



Flocculation kinetics of precipitated calcium carbonate induced by electrosterically stabilized nanocrystalline cellulose



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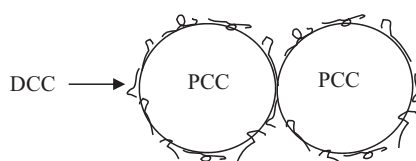
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HIGHLIGHTS

- The kinetics of precipitated calcium carbonate (PCC) aggregation in distilled water with dissolved carboxylated cellulose (DCC) and electrosterically stabilized nanocrystalline cellulose (ENCC) was studied.
- Negatively charged ENCC and DCC can be used as effective flocculants for PCC even at low dosages.
- At low dosage, the flocculation of PCC induced by ENCC and/or DCC is due to charge neutralization.
- At high dosage, PCC flocculation in ENCC/DCC mixture is due to the bridging of DCC-coated PCC by ENCC.

GRAPHICAL ABSTRACT

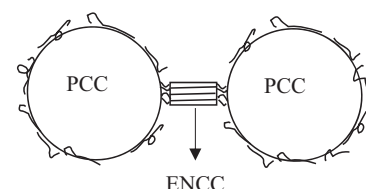


At low dosage:

Charge neutralization

PCC positive charged, DCC negative charged

DCC: dissolved carboxylate cellulose; PCC: precipitated calcium carbonate



At high dosage:

Bridging by ENCC

ENCC: electrosterically stabilized nanocrystalline cellulose

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ABSTRACT

The interactions between precipitated calcium carbonate (PCC) and electrosterically stabilized nanocrystalline cellulose (ENCC), dissolved carboxylated cellulose (DCC), and a mixture of DCC and ENCC have been studied. ENCC can be produced by periodate, chlorite and TEMPO oxidations of cellulose fibers. It has a large surface area, a high carboxylated content and can be used as a high-performance reinforcement material. It is likely to effectively bind and flocculate with PCC to improve the properties of filled paper. Dissolved carboxylated cellulose (DCC) is produced together with ENCC in the oxidation reactions, and also has a high charge density.

The flocculation of PCC induced by ENCC/DCC was measured by photometric dispersion analysis (PDA). It was demonstrated that ENCC or DCC adsorbed first on PCC particle surfaces due to electrostatic attractions, and subsequently ENCC or DCC induces flocculation between PCC. The repulsive electrostatic force reduces to zero when the isoelectric point of PCC particles is reached, allowing attractive van de Waals forces to dominate, resulting in maximum PCC flocculation. After the charge neutralization point was reached, more dissolved carboxylated cellulose adsorbed on the PCC particles, resulting in net negative charges on PCC surfaces, and dispersion of flocs due to electrostatic

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repulsion. For mixtures of dissolved carboxylated cellulose (DCC) and ENCC, DCC adsorbed faster on PCC than ENCC. Excess ENCC resulted in the exchange of DCC with ENCC, with ENCC particles able to bridge PCC. This mechanism was proved by photometric dispersion analysis and dynamic light scattering experiments.

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1. Introduction

Fillers are one of the most significant additives in paper making. Precipitated calcium carbonate (PCC) is a popular filler that is added to paper products to improve optical properties (opacity and brightness), smoothness, printability and sheet formation [1–4]. Moreover, because PCC is less expensive than pulp fibers, the advantage of using fillers to replace fibers is obvious. However, PCC also reduces the mechanical strength of paper by interfering with inter-fiber bonding [5].

Synthetic polyelectrolytes are commonly used to increase retention of fines and fillers and hence to improve paper quality [6]. Gaudreault et al. summarized the chemistry and optimum conditions that induce PCC flocculation by a wide range of polyelectrolytes (PEI, PEO, c-PAM, a-PAM, etc.) [5]. In recent years, scientists focused their attentions on the flocculation of fillers with natural carbohydrates (starch, dextran, lignin etc.). Modgi et al. showed that tapioca starch flocculated PCC resulting in higher rates of flocculation and larger floc sizes compared to potato starch [7]. Ghimici et al. demonstrated the usage of dextran as a flocculating agent with clay fillers [8]. Meanwhile, cellulose and its derivatives have also gained significant attention as flocculants of fillers. Biswal and Singh studied grafted carboxymethylated cellulose as a flocculant of fillers and Liimatainen et al. examined cationically modified cellulose biopolymers to induce aggregation of fillers [9–11].

Korhonen et al. studied the effect of both anionic and cationic cellulose nanofibrils (CNF) on the flocculation of PCC [12]. They observed that higher negatively charged CNF resulted in stronger electrostatic interactions and thus a higher flocculation efficiency of cationic PCC particles. These authors also claimed that the flocculation mechanism for CNF is a hybrid between bridging/network formation and the patch flocculation mechanism [12]. Manfredi et al. prepared a microparticle retention aid model for fillers using polystyrene (PS) microspheres with PEI (polyethylene imine) grafted to PS surfaces and nanocrystalline cellulose (NCC) as the bridging agent [13].

Electrosterically stabilized nanocrystalline cellulose (ENCC) has been recently developed by our group. It has a large surface area, high aspect ratio, a large charge density (typically 3.5 mmol/g and higher), and has similar dimensions as the NCC produced by acid hydrolysis [14]. It can form a network in very dilute aqueous suspensions and may potentially bind and induce aggregation between PCC. In this study, ENCC is produced together with dissolved carboxylated cellulose (DCC) by oxidizing cellulose fibers, where DCC is derived mostly from the amorphous parts of cellulose. The aim of this study was to prove the mechanism about flocculation between nanocellulose material and PCC. Moreover, we intent to explore the use of DCC, ENCC and its mixture on their flocculation of PCC as flocculants. ENCC can be produced by a three step reaction: periodate, chlorite and TEMPO oxidation. After these oxidations, cellulose fibers are separated into nanocrystalline cellulose without intensive mechanical treatment [15]. ENCC can also be produced without TEMPO oxidation, but periodate oxidation results in cellulose degradation, which can be minimized by using less periodate and more TEMPO.

PCC has a positive charge in distilled water, but in tap and mill water PCC carries a negative charge due to the presence of impurities in the water [16]. PCC used in this study has an average particle diameter of 1.3 μm while ENCC is about 200 nm in length and 5 nm in width. ENCC has a high negative charge because of high carboxylated content in the cellulose structure. Equally DCC has a large charge density as well. Thus, ENCC and DCC will adsorb on PCC surfaces by electrostatic attraction in distilled water. We can also prepare ENCC with positive charge by introducing quaternary amine groups which may induce PCC flocculation in mill or tap water [17]. Moreover, ENCC adsorbed on a given PCC particle can attach to the bare surface of another particle, thus forming particle–particle bridges (bridging flocculation). Thus, a strong aggregation between ENCC and PCC can be expected based on these two mechanisms.

2. Experimental

2.1. Precipitated calcium carbonate

Precipitated calcium carbonate (PCC) was provided by Domtar Inc. Its nominal diameter and specific surface area are respectively 1.3 μm and 10.1 m^2/g [18]. We only use distilled water in this study. Pure PCC is positively charged in distilled water [16,19].

2.2. Electrosterically stabilized nanocrystalline cellulose (ENCC)

Nanocrystalline cellulose (ENCC) was prepared by a three-step oxidation process. It consisted of three reactions in sequence: periodate, chlorite and TEMPO oxidations. Periodate oxidation of cellulose breaks the C2–C3 bond in the glucose repeat unit of cellulose, forming two adjacent aldehyde groups [20,21]. Chlorite oxidation further oxidizes the dialdehyde group to a dicarboxyl group [22]. TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl) is the name for a series of water-soluble and stable nitroxyl radical compounds. TEMPO can be used as a catalyst to selectively oxidize primary hydroxyl groups in the presence of secondary ones. Two oxidation systems are commonly employed. The first one is TEMPO/NaClO/NaBr system used in a mildly alkaline environment [23]. The second one is TEMPO/NaClO/NaClO₂ system which performs best in neutral or weak acid media and is used in this study. Its proposed mechanism shows that NaClO firstly oxidizes TEMPO to nitrosonium ions, which oxidize the primary hydroxyl groups to aldehyde groups. Then, NaClO₂ oxidizes the aldehyde groups to carboxyl groups and is reduced to NaClO. In the last step, the NaClO oxidizes hydroxylamine to nitrosonium ions again [24]. In this study, pulp (5 g) was mixed together with 3.33 g of NaIO₄ and 19.5 g of NaCl in 333 mL distilled water. The oxidation reaction was performed at room temperature and stirred at a speed of 107 rpm. The reaction beaker was covered with several layers of aluminum foil to prevent the entry of light for 36 h [21].

Then the oxidized pulp was thoroughly washed and suspended in 250 mL distilled water. 3.56 g of NaClO₂, 14.6 g of NaCl, and 3.3 g of H₂O₂ were added to this mixture for chlorite reaction [22]. The mixture was stirred at a speed of 107 rpm at room temperature,

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