

## Adsorption of tailored carboxymethyl cellulose polymers on talc and chalcopryrite: Correlation between coverage, wettability, and flotation

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### ABSTRACT

A carefully chosen selection of CMC polymers was used to probe the depression of talc and chalcopryrite and to highlight the effect of specific alterations of the bulk polymer chemistry on the power and selectivity of the depressants. In situ tapping mode atomic force microscopy (AFM), captive bubble contact angle measurements, and flotation tests were used to study the adsorption of the CMC polymers on chalcopryrite and talc. The degree of substitution and distribution of substitution of carboxymethyl groups on CMC was seen to have a strong influence on the ability of the polymers to adsorb onto the surface of talc and chalcopryrite. High substitution results in low coverage on both minerals whereas low substitution results in higher coverage. The influence of distribution of substitution (random or clustered) was also seen to affect the adsorption, with more random/even substitution giving rise to greater adsorption on talc and chalcopryrite. The contact angle of talc and chalcopryrite is significantly altered in the presence of the three polymers, with increased surface coverage (determined by AFM) correlating with the ability of the polymers to decrease the wettability of talc and chalcopryrite. This trend was also followed in single and mixed mineral talc and chalcopryrite flotation.

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### 1. Introduction

Carboxymethyl cellulose is a commonly used polymer depressant for the rejection of talc in flotation (Bulatovic, 2007; Pugh, 1989). The literature on talc depression with CMC is extensive, with many studies on the role of solution pH and ionic strength (Khraisheh et al., 2005; Morris et al., 2002) in controlling adsorption and depressant efficacy, studies on the binding of CMC with talc in the absence and presence of metal ions (Burdukova et al., 2008; Cuba-Chiem et al., 2008a,b; Parolis et al., 2008; Wang and Somasundaran, 2005), and on the effect of degree of substitution and molecular weight (Khraisheh et al., 2005; Shortridge et al., 2000; Steenberg and Harris, 1984) on depressant action. A new aspect to polymer depressant research is the methodology of studying adsorbed polymer layer properties on mineral surfaces in situ, i.e. while the polymer is hydrated and in solution (Beaussart et al., 2009a; Mierczynska-Vasilev et al., 2008). This approach has been useful in linking the adsorbed layer properties (i.e. coverage, thickness) with contact angle reduction and flotation recovery (Beaussart et al., 2009b). The goal of this study has been to apply the same approach to CMC adsorption on talc, and to combine it with a similar study of the CMC polymers on a valuable mineral phase, in this case chalcopryrite.

In addition to combining a study of polymer adsorption on gangue and valuable minerals, the bulk polymer chemistry of the polymer material has been varied systematically. The major structural variable altered is the degree of substitution (DS) in the polymer. CMC contains carboxymethyl groups substituted along the polymer chain that can protonate or deprotonate depending on the solution pH. This charge along the polymer chain can have a major influence on the polymer conformation (due to varying intramolecular electrostatic repulsion) and adsorption on mineral surfaces (Hoogendam et al., 1998a,b). The degree of intramolecular and inter-molecular electrostatic repulsion can be modified by changes to the solution ionic strength which, when increased, can shield neighbouring charges from each other's influence. Due to this interplay between solution conditions and polymer adsorption, the pH and ionic strength were kept constant to ensure that the differences in polymer chemistry are allowed to assert their influence on the depressant efficacy. A low ionic strength ( $1 \times 10^{-3}$  M KCl) and a mild alkaline pH value (9) were selected.

The techniques used to determine the adsorbed layer characteristics (tapping mode atomic force microscopy (Putman et al., 1994) and captive bubble contact angle measurements) were chosen to ensure that the polymers were studied in situ, so that the information obtained was as relevant as possible to flotation. The study indicates that degree of substitution and distribution of substitution does have a major influence on the ability of CMC to adsorb onto the gangue and valuable minerals, and that this difference

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plays out in a complicated fashion in the single and mixed mineral flotation of talc and chalcopryrite.

## 2. Materials and methods

### 2.1. Materials

Three different carboxymethyl cellulose polymers were used as supplied from CP Kelco. These polymers have different degree of substitution (the average number of carboxymethyl groups attached into the glucose unit) and different relative blockiness (carboxymethyl sequence distribution within the cellulose chain; low blockiness implies random/even distribution along the polymer chain): HSHB – high substitution, high blockiness; LSHB – low substitution, high blockiness; LSLB – low substitution, low blockiness. The general chemical structure of carboxymethyl cellulose is presented in Fig. 1. The molecular weight and polydispersity of these polymers are summarized in Table 1, as provided by CP Kelco. Carboxymethyl cellulose stock solutions of 2000 ppm were prepared by weighing the appropriate mass of solid polymer in  $10^{-3}$  M KCl Milli-Q water and were stirred overnight to ensure complete hydration. The resulting mixtures were diluted afterwards to the desired concentration with background water ( $10^{-3}$  M KCl solution) and adjusted to pH 9.

High-purity Milli-Q water was supplied by an Elga UHQ water system and had conductivity less than  $1 \times 10^{-6}$  S  $\text{cm}^{-1}$  and surface tension of  $72.8 \text{ mN m}^{-1}$  at  $25^\circ\text{C}$ . The solution pH was adjusted with small additions of analytical grade HCl and KOH solutions. Talc particles were purchased from Merck, Germany (>99% pure). X-ray photoelectron spectroscopy (XPS) analysis showed that the surface was free from impurities. The particle size distribution was  $0.5\text{--}100 \mu\text{m}$ , with a  $D_{50}$  of  $15 \mu\text{m}$ , as determined using a Mastersizer X (Malvern Instruments, UK). The chalcopryrite flotation sample was purchased from Ward's Scientific. The mineral sample (approximately 50 mm in size) was crushed using a ring crusher and an <math>850 \mu\text{m} size fraction collected. This sample was then riffled to the mass required for flotation experiments.

The talc and chalcopryrite samples used for atomic force microscopy imaging experiments were provided by the Mineralogy Department of the South Australian Museum and originally sourced from Delaware, USA (talc) and Huanzala, Peru (chalcopryrite). Both mineral samples were determined to be pure and free from impurities using XPS (talc) and electron microprobe (chalcopryrite). Talc samples were cleaved immediately prior to use using sticking tape, producing a very smooth surface with an RMS roughness of  $0.06 \text{ nm}$  (see Fig. 2). Chalcopryrite samples were first shaped with 1200 grit sandpaper then polished with a paste of  $0.3 \mu\text{m}$  polishing grade alumina powder in Milli-Q water on a Buehler Trident

polishing cloth, producing a surface with an RMS roughness of  $1.2 \text{ nm}$  (see Fig. 2). The surface was rinsed with Milli-Q water before a final polish with  $0.015 \mu\text{m}$  alumina particles. The sample was then rinsed with Milli-Q water, and sonicated in acetone (20 min) and methanol (20 min) to remove polishing particles.

### 2.2. Atomic force microscopy (AFM) imaging

The AFM imaging was performed in situ, in tapping mode using a Multimode Nanoscope III (Digital Instruments, Santa Barbara, California). A piezoelectric tube scanner E with the  $10 \times 10 \mu\text{m}$  scan size in the XY direction and a  $2.5 \mu\text{m}$  vertical range was used. Tapping mode cantilevers (V-shape cantilever configuration) for imaging in liquid conditions, with normal tip radius of curvature between 20 and 60 nm and cantilever lengths 100 and 200  $\mu\text{m}$  were used after cleaning (immersion in ethanol, rinsing with high quality Milli-Q water and drying under high purity nitrogen). All experiments were conducted in a class-100 clean room at  $22^\circ\text{C}$ .

Bare mineral surfaces were conditioned in the AFM liquid cell for 30 min in a polymer solution of known concentration. After conditioning the polymer solution was exchanged for the background water solution. The apparent layer thickness ( $\Delta\text{PTV}$ ) was calculated as the difference between the peak to valley distances for the bare surface and the polymer covered surfaces (calculated using the Nanoscope software). Image J image processing software was used to determine the surface coverage of polymer visually observed on the images (Abramoff et al., 2004). Height images obtained from the Nanoscope software were converted to 8-bit grey-scale images. Images were then segmented into polymer features and background mineral by visually setting a threshold in accordance to the height and phase AFM images. The pixels were then converted to black or white if their brightness was respectively, higher or lighter than the threshold brightness, and the area coverage was then determined (Beaussart et al., 2009a).

### 2.3. Contact angle measurements

The contact angle was measured by the captive bubble method in a quartz cell with background solution (potassium chloride  $10^{-3}$  M). The silhouette of the bubble was captured and imaged with a progressive scan CCD camera (JAI CV-MBX, Japan). Advancing and receding contact angles were determined by drawing a tangent close to the edge of the droplet (using Image J software – Drop-Snake) (Abramoff et al., 2004). Three measurements were taken for three bubbles at three different places on the surface. The values reported are an average of these measurements. Experiments were conducted at  $22^\circ\text{C}$  in a class-100 clean room. The effect of adsorption of three CMCs on the contact angle was investigated for talc and chalcopryrite surfaces. The measurements were conducted after 30 min stirring the polymer solution in the cell.

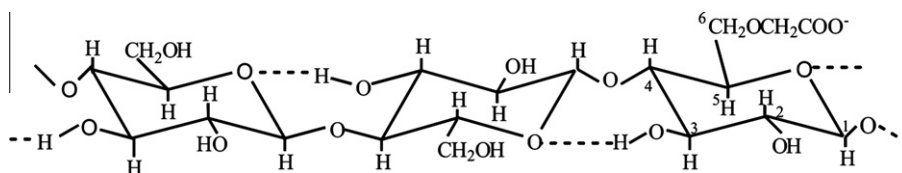
### 2.4. Flotation

#### 2.4.1. Single mineral flotation

Batch flotation experiments were carried out in a 1 L cell using a Denver flotation machine. Talc or chalcopryrite suspensions (2 wt.%

**Table 1**  
CMC molecular weight characteristics and Zeta potential values at pH 9,  $1 \times 10^{-3}$  M KCl.

CMC	$M_w$ [kDa]	$M_n$	$M_w/M_n$
LSLB	157,000	43,000	3.6
HSHB	165,000	44,000	3.8
LSHB	112,000	27,000	4.1



**Fig. 1.** General chemical structure for Carboxymethyl cellulose (CMC).

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