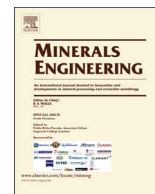




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## Application of Dynamic Vapor Sorption for evaluation of hydrophobicity in industrial-scale froth flotation

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## ABSTRACT

The particle surface properties are essential for understanding froth flotation, particularly for the evaluation of various chemical or reagent effects.

Dynamic Vapor Sorption (DVS) is used in the pharmaceutical industry for the evaluation of surface properties and has to the knowledge of the authors not been used for applications in mineral processing. This paper describes an evaluation of industrial ore samples using DVS.

Four samples (feed, CuPb concentrate, Cu concentrate and Pb concentrate) from each of the Cu – Pb flotation processes in the Boliden and Garpenberg concentrators, Sweden, were analyzed by DVS in order to investigate if this technique could be used to estimate differences in their hydrophilicity. The DVS measures the water uptake as a function of the relative humidity (%RH) at constant temperature.

For both series of four samples, it was found that the DVS-data are in precise agreement with the flotation theory on hydrophobicity (indicated by differences in water uptake). The feed material, without any collectors, adsorbed more water compared to the CuPb bulk concentrate, which in turn adsorbed more water than the Cu concentrate. The lead concentrate on the other hand, which had been depressed by dichromate and should be more hydrophilic, showed a higher adsorbance of water than that of the CuPb concentrate.

The repeated measurements of three sub samples from one of the ore samples gave a mean value and an estimated standard deviation of  $0.13 \pm 0.01\%$ . This shows that the method gives highly reproducible results and that the differences between the samples had high significance. This also shows that the DVS method can serve as a useful complement to traditionally used contact angle or capillary absorption-based measurement methods, especially when screening for new flotation reagents on industrial ore samples.

### 1. Introduction

In froth flotation crushed and ground mineral particles in aqueous suspension are mixed with one or more collectors. These are typically amphiphilic molecules with polar and non-polar end groups. Under the right conditions, the polar ends of the collector molecules can selectively bind to the surface of certain minerals, thus leaving them with a surface more or less covered with non-polar molecular tails. This renders these mineral particles more hydrophobic than mineral particles where no collector has attached. These differences in hydrophobicity between the different minerals forms the basis of the flotation process. The choice of collector reagent, together with the chemical conditions in the pulp, affects the degree of hydrophobicity on selected minerals (Wills, 1997).

In a flotation process, a number of flotation steps are utilized and in

the case where several valuable minerals have been floated and are to be separated from each other, a so-called depressant may be used. A depressant can act in two ways, first to selectively inhibit the adsorption of collector to ascertain mineral, or secondly to restore the hydrophilicity of particles which have floated in a previous step. Consequently, the depressed minerals will either go to the middling product for further processing or end up as a final concentrate. Both the action of the collector and depressant is strongly pH dependent. In this way, different minerals may be separated by manipulating their surface properties (Wills, 1997; Kawatra, 2011).

Dichromate is one of those depressant reagents, which can be used for the separation of copper and lead. The depressant is still used in the Garpenberg and Boliden concentrators, both owned by Boliden Mineral. The process employs bulk flotation of copper and lead with PAX (potassium amyl xanthate) and in a second stage galena is depressed by

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using sodium dichromate (Bolin et al., 2003; Bulatovic, 2007)

The problem with using dichromate is that it is both carcinogenic and an environmental hazard. Since ECHA (echa.europa.eu) has put dichromate on the authorization list, the ongoing research for finding a suitable alternative reagent has been intensified (Laskowski et al., 1991; Bolin and Laskowski, 1991; Javadi and Rao, 2016). The work described in this paper is a part of a larger research program included in studying the reason why dichromate is successful as a depressant of galena, which might aid in the selection of a similar reagent that works in the same way (Lundmark and Ymén, 2017).

In order to identify new suitable depressants for the inhomogeneous mineral samples collected in the concentrators, one way is to compare the hydrophobicity between two identical ore samples treated with different reagents. In practice, there are several ways to compare the hydrophobicity of related materials. Such methods typically entail measuring the contact angle either on a flat surface of a mineral or as capillary methods e.g. the capillary rise method on a compacted powder or the capillary pressure method, (Akdemir, 1997; Qiu et al., 2004; Chau, 2009; Iveson et al., 2000; Alghunaim et al., 2016; Iveson et al., 2004). However, these methods suffer from serious drawbacks, e. g. that the direct contact angle measurement must be made on a large, reasonably flat and preferably non-permeable surface. This is not possible on a mineral sample taken directly from a flotation process (Susana et al., 2012). The capillary methods on the other hand are sensitive to particle properties, such as size and shape distributions and surface properties. In addition, the methods require the use of packed mineral powder samples where the packing procedure is of importance and has been shown to affect the reproducibility (Susana et al., 2012; Teipel and Mikonsaari, 2004).

Other factors that affect the contact angle measurements could be the surface roughness, particle size or shape and the heterogeneity of the ore sample. The selection of liquids has also been shown to be sensitive to the equipment design (Teipel and Mikonsaari, 2004; Kirdponpattara et al., 2013).

In this work Dynamic Vapor Sorption, DVS (sometimes also referred to as Gravimetric Vapor Sorption, GVS) was used for characterization of wettability properties of minerals. The DVS method is an analytical method where the adsorption of water (or solvent) onto the particles, in the form of a powder, is measured as a function of the relative humidity (or the solvent partial pressure) at a constant temperature. In the pharmaceutical industry this technique has been used for a long time to measure the moisture sensitivity of active pharmaceutical ingredients, but has to the knowledge of the authors not been used for applications in mineral processing (Buckton and Darcy, 1995; Heng and Williams, 2011).

The purpose of this work was to investigate the effect of the depressant collector system on the surface properties of mineral samples. Four samples from different locations of the Cu/ Pb- flotation circuits of two concentrators were evaluated with the DVS method and compared with each other. The samples were also analyzed with XRPD and the particle size analyzed by laser diffraction for obtaining a better understanding of the results from the DVS measurement.

## 2. Materials and methods

### 2.1. Materials

Four pulp samples were collected from each of the flotation processes in Garpenberg and in Boliden. The samples were (a) ore feed to the 1st flotation step, (b) the CuPb concentrate, before Cu- Pb separation, (c) the Cu-concentrate, which floated in the Cu-Pb separation step and (d) the Pb concentrate, which was depressed with dichromate in the Cu-Pb separation step.

The samples were filtered and dried in an oven at 80 °C.

### 2.2. Methods

#### 2.2.1. Dynamic Vapor Sorption

A DVS-instrument is basically a very sensitive balance, with a sample cup and an empty reference cup, which are both flushed with an extremely well controlled moist gas stream. The %RH of the gas stream is obtained by careful mixing of two gas streams, one with 0 %RH and one with 100 %RH. The mass flow control meters of the two gases are extremely accurate giving very exact %RH-values. The %RH obtained is also checked with a dew-point sensor.

The instrument used in this paper is a Surface Measurement Systems DVS Advantage instrument, in which the balance can operate at a constant temperature between 5 and 60 °C. The temperature is controlled by keeping the whole assembly in a closed cabinet with very sensitive temperature sensors and heaters/coolers. It uses a sample amount between 1 and 150 mg (5–70 mg in this work) with a sensitivity of 0.1 µg and, if water is used, with a percent relative humidity between 0 and 98 %RH and an accuracy of ± 0.5 %RH.

The instrument was used to measure the water uptake as a function of the relative humidity at 25.0 °C. A % Partial Pressure Method was used. The sample weight was monitored while the sample was exposed to different relative humidities (%RH). The sample was first dried with dry nitrogen gas for 1 hour before the 1st cycle. The sample was then allowed to adsorb water in the 1st sorption/desorption cycle, where the %RH was increased stepwise up to 95 %RH (10, 20, 30, 40, 50, 60, 70, 80, 90 and 95 %RH) and then down to 0 %RH again. The sample was once again dried for 1 hour at 0 %RH, before another identical cycle, the 2nd sorption/desorption cycle, was run. In both sorption/desorption cycles, at each step, the sample was kept at the set relative humidity until  $dm/dt < 0.002\%$ , over a period of 5 min (a lower value of  $dm/dt$  will increase accuracy and lower the sample hysteresis, at the expense of increased measuring time).

#### 2.2.2. Particle size measurements by laser diffraction

The particle size distribution data for the collected ore sample were obtained with a Malvern Mastersizer 2000, equipped with a Hydro 2000S presentation unit. A sample RI of 2.000 was used as well as a dispersant Miglyol, with an RI of 1.449 and an absorption value of 0.05. Measurements were performed by adding each sample directly to the presentation unit, which was stirred at 2500 rpm. Three measurements were performed on each sampling and each ore sample was sampled four times, two with and two without prior ultrasonication. Each ore sample was thus measured 12 times. From the analyses mean values of different particle size parameters were calculated with and without ultrasonication.

#### 2.2.3. X-Ray powder diffraction (XRPD)

XRPD-analyses were performed at 22 °C on a PANalytical X'Pert PRO instrument, equipped with a Cu, long fine focus X-ray tube and a PIXcel detector. Automatic divergence and anti-scatter slits were used together with 0.02 rad Soller slits and a Ni-filter. Samples prepared at 22 °C were ground in an agate mortar and were then smeared out on cut Silicon Zero Background Holders (ZBH). In order to increase the randomness of the samples they were spun during the analysis. All samples were analyzed between 2 and 80° in 2θ. The full detector capacity of 256 channels was used and all samples were scanned continuously with a 2θ step size of 0.007° and a measuring time of 39.27 s per step.

## 3. Results and discussion

The XRPD-data for the ore samples are given in Figs. 1 and 2.

The data were matched against the JCPDS-database (PANalytical ICDD PDF-2 database). The minerals in Table 1 were found to match with the diffraction data. It should be noted that pyrite and sphalerite have X-ray peaks at very similar positions and are therefore difficult to separate, even when they are present in significant amounts. When

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