



# Modification of wollastonite by acid treatment and alkali-induced redeposition for use as papermaking filler<sup>☆</sup>



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## ABSTRACT

In order to increase the applicability of wollastonite in the papermaking industry, it was modified by an acid treatment and alkali-induced redeposition. The modification process consisted of two steps: first, sulfuric acid was mixed with wollastonite, resulting in the partial dissolution of the wollastonite particles. Next, the pH of the mixture was adjusted to make it alkaline so that the dissolved material was redeposited on the surfaces of wollastonite particles, which maintained their original acicular morphology. The resulting wollastonite particles were larger, had a higher zeta potential, and were brighter than were those of the original sample. The properties of paper sheets, especially bulk and brightness, in which a  $\text{Na}_2\text{SiO}_3$ -induced redeposited wollastonite sample was used as the filler were superior to those in which the original wollastonite sample or other alkali-induced redeposited wollastonite samples were used.

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## 1. Introduction

Globally, fillers are now being used in papermaking to meet the needs of the paper industry. For most grades of paper, fillers are usually the second most important component of the paper stock in terms of their added amounts [1]. Fillers, which are used as replacements for fibers, result in decreases in the cost and energy involved as well as improvements in the optical properties of paper and smoothness [2]. However, a challenge associated with the use of fillers, especially when added in large amounts, is a decrease in fiber bonding, which leads to a reduction of the paper strength, and an increase in the phenomena of abrasion, dusting, and bad filler retention [3]. If these negative impacts were to be mitigated, it would allow for huge cost and energy savings owing to the use of fillers in larger amounts [4,5].

Chain silicates have been studied more extensively as fillers than have other organic and inorganic fillers because these silicates have potential for use in a vast variety of applications, including in plastics, paint, cosmetics, and papermaking. The use of chain silicates, especially calcium silicate (wollastonite), is highly dependent on their properties, including their chemical purity, specific surface area, particle size, and morphology [6,7]. Wollastonite belongs to an interesting new class of functional materials. It is a form of the naturally occurring mineral white calcium silicate ( $\text{CaSiO}_3$ ). Wollastonite is a hard material (Mohs hardness of 4.5–5.0) and has a specific gravity of 2.78–2.91. Because

of its acicular nature (its aspect ratio is usually 5–20), wollastonite is easy to combine with plant fibers and form fiber-mineral network structure which is helpful to increase the filler retention of wollastonite compared to the traditional calcium carbonate filler [8,9]. Tensile and bursting strength of papersheets with wollastonite as filler are significantly higher than those with ground calcium carbonate (GCC) or precipitated calcium carbonate (PCC) as filler [10]. However, the brightness of papersheets with wollastonite as filler is lower than that with GCC or PCC as filler [11].

Accordingly, methods of modifying and characterizing wollastonite have been studied extensively. Many of the disadvantages of using wollastonite-based fillers in industry can be overcome by synthesizing and modifying calcium silicate composites. Wollastonite is commonly modified by mechanochemical methods [12], the envelope method [13], the coupling method [14], and nanocoating [15]. This is done mainly to improve the compatibility of wollastonite with respect to polymers. However, these modification methods often result in hydrophobic products and are not conducive for use in papermaking wet-end processes. To increase the use of wollastonite in papermaking wet-end processes, another method of modifying wollastonite powder, namely, cationization, is being currently studied [16,17]. This method increases the retention rate and bonding strength of wollastonite as a filler for use in papermaking and reduces the wear and tear of the dewatering elements of the papermaking machines.

Wollastonite exhibits good chemical stability but will decompose under highly acidic conditions. It can be used for the preparation of silicon dioxide (white carbon black) [18,19]. Owing to these facts, in this study, we first partially dissolved a wollastonite powder in sulfuric acid and then redeposited the dissolved substances onto the surfaces of

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the wollastonite particles under alkaline conditions. As a result, we could reconstruct the structures of the surfaces of the wollastonite particles. After the characterization of the modified wollastonite samples, we investigated the effects of different alkalis on the structural property of the modified wollastonite samples and those of the paper sheets in which they were used as fillers.

## 2. Experimental

### 2.1. Materials

The wollastonite sample used as the substrate in this study was provided by JiangXi H&J Mineral Fiber Technology Co., China. Sulfuric acid, sodium hexametaphosphate (SHMP), sodium hydroxide (NaOH), sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), calcium hydroxide ( $\text{Ca}(\text{OH})_2$ ), and sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) were purchased from Tianjin Jiangtian Co., China.

Bleached softwood kraft pulp (BSKP) and bleached hardwood kraft pulp (BHKP) were beaten with a valley beater to 35°SR freeness before used. Then BSKP (30%) and BHKP (70%) were mixed and spared. The cationic polyacrylamide (CPAM) used (molecular weight = 8,000,000) and GCC were industrial grade.

### 2.2. Wollastonite preparation

In a 250-ml three-necked flask, 10 g of wollastonite and 0.2 g of SHMP (both on a dry weight basis) were added. Distilled water was then added to result in a mixture of 100 g. Under stirring at 150 rpm, the mixture was heated to 55 °C using a water bath and cooked for 20 min at this temperature. Next, 0.95 ml of sulfuric acid (98%) was added (sulfuric acid doses of 20% was used based on weight of wollastonite in this study), and the resulting mixture was cooked at 60 °C for 10 min. The pH was adjusted to 9.0; make the mixture alkaline by stirring at 200 rpm (this was done by adding a 20 wt% alkaline solution of NaOH, or  $\text{Na}_2\text{SiO}_3$ ,  $\text{Na}_2\text{CO}_3$ ,  $\text{Ca}(\text{OH})_2$ ), so as to redeposit the dissolved material on the surfaces of the wollastonite particles. The slurry was filtered to remove the excess ions and dried in an oven at 105 °C. The process flow of wollastonite modification method is shown in Scheme 1. The resulting dried powder was directly used for paper sheet preparation.

### 2.3. Characterization

The morphology of the obtained powder was analyzed using a scanning electron microscopy (SEM) system (Hitachi SU-1510), the test samples were prepared as the standard GB/T2679.11-2008. The particle diameter was measured with laser diffraction particle size analyzer (LS13-320, Beckman Coulter). The precipitated powder was also subjected to X-ray diffraction (XRD) analysis (Tongda TD-3500). The diffraction patterns were recorded for  $2\theta$  values of 10–50°, with the step scan size being 0.02° (1 s at each step). The zeta potential was measured with a fiber potential analyzer (AFG Analytic GmbH, Germany).

The brightness of the wollastonite powder was determined using an optical tester (Elrepho, L&W, Sweden), and the test samples were prepared as the standard GB/T26464-2011.

### 2.4. Paper sheet preparation and determination of paper properties

Paper sheets with a target basis weight of 60 g/m<sup>2</sup> were prepared using an automatic sheet former system (RK-ZA-KWT, PTI, Austria). The fabricated sheets were then pressed for 5 min using a hydraulic press and dried at 95 °C. The target loading level was controlled at 5%, 10%, 15%, 20%, and 25% (based on the total dry weight of the pulp and filler), and 0.05% CPAM was used during the sheet formation processes. The paper sheets were conditioned in a controlled environment (temperature of  $23 \pm 1$  °C and relative humidity of  $50 \pm 1\%$ ) before analysis.

The strength properties (tensile and burst strengths) of the paper sheets were determined using a tensile strength tester (L&W, Sweden) and a bursting strength tester (L&W, Sweden). The thickness of the sheets was measured using a paper/paperboard thickness tester (L&W, Sweden). The values of the bulk were calculated on the basis of the thickness and grammage. All properties of the paper sheets were determined in keeping with the China standards.

### 2.5. Filler retention in the paper sheets

After the filler samples and paper sheets had been calcined at 575 °C in a muffle oven, the residue consisted mainly of wollastonite. The weight of the residual ash was determined and the filler retention rate was calculated using the following equation:

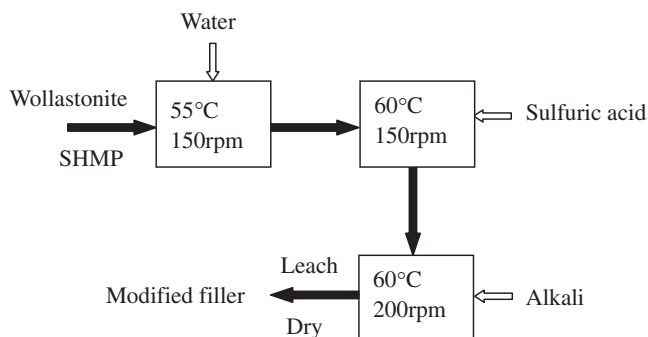
$$R(\text{filler, \%}) = \frac{A-B}{(1-D) \times C} \times 100\%$$

where *A* is the total weight of the ash from the sheet, *B* is the weight of the ash of a sheet of the original pulp fibers, *C* is the amount of the filler incorporated in each sheet, and *D* is fillers corresponding weight loss.

## 3. Results and discussion

### 3.1. Morphology of wollastonite powders

The wollastonite sample used in this study was a white powder, as can be seen from the exterior appearance in Fig. 1. The surface morphology of the modified wollastonite sample was significantly different from that the unmodified sample. As can be seen from SEM image in Fig. 2, the wollastonite particles were of different sizes, with the majority of them being acicular and having a large aspect ratio. A small number of the particles had a blocky structure; this was mainly owing to the crystal structure of wollastonite, and the processing technique used. Further, calcium silicate often contains calcium carbonate. This can also generate a certain amount of blocky particles during processing. By increasing the



**Scheme 1.** Process flow for modifying wollastonite through acid and alkali treatments.



**Fig. 1.** The exterior appearance photo of original wollastonite.

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