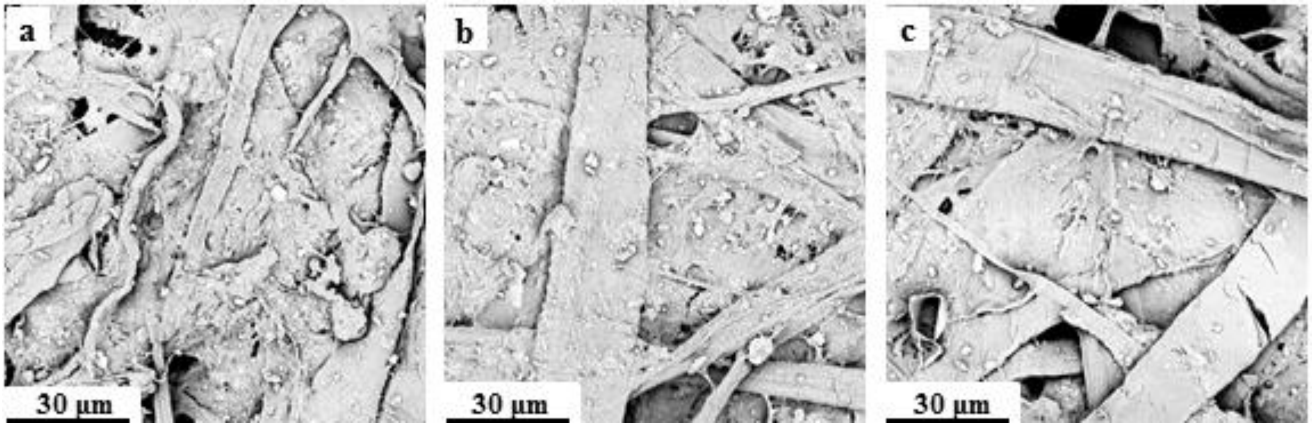

Fig. 1. SEM of OCC pulping under raw whitewater (a), DCS water (b), and distilled water (c)

Speciation Analysis of CS

The particle size of CS was fitted to a normal distribution with an average particle size of 483.3 nm (see Fig. 2). The particle size range was between 255 to 1000 nm, and most samples were approximately 500 nm. Because the DS was removed and the CS was fluffy, it led to an increased CS particle size. With the Dindal effect, a bright "pathway" could be observed in the colloid. Therefore, stability theory of colloidal system can still be introduced when discussing DCS stability (Wågberg *et al.* 2010).

Composition Analysis of DCS by Py-GC-MS

There were many polymer substances in the DCS component, which had a large molecular weight and could not be completely gasified. Therefore, they were decomposed into small substances and detected by Py-GC-MS. Then, its composition and possible sources were further speculated (Yuan *et al.* 2012). The results of the comparative spectrum are shown in Table 2.

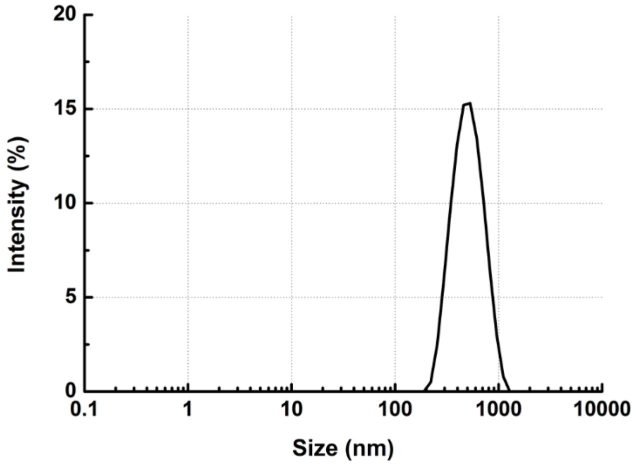

Fig. 2. Particle size distribution of CS

The DCS pyrolysis product contained abundant compounds, such as acetone, 2-butanone, 2-methyl-2-cyclopentene-1-ketone, styrene, toluene, pentadecanoic acid (palmitate), and octadecanoic acid (stearic acid). From these different physical properties, possible sources can be roughly inferred. For example, 2-butylketone was an organic solvent for adhesives and coatings, 1,3-dimethoxybenzene, o-xylene, and 2-methyl phenol were products of lignin. Styrene was an important copolymer of styrene-butadiene-styrene (SBS) and styrene-styrene-styrene butadiene rubber (SBR) as well as a copolymer of latex coatings (Sarja 2007). Further, there were additional chain alkanes, alkene-type material, such as undecene, undecane, tridecane, pentadecene, and so on, which came from active agent or plasticizer. Hexadecanoic acid (palmitic acid) and octadecanoic acid (stearic acid) may have come from dispersants, surfactants, or in-pulp sizing agents in the papermaking process. Octadecanoic acid may also come from resins in the fibrous material itself. From the Table 2, it can be determined that DCS was complex and included the resin and lignin from wood, as well as the synthetic compounds of various adhesives, coating fixers, plasticizers, and other substances.

TG Analysis of DCS

The whitewater mainly contained DCS, fine fibers, fillers, and other substances, so thermogravimetric experiments were performed on DCS and whitewater to investigate its difference.

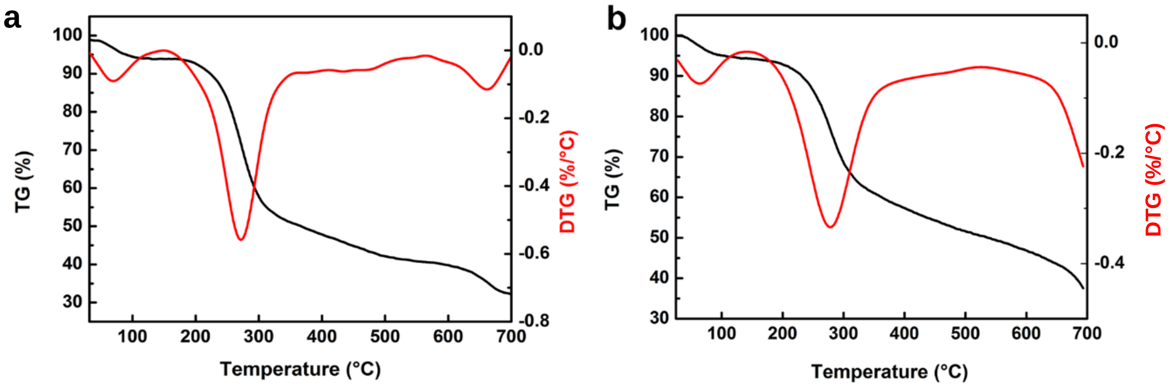

Fig. 3. TG and DTG curves of DCS (a) and whitewater (b)

Table 2. Composition Analysis of DC*S* by Py-GC-MS

Time (min)	Con-tent	Molecular Formula	Name	Possible Source
1.901	11.36	CO_2	Carbon dioxide	Deep cracking of organic matter or filler
3.226	6.89	C_4H_8O	2-Butanone	Adhesive
5.152	1.29	C_7H_8	Toluene	Deinking agent, defoaming agent, *etc.*
6.966	1.49	$C_8H_{10}O_2$	1,3-Dimethoxybenzene	Lignin
7.123	0.93	C_8H_{10}	O-xylene	Lignin
7.539	5.31	C_8H_8	Styrene	Polystyrene, SBR, SBS, *etc.*
7.865	1.03	C_6H_80	3-Methyl-2-cyclopenten-1-one	Scouring agent, activator, *etc.*
8.572	2.18	C_9H_{10}	Allylbenzene	Sizing agent, drying agent and filter agent for processing paper chemicals, *etc.*
9.492	1.54	$C_{10}H_{22}$	Decane	Organic synthesis of resins
10.476	1.02	C_7H_8O	2-Methyl phenol	Resins, dyes, antioxidants and antimicrobial agents, *etc.*
10.981	1.42	$C_{11}H_{22}$	Hendecene	Surface-active agent
11.093	1.09	$C_{11}H_{24}$	Hendecane	Surface-active agent
12.273	1.27	$C_{12}H_{24}$	Dodecene	Surface-active agent, plasticizer, *etc.*
13.376	1.08	$C_{13}H_{28}$	Tridecane	Surface-active agent
14.165	1.83	$C_{14}H_{28}$	Pentadecene	Plasticizer, activator, *etc.*
14.912	1.62	$C_{15}H_{30}$	Toluene	Plasticizer, activator, *etc.*
15.575	2.13	$C_{16}H_{34}O$	Hexadecanol	Detergent, plasticizer, *etc.*
16.619	1.07	$C_{14}H_{28}O$	2-Tetradecanone	Scouring agent, activator, *etc.*
17.635	2.89	$C_{16}H_{32}O_2$	Pentadecanoic acid	Sizing agent, disperser, *etc.*
19.522	1.04	$C_{19}H_{38}O$	Nonadecanone	Internal sizing agent, *etc.*
21.072	4.73	$C_{18}H_{38}O_2$	Octadecanoic acid	Surface-active agent, disperser, *etc.*

It can be determined from Fig. 3 that there was a difference between DCS and whitewater, which was specifically manifested in the wide temperature range of the thermal cracking, with remarkable weight loss at 150 to 350 °C and 650 °C of DCS, and whitewater at 150 to 370 °C and 650 to 700 °C. Figure 3 shows that the composition of DCS was complex and contained a variety of organic compounds, including low-molecular weight organics that were easy to be pyrolyzed at a wide and lower temperature (150 to 350 °C), and macromolecules, such as cellulose, of whitewater that were pyrolyzed near 370 °C. In Fig. 3(b), the weight loss at 150 to 370 °C may be from the pyrolysis of cellulose and DCS. The weight loss at 650 °C indicated that there were inorganic salt electrolytes in DCS (Miranda *et al.* 2006). There was a more remarkable weight loss in whitewater than DCS at 650 to 700 °C, mainly due to the pyrolysis of calcium carbonate, the fillers that were not found in DCS (Wang *et al.* 2012).

Electric Charge Characteristics of DCS

The cationic demand and zeta potential are commonly used to evaluate the charge characteristics of DCS. The CD value can reflect the negative charge content of DCS while zeta potential can show the potential difference between the continuous phase of the colloid and the fluid stable layer attached to dispersed particles, which was closely related to the stability of DCS. The CS water was adjusted to different pH values and then the cationic demand and zeta potential of the solution were measured. The results are shown in Fig. 4.

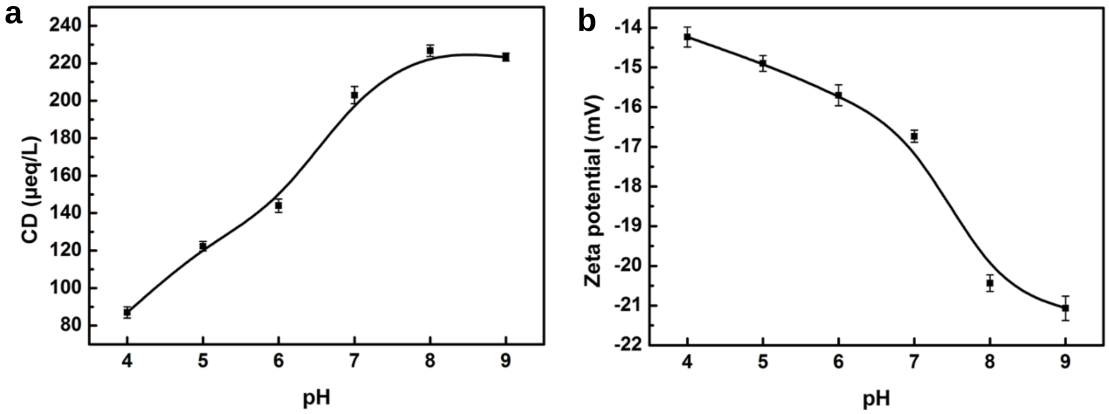

Fig. 4. Effects of different pH conditions of CS on CD (a) and Zeta potential (b)

As the pH value increased, the CD value and the absolute value of zeta potential kept increasing. This was attributed to the fact that CS mostly contained carboxyl groups and the ionization degree was affected by the pH value. Under the condition of low pH value, the carboxyl protonation degree was more protonated. With the increase of pH value, the carboxyl group was gradually dissociated into carboxylate anions, which increased the amount of negative charge carried by CS, increasing the CD value. At the same time, the increase of carboxyl ionization degree enhanced the negative charge of colloidal particles in CS, so the absolute value of zeta potential increased and the system stability improved (McLean *et al.* 2005; Lee *et al.* 2012). However, when the pH value was 9, the CD value and zeta potential were stable due to the fixed colloid content in the CS water. This indicated that the charge characteristics of DCS were related to the number of colloids, and zeta potential and CD values did not always increase with the increase of pH.

Effect of Reaction Time

Turbidity is caused by suspended solids (SS) and CS in the water. In the absence of SS, its value can indirectly represent the content of CS (Sundberg *et al.* 1993). The residual turbidity of the supernatant after centrifugation of the water was detected to reflect the content of CS.

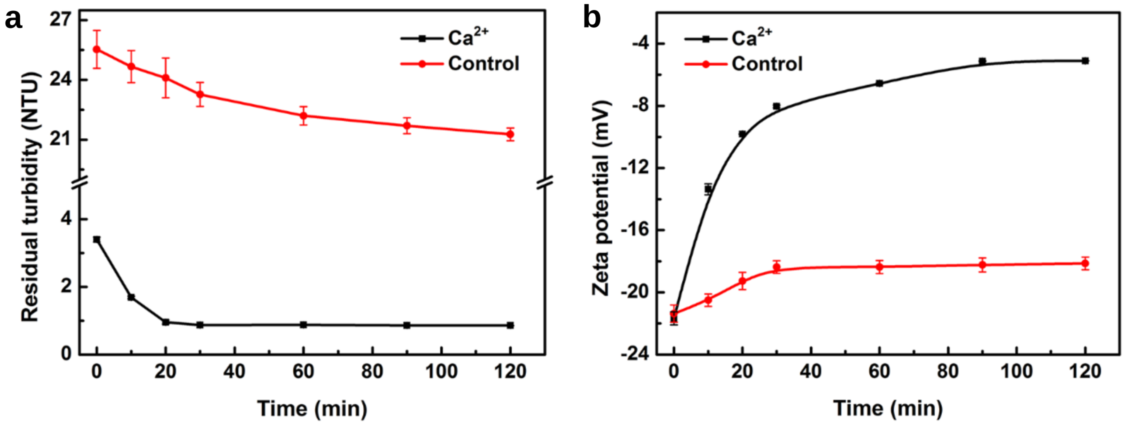

Fig. 5. Effects of Ca^{2+} addition at different times on residual turbidity of CS (a) and zeta potential (b)

Ca^{2+} was added into the CS water at a concentration of 8 mmol/L, the supernatant was centrifuged at 2000 rpm for 20 min. The effect of inorganic electrolyte on the stability of CS with time were tested by the change of turbidity and zeta potential.

As shown in Fig. 5, when adding Ca^{2+} to CS water, the residual turbidity decreased immediately from 25.5 NTU to 3.5 NTU. Turbidity then decreased slowly from 3.5 to 0.85 NTU during the next 30 min. This determined that Ca^{2+} can affect the stability of CS immediately, lose stability, and regain stability in 30 min. Concurrently, with the extension of time, the absolute value of zeta potential of CS continued to decrease and become stable after 30 min. This was due to the carboxyl group in DCS ionizing and reacting with Ca^{2+} to form a non-ionizing interfering substance, thereby weakening the electronegativity of the surface of colloidal particles. This indicated that Ca^{2+} had a great effect on the stability of CS, and the CS was completely unstable and gained stability in 30 min.

Effects of Ca^{2+} and Na^+ Alone and Synergistic Action on the Stability of CS

In Figs. 6 and 7, the inorganic electrolytes had a great influence on the stability of CS, with the residual turbidity of added Ca^{2+} decreasing more remarkably than Na^+.

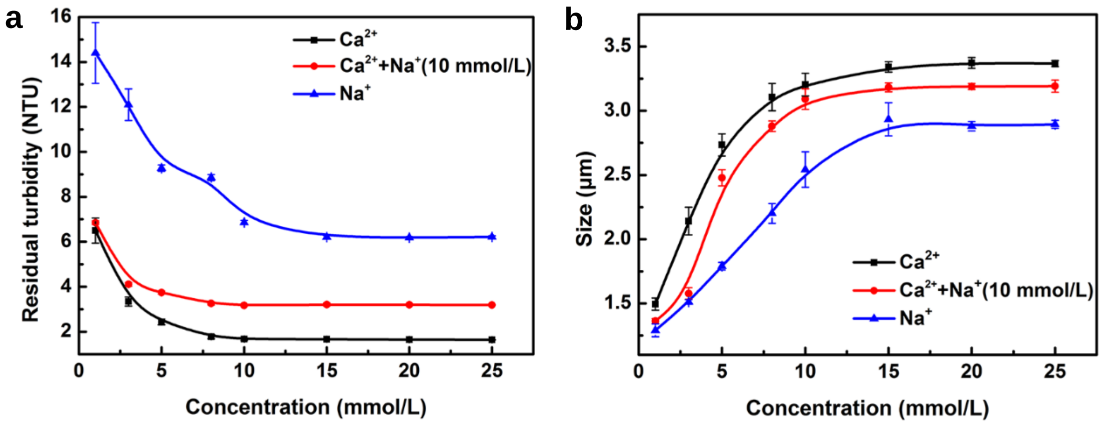

Fig. 6. Effects of inorganic electrolyte concentration on residual turbidity (a) and on size (b) (· Na^+ was fixed at 10 mmol/L, adding different Ca^{2+})

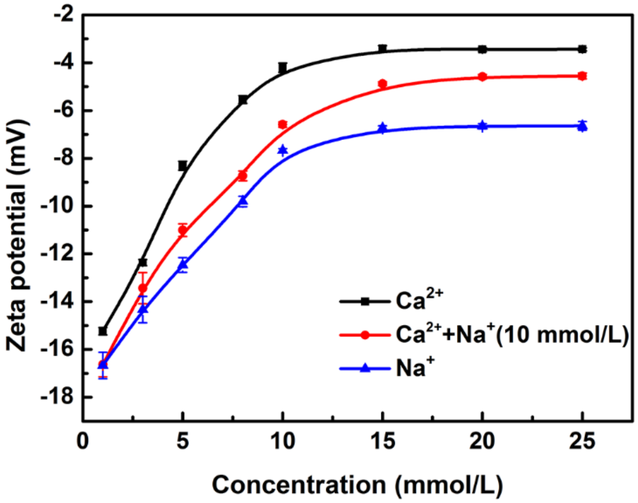

Fig. 7. Effects of inorganic electrolyte on zeta potential

When metal ions were added, they would enter the electric double layer of colloidal particles through charge neutralization, thus the repulsive force between colloidal particles was reduced and it was easy to attract and aggregate with each other. This effect increases with the increase of the concentration of counter-ions (Verwey 1947). In addition, CS was noticeably negative and adsorbed with Ca^{2+} to balance a part of the electrical property. After the metal ions were pressed into the electric double layer, the charge reversed and the absolute value of zeta potential decreased, thus destabilizing the system (Bobacka *et al.* 1998; Stack *et al.* 2019).

The addition of Na^{+} had little effect on the change of the average particle size of CS. This may have been because Ca^{2+} reacted with the resin in CS to form an insoluble viscous substance, while Na^{+} only compressed the double-electron layer, making the colloidal particles easily aggregate into loose flocculation, it cannot be combined with CS to form viscous insoluble substances (Lee *et al.* 2012).Therefore, Ca^{2+} made colloid particle size change more prominent than Na^{+}.

A surprising finding was that when Ca^{2+} and Na^{+} were added together, the system instability was between the Ca^{2+} and Na^{+} alone in Figs. 6 and 7. However, when the Ca^{2+} concentration reached 10 mmol/L and continued to rise, the turbidity, average particle size, and zeta potential of CS tended to be gentle and close to the effect of Ca^{2+} alone. This may have been due to the constant concentration of Na^{+} in the solution, which was equivalent to acting as a "buffer" in the system (Chen 2016). In addition, there is competition between Na^{+} and Ca^{2+} for adsorption sites. When the calcium ion forms a complex with a pair of carboxyl groups at a fiber surface, such complexation will have a greater contribution on colloidal instability. Such complexation would be discouraged by the presence of Na^{+}.

CONCLUSIONS

1. The cationic demand and turbidity of DCS were mainly contributed by CS, while the electrical conductivity was mainly contributed by DS. Its sources were complex, including resin and lignin of the raw material itself, a variety of adhesives, fixatives, and fillers. There was a difference in the thermal stability of DCS and solids. The main weight loss substances of DCS were organic matter and inorganic salts, and the solids were mainly the weight loss of calcium carbonate during pyrolysis.
2. Both Ca^{2+} and Na^{+} affect the stability of CS, but Ca^{2+} was more likely to cause CS instability to produce flocculent precipitation, and the average particle size of CS increased. When Ca^{2+} and Na^{+} were added together, the instability degree of the system was between Ca^{2+} and Na^{+} addition alone. Na^{+} appeared to play a "buffering" role.

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