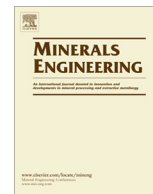




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Surface energy of minerals – Applications to flotation

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ABSTRACT

Inverse gas chromatography (IGC) is a versatile and powerful technique for characterizing physicochemical properties of materials. One such property, the surface energy, plays an important role in the adhesiveness, wettability, and consequently flotation of minerals. In this work, quartz was chosen as a naturally hydrophilic mineral, and dodecylamine was used to hydrophobise the surface. To study the correlation between the surface properties as measured from IGC experiments and flotation, microflotation experiments were also conducted. It was shown that with treatment by dodecylamine, the surface energy decreased, as did the work of adhesion to water. The recovery of quartz increased after the treatment, indicating a correlation between surface energy and flotation response.

The results indicate that IGC has the capability to determine the thermodynamic properties of quartz, pre- and post-conditioning. Relating the hydrophobicity and flotation of minerals to the surface energy can be observed from combining the results of IGC and microflotation experiments.

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1. Introduction

Surface energy measurements have attracted the attention of researchers in a wide variety of fields due to its importance in physical and chemical reactions.

Inverse gas chromatography (IGC) has been applied to the characterization of solid surfaces for more than 40 years (Lloyd et al. 1989). It is an analytical technique for evaluating physicochemical properties of materials, including surface energy. The most attractive advantage of the IGC method is its capacity to accurately characterize the surface and bulk properties of materials of various shapes such as powders, particulates, fibres (Thielmann 2004; Voelkel et al. 2009, 2012), and films (Santos et al. 2001; Surana et al. 1998; Gavara et al. 1997; Romdhane et al. 1995). The technique has been recently expanded to determine the surface energetic heterogeneity of surfaces (Gamble et al. 2013; Ali et al. 2013; Ho et al. 2012, 2010, 2009; Thielmann and Pearse 2002), which is a useful parameter for characterizing complex surfaces (Ylä-Mäihäniemi et al. 2008).

Mineral flotation is dependent upon the hydrophilic or hydrophobic characteristic of particles. Surface energies of both valuable minerals and gangue play an important role in the separation process. In other words, the nature and quantity of the excess energies

on the surface of components of ore has a decisive role in flotation process.

Contact angle measurements are one of the most widely utilized methods for determining surface energy of solids. However, the results obtained for powders are difficult to obtain compared to those of smooth, flat surfaces due to surface roughness, porosity and irregular shapes. A comprehensive review by Buckton (1990) discussed the limitations of various methods for measuring contact angle for powders. Hydrophobicity has also been determined through Washburn and Equilibrium Capillary Pressure (ECP) methods. However, particle size, packing time, and pore geometry can all affect these measurements, which are the limitations of liquid-based surface energy techniques. Parsons et al. (1992) outlined the inherent errors associated with contact angle measurements using liquid penetration. For example, determining the liquid penetration rate through the powder was suggested as the major source of error in liquid penetration experiments. Also, it was concluded that the error of the choice of perfectly wetting liquid with small contact angle was significant. Furthermore, wetting techniques typically only yield the average surface free energy. Additionally, the Washburn method, in which the capillary rise of various liquids is monitored, also fails because of absorption of many liquid probes into the bulk of the sample during the analysis (Segeren et al. 2002); this can be particularly challenging for polymers and other organic materials. However, since IGC is a gas-phase technique, it is not affected by the same surface roughness problems of contact angle methods and, the bulk absorption

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phenomenon can be minimized. Hydrophobicity of minerals has also been determined through contact angle measurements by use of Time of Flight Secondary Ion Mass Spectrometry (TOF-SIMS) (Brito e Abreu and Skinner 2012, 2011a,b, 2010, Priest et al. 2008). It is a destructive technique which causes some samples to show different characteristics after being analysed (Krebs 2012). For further information on the use of TOF-SIMS with regards to mineral processing, please see the review by Chehreh Chelgani and Hart (2014).

Determining the surface characteristics of minerals accurately leads to an understanding of the mechanism of interfacial behaviour. All of the above techniques have their merits and limitations in measuring the hydrophobic nature of particles. This paper discusses the applicability of IGC to investigate the flotation of particles.

2. Theory

The interfacial behaviour of a material is an important characteristic, controlled mainly by surface properties, one of the most important of which is the surface free energy. Thermodynamically, surface free energy of a solid is the required work to reversibly create a unit area of a surface. It is, in fact, an excess energy on the material surface which arises from fewer bonds between surface molecules comparing to bulk molecules. This energy is analogous to surface tension of liquids in which the atoms are able to move from the higher-energy surface to the lower-energy bulk, resulting in changes in the surface area. However, geometry and mechanical state of solids affect the apparent values of surface energy (Yudin and Hughes 1994) and therefore, makes its measurement more complex than that of liquid surface tension. The surface energy of solids plays a key role in the industrial processes that rely on wetting phenomena and coating process, such as polymers, pharmaceuticals, surfactants, and mineral processing. Understanding the surface characteristics of materials and the effect of the collectors and depressants on mineral surface chemistry is a necessity of modern flotation. In an aqueous suspension, different phenomena occur as a result of different interfacial reactions between molecules. For example, the wetting process occurs when the adhesion force between a solid and liquid is greater than the cohesion force between the liquid's molecules. The applicability of IGC to flotation was introduced by Ali et al. (2013). It was shown that IGC is a powerful technique for determining the surface energy of galena and quartz at different surface coverages; however, the effect of reagent addition was not investigated.

The surface energy of solids is defined as the sum of dispersive and specific (acid–base) components. The dispersive component, also called non-specific interactions, is attributed to molecular interactions due to London (van der Waals) forces. The specific component (also termed acid–base interactions) results from polar interactions such as hydrogen bonding and electron donor–acceptor behaviour (Voelkel 2012; Ylä-Mäihäniemi et al. 2008; Voelkel 1991). However, for a real solid, a single value is not considered as the energy of the whole surface of the solid; rather, there is a range of energetic sites of different levels. Altering the distribution of the energetic sites of higher and lower energy on a solid surface causes surface energy heterogeneity, which affects intermolecular interactions between surfaces. Different parameters related to surface heterogeneity have been studied, including the ratio of hydrophilic and hydrophobic sites on the surface (Kokkoli and Zukoski 2001); existence of hetero atoms and functional groups on the surface (László et al. 2001); also impurities, oxidized surfaces, and crystal orientation.

Inverse gas chromatography provides important information such as surface area, surface energetics, work of adhesion, and

surface energy heterogeneity. In this method, the sample is placed in a column (most commonly a glass tube) and characterized by passing solutes (called molecular probes) through the column. Adsorption and desorption phenomena occur at the sample which result in delayed exit of the probe from the column (retention). Therefore the fundamental data obtained from IGC is a plot of the detector signals as a function of retention time (Fig. 1). The retention time refers to the time the probe molecules require to generate a peak as a result of interactions with the stationary phase. Various characteristics of materials can be calculated by analysing the nature and quantity of these interactions.

The surface energy of a solid can be obtained from IGC by applying various relationships. The two commonly used approaches for characterizing surface energetics of materials are the Dorris–Gray and Schultz methods. Dorris and Gray (1980) developed their method based on the contribution of CH_2 groups in the free energy of desorption of n-alkane at zero surface coverage. Schultz et al. proposed a method for determining the dispersive component of a solid's surface energy on the base of applying n-alkanes as probe molecules at zero surface coverage (Schultz et al. 1987; Schultz and Lavielle 1989).

In this study, the Schultz approach was applied to calculate the dispersive component of the surface energy and specific free energies of quartz with different polar probe molecules. Eq. (1) shows the relationship between the different parameters:

$$RT \ln V_N = N_A a 2 \sqrt{\gamma_S^D \gamma_L^D} + C \quad (1)$$

where R is the gas constant ($\text{J mol}^{-1} \text{K}^{-1}$), T is absolute temperature (K); V_N is net retention volume (ml g^{-1}); N_A is Avogadro's constant (mol^{-1}), a is the cross sectional area of the probe molecule (m^2); and γ_S^D and γ_L^D are dispersive components of surface free energy of the solid and probe molecule (mJ m^{-2}), respectively. The constant C depends on the chosen reference. It was shown that the plot of $RT \ln V_N$ as a function of a $\sqrt{\gamma_L^D}$ for a series of n-alkanes is linear and the slope gives the dispersive surface free energy of the stationary phase (Fig. 2).

The specific free energies of desorption (ΔG) are determined by measuring the retention volume of polar probe molecules (e.g. toluene and dichloromethane) on the samples. Points representing polar probes are located above the alkane straight line (Fig. 2). The distance to the straight line is equal to the specific component

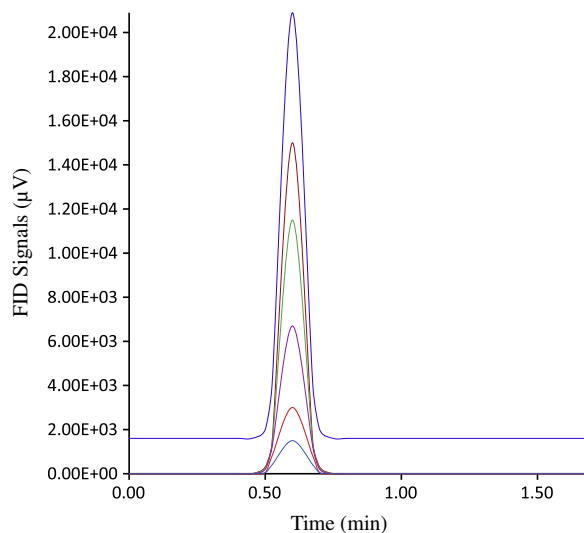


Fig. 1. IGC chromatogram of n-hexane adsorption on a quartz sample. The peaks correspond to different fractional surface coverages with the larger peak being the larger coverage (i.e. more probe vapour injected).

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