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Measurement of Colloidal Forces between Glass Sphere and Colemanite Surface in NaCl Aqueous Electrolytes

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Abstract - The main objective of the present work was to analyse the interactions of colemanite crystal surface with surrounding fluids and colloidal particles. Specifically, in this research, the interactions between colemanite surface and glass colloidal probe were studied using the atomic force microscopy (AFM). The colloidal forces were measured in $1.0x10^{-3}$ $molL^{-1}$ NaCl aqueous solutions at pH values from ~7 to 10 to explore a transition from attractive to repulsive forces when the surface charge potential for colemanite changes from positive to negative at isoelectric point (IEP) located near pH~10.2. As expected, at pH 10 when the colemanite and glass colloidal probe surfaces are similarly charged, a repulsive force was observed. On the other hand, at pH values of 7, 8 and 9 when the surfaces oppositely charged, attactive forces were observed.

Keywords: AFM (Atomic Force Microscopy), colemanite, crystal, colloidal forces

1. Introduction

Atomic force microscopy and its capability of adhesion and colloidal force measurements have become an important characterization device in numerous fields of fields such as surface science, materials engineering, chemistry, biochemistry, and biology. Additionally, the adhesion, colloidal, molecular and atomic forces that can be quantified with the AFM technique provide foundation for theoretical analysis and modeling of these forces [1-3].

Since 1989, several techniques of force value acquisition and analysis have arisen, and an increasing number of systems revealing unexpected forces have been analyzed. AFM force-distance curves are routinely used in several kinds of measurement including the determination of material's elasticity, material or system Hamaker constant, surface charge density, degrees of surface hydrophobicity, and many others [5]. Several previous studies demonstrated the capability of the AFM method to investigate dynamic force microscopy methods [4, 5], to examine the interfacial forces [6], and to quantify colloidal forces [3, 7]. In mineral processing, the colloidal force measurements provide a better understanding of the colloidal and molecular mechanisms of mineral-mineral and mineral-gas bubble interactions, often leading to better design of mineral processing technologies such as flotation process and selection of solution chemistry for treatment of minerals.

2. Background

2.1. Atomic Force Microscopy (AFM)

Atomic force microscopy (AFM) is an imaging technique used to determine topography and surface characteristics. It is also be used for force measurements such as electrostatic, adhesion, van der Waals, colloidal, capillary, magnetic and other forces.

AFM consists of a cantilever with a sharp tip or a colloidal probe at its end that is used to not only scan the surface but also measure the forces of interactions with a substrate in various environment. The cantilever with a sharp tip is typically silicon or silicon nitride with a tip radius of curvature on the order of nanometers.

When the tip is brought into proximity of a sample surface, forces between the tip and the sample lead to a deflection of the cantilever according to Hooke's law [4].

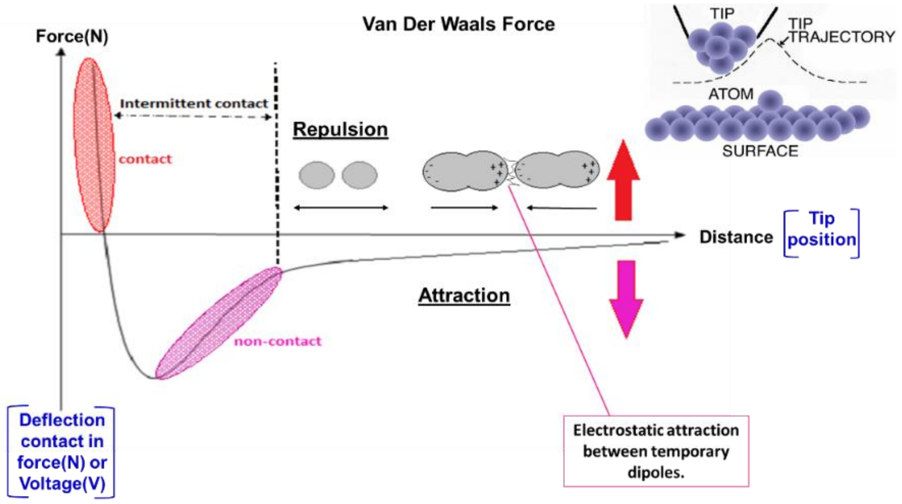

Figure 1. Schematic of a typical force-separation curve and parts of an approach-retraction cycle of the tip.

As shown in Figure 1, the AFM tip away from the sample, the cantilever is not affected by interatomic forces and keeps its equilibrium position. When it brought closer to the sample surface, attractive forces act upon the tip to bend the cantilever towards the sample [8].

But, when the tip is pushed towards the surface repulsive forces dominate, deflecting the cantilever backward. The thick lines indicate the normal ranges of operation for contact and non-contact modes and the long horizontal arrow represents the usual amplitude for intermittent contact [9].

2. Materials and Methods

2.1. Chemicals and devices

The colemanite sample was obtained from Eti Mine Works as lump-sized crystals from Kestelek deposit of Turkey which was cleaved 15×15×5 mm and polished for the AFM measurements. The glass probe (DNP-S10 model) was purchased from Bruker.

The experiments were performed using a Nanoscope E AFM (Digital Instruments Inc.) in a fluid cell (Figure 1). X-ray diffraction (XRD) measurements were carried out with Bruker D8 Discover diffractometer. All colloidal forces were measured in $1.0x10^{-3}$ $molL^{-1}$ NaCl aqueous solutions at pH values from ~7 to 10.

2.2. AFM analysis procedure

Interaction forces between glass colloidal probe (which used with 10-micron borosilicate glass) and colemanite crystal substrate at various pH values were measured by atomic force microscopy (AFM). All of the force curves were analyzed with the SPIP software (Image Metrology, Lyngby, Denmark), which translates the cantilever deflection-piezo extension/retraction data to force-separation profiles.

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3. Results and Discussions

In this study, we studied to analyse the interactions of colemanite crystal surface with surrounding fluids and colloidal particles.

Before force measurements topographic AFM images of the tested colemanite surfaces presented in Fig.2.

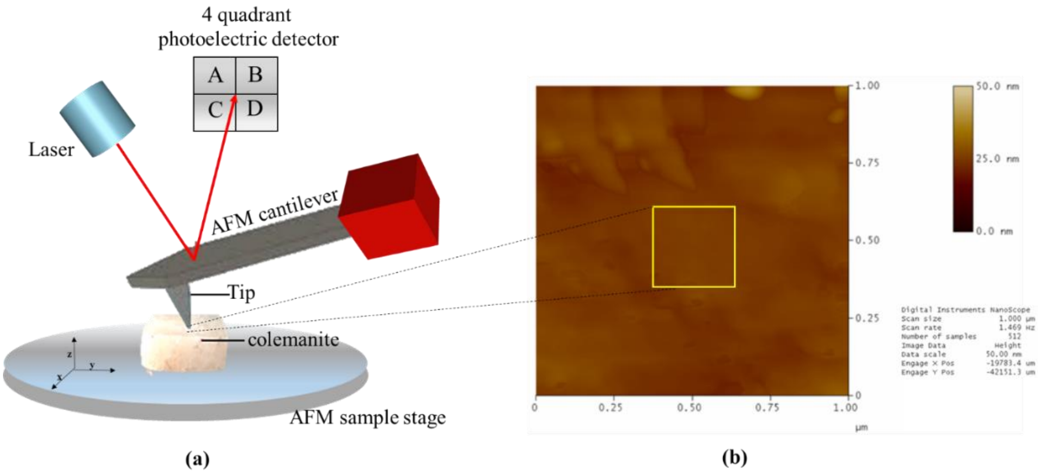

Figure 2. (a) Schematic diagram of AFM (b) 3D AFM height images of colemanite crystal with a smooth surface.

As shown in Fig. 2 (a) the sharp tip scans over the colemanite surface and the deflection of the cantilever is quantified through a laser beam reflected off the backside of the cantilever and received by the photoelectric detector. Finaly, the measured cantilever deflection allows the computer to generate a map of the surface topography [10].

The obtained topography image corresponds to the measured height values, z(x,y) (Figure 2(b)) The height value is associated to a pair of surface coordinates (x,y) [11]. XRD patterns of colemanite is shown in Fig. 3.

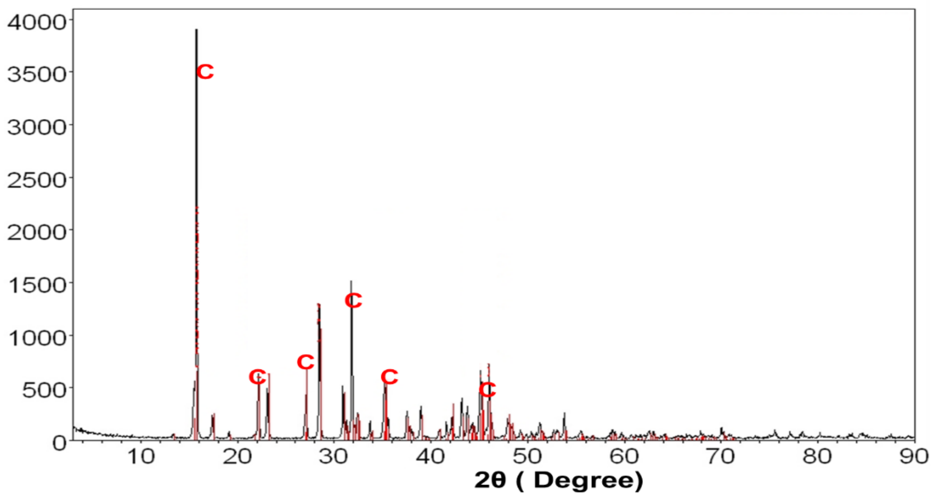

Figure 3. X-Ray diffraction (XRD) patterns of the colemanite.

As shown in Fig. 3 the expected peaks for colemanite, revealing that the sample has a high purity.

The colemanite is a semi-soluble mineral. For this reason, it has a low solubility in water, but it dissolves readily in acid [12]. The colemanite cannot be practically measured below pH 6 due to dissolution of mineral in HCl. In order to obtain reliable AFM results, all measurements in should be either range from pH 6 to 11.

Figure 4 shows the colloidal forces measurements between glass probe and colemanite crystal. It can be seen this figure that interaction forces undergo a change in four different pH values.

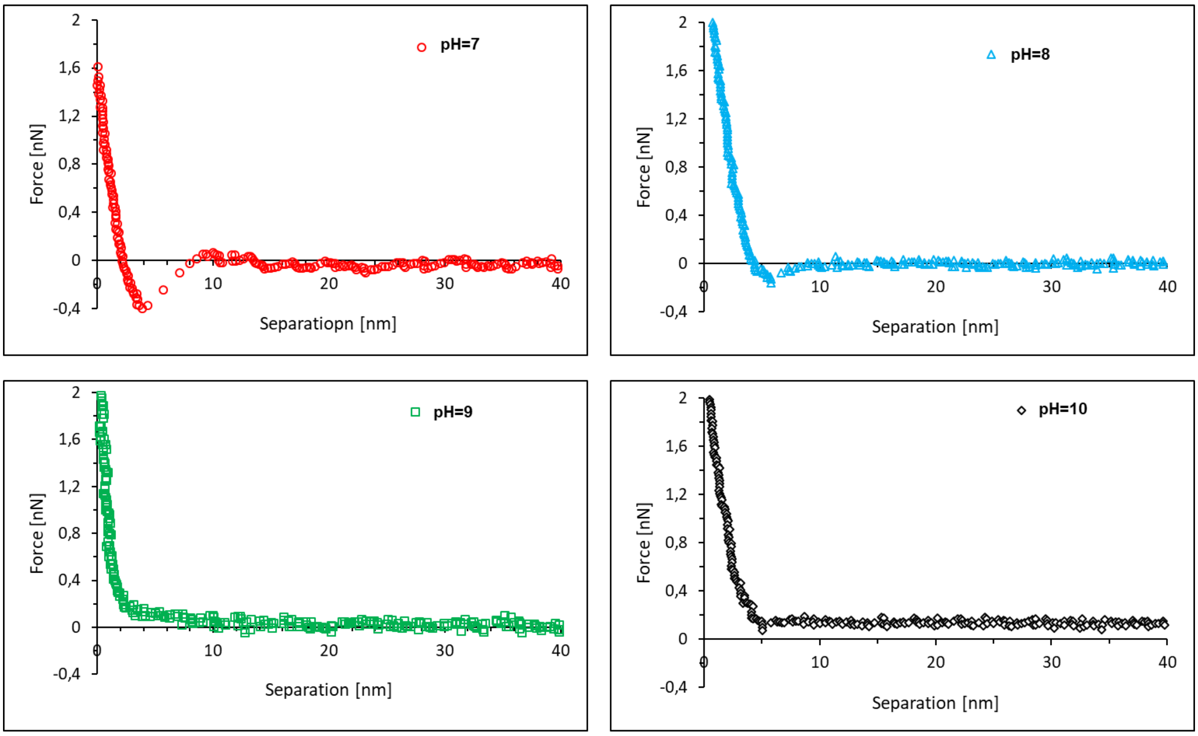

Figure 4. The force versus separation curves obtained for a glass probe tip interacting with Kestelek crystals in $1.0x10^{-3}$ $molL^{-1}$ NaCl suspension at 4 different pHs.

The point of zero charge (PZC) of colemanite is about range of 10.2-10.5 [13]. As expected attractive interactions between negatively charged glass probe and positively charged colemanite crystal dominate at pH 7 and 8. These attractive forces weakened at pH 9 and changed to repulsive at pH 10. The transition from attractive to repulsive is associated with reversal of the surface charge for colemanite crystal from positive to negative at pH 10.

4. Conclusions

The colloidal forces between colemanite crystal and glass probe were investigated by AFM. In $1.0x10^{-3}$ $molL^{-1}$ NaCl at pH 7, attractive forces have been recorded between the AFM glass probe and colemanite surface. Attractive electrostatic forces result from the interaction between the negatively charged glass probe and positively charged colemanite surface. In the alkaline solution of NaCl pH 8 and 9 weak attractive forces were observed while pH 10 only repulsive forces were observed between the AFM tip and colemanite crystal. As a result, at the pH values (7, 8, 9 and 10) where the AFM measurements were conducted, the nature of the interaction forces depends on the surface charge of colemanite.

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