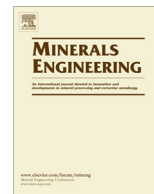


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The colouring effect of pyrrhotite and pyrite on micronised calcium carbonate slurries for the paper industry

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ABSTRACT

Brightness (R457) is the main quality parameter in the production of ground calcium carbonate (GCC) slurries for the paper industry. In order to study how the presence of trace amounts of pyrrhotite ($\text{Fe}_{(1-x)}\text{S}$) and pyrite (FeS_2) affect the brightness of GCC slurries, experiments were performed in which a high quality calcite concentrate was spiked with these sulphides prior to micronisation and subsequent spectrophotometric measurements. The results show that even very small additions (<0.05 wt%) have a significant detrimental effect on the brightness of the product. Pyrrhotite reduces the brightness of the product more than pyrite, probably as an effect of differences in inherent optical reflectivity, specific surface area after micronisation and smearing. In addition, the experiment shows that when brightness is presented as a function of iron concentration, magnetite (Fe_3O_4) produces a different result compared to the sulphides. This indicates that a simple iron analysis is not sufficient to predict brightness of the GCC slurry when several iron-bearing minerals are present. The particle size distribution of the contaminant phase has proven to have a significant effect, as brightness is reduced when the contamination becomes more finely distributed.

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1. Introduction

Minerals are found in nearly every grade of paper, as filler or coating. Both applications are widely used to improve optical properties and surface properties of paper, such as brightness, opacity, gloss, dimensional stability, printability and writeability (Ciullo, 1996; Shen et al., 2011; Kumar et al., 2012). In addition, mineral filling pigments represent a reduction in price of the paper, by substituting for more expensive fibre (Ciullo, 1996).

One of the most common pigments used for fillers in paper is ground calcium carbonate (GCC). This pigment is derived from naturally occurring calcite which is purified, usually by flotation, followed by subsequent micronisation to produce very fine grained (<3 μm) GCC slurries. In 2011, the world consumption of GCC reached 60 million tonne, where paper is the largest market accounting for 39% of the total consumption (Roskill, 2012). During the last decade, the production of GCC has increased dramatically, because of increasing demand from paper industry (Feytis, 2011). Continuous improvements of GCC slurries into more specialised and higher grade products and further improvements of the dressing process in order to increase efficiency and minimise losses has led to new challenges. As the products become more refined, the dressing process also becomes more sensitive to the geologically

derived variations in the raw material. Hence, these variations could have a significant effect on product quality. Brightness is the main quality parameter for all GCC products, and the removal of colouring minerals is essential during the processing performance.

Norsk Mineral is a Norwegian company and a major supplier of raw material for GCC production. The subsidiary Brønnøy Kalk operates the Akselberg open pit deposit in Brønnøysund, 250 km north of Trondheim. Here, some 2 million tonnes of GCC raw material are produced annually. Coarse grained calcite marble constitutes the dominant raw material with graphite and silicates as the main contaminant phases (Watne, 2001). Graphite and silicates are easily separated from the calcite marble by froth flotation, thus resulting in a white product. However, in parts of the deposit, the calcite marble also contains sulphides. These sulphides either occur as massive semi-continuous lenses <1 m in thickness in the marble, or as sulphide grains (20 μm – 3 mm) evenly distributed in the calcite marble itself. In both cases pyrrhotite ($\text{Fe}_{(1-x)}\text{S}$) is the main sulphide mineral, accounting for approximately 70% of all non-calcite grains. The remaining 30% is predominantly pyrite (FeS_2). Furthermore, results from X-ray Diffraction (XRD) analysis and optical microscopy have revealed the presence of two crystallographic phases of pyrrhotite, traditionally referred to as magnetic monoclinic 4C pyrrhotite and non-magnetic hexagonal NC pyrrhotite. However, ongoing research on pyrrhotite crystallography indicates that the latter NC pyrrhotite might have an orthorhombic or

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even monoclinic space group (de Villiers et al., 2009; Elliot, 2010; Liles and de Villiers, 2012). The crystallographic structures of pyrrhotite are not the main focus in this study and the traditional nomenclature of *monoclinic* and *hexagonal* pyrrhotite is kept.

Although it is known that sulphides reduce the brightness of the product, it is still of great importance to utilise the sulphide bearing marble, as the calcite content is >98%. It is estimated that this comprises approximately 10% of the marble deposit. In order to optimise the recovery from the deposit, it is essential to gain more knowledge on the colouring properties of these sulphides.

The objective of this paper is to determine how and to what extent the sulphides found in the Akselberg deposit affect brightness of the GCC slurry. It is of particular interest to determine the critical level of sulphides that will compromise product quality. By adding small amounts of pyrrhotite and pyrite to purified calcite concentrate, followed by micronisation and subsequent brightness measurements, the colouring effect of the respective sulphide phases is studied. Initial experiments to study the effect of particle size and the extent of surface oxidation have also been performed.

As the pyrrhotite and pyrite samples were retrieved from massive sulphide lenses in the Akselberg open pit deposit, the sulphide grains were not enclosed in calcite marble. Particle liberation analysis on the liberation between sulphides and calcite were therefore not required in this study.

2. Experimental

2.1. Origin of the samples

The samples in this study were collected from the Akselberg deposit. Calcite marble was handpicked, with the focus on obtaining the purest sample available. Respective samples of pyrrhotite and pyrite were handpicked from the massive sulphide lenses. The magnetite concentrate (Fe_3O_4) was supplied by Rana Gruber AS, Norway. A summary of the key mineralogical characteristics of the samples used in this study is given in Table 1.

2.2. The calcite marble

Crushing and dry milling of the sample was carried out using a jaw crusher with 3 mm opening, and a Retch laboratory beater wheel mill with a 250 μm sieve. Contaminating minerals were removed from the calcite marble through froth flotation with graphite and silicate collectors and water at 25 °C. The calcite concentrate was then dried for 7 min in a Panasonic microwave oven at 1800 W. To avoid overheating of the sample holder, the sample was repositioned in the microwave oven after 5 min. This

calcite concentrate represents a high quality GCC slurry and represents the uncontaminated reference material in this study. However, even in this product fine graphite inclusions can be found in the bulk of the calcite particles. The acid insoluble residue, found by dissolving the calcite concentrate in HCl, is on average 0.04 wt%. This residue includes graphite, silicates and sulphides, and the content of graphite in the calcite concentrate is therefore less than 0.04 wt%. The amount of graphite was too low to be detected by Leco analysis (detection limit 0.07%).

The chemical composition of the calcite concentrate was determined by X-ray Fluorescence (XRF), using a Bruker AXS S8 Tiger equipped with wavelength dispersive spectrometer. Element analysis were obtained using a pressed powder disk. The mineralogy of the calcite concentrate was then calculated from the XRF analysis. The results are shown in Table 1.

2.3. The sulphide and magnetite concentrates

The pyrite and pyrrhotite samples were crushed using a jaw crusher with 3 mm opening and milled using a Fritsch P6 Pulverizette planetary mono mill at 400 rpm for 60 s. All further sample treatment and processing were conducted on the 45–100 μm particle size fraction, which was obtained from the mill product by dry sieving. No further processing was conducted on the pyrite concentrate, whereas the pyrrhotite material was immediately separated into magnetic fractions using a Frantz LB-1 isodynamic laboratory magnetic separator. The separator was operated with a side slope of 20° and a forward tilt of 15°. The most magnetic material, separated at 0.001 A, was named *monoclinic pyrrhotite concentrate*, while the less magnetic material, retrieved between 0.02 and 0.1 A, was named *hexagonal pyrrhotite concentrate*.

The mineralogy of the three sulphide concentrates: pyrite, monoclinic pyrrhotite and hexagonal pyrrhotite were determined by XRD, using a Bruker D8 Advance diffractometer with a Kristalloflex K780 Generator, scanning from 2° to 80° (2θ) with monochromatised Cu $K\alpha$ radiation. Quantification with Rietveld refinement was carried out using Topas 4.2 software developed by Bruker AXS. The mineralogy of each concentrate is given in Table 1.

In order to limit oxidation, the sulphide concentrate samples were sealed in plastic bags and placed in a freezer immediately after milling (pyrite) or magnetic separation (pyrrhotite) until needed for the experiment. In addition, as a contrast to the unoxidised samples, a few highly oxidised samples were also produced. This was achieved by placing 1 g of each respective sulphide concentrates in separate petri dishes and exposing them to accelerated oxidising conditions in a heating cabinet at 45 °C for 5 days.

Table 1
Mineralogy of the concentrates.

Mineral (wt%)	Calcite concentrate	Monoclinic pyrrhotite concentrate	Hexagonal pyrrhotite concentrate	Pyrite concentrate	Magnetite concentrate
Monoclinic pyrrhotite		58.9	12.8		
Hexagonal pyrrhotite		37.3	61.4	<0.1	
Pyrite	<0.1			92.1	
Chalcopyrite			0.4	0.3	
Magnetite					94.0
Calcite	98.7	0.9	17.8	2.5	
Calcite, magnesian		0.5			1.5
Dolomite	0.5	0.3	0.4	1.0	
Chlorite	<0.1	0.4	1.9	<0.1	
Quartz	<0.1	1.1	2.8	1.3	4.5
Diopside		0.9	0.3	0.2	
Muscovite			0.2	2.5	
Actinolite		0.24	1.6		
Graphite	<0.04				

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