

Exploring mechanism of xanthate adsorption on chalcopyrite surface: An atomic force microscopy study

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Abstract

In this work, adsorption of the potassium amyl xanthate collector on the pure chalcopyrite surface was studied by applying atomic force microscopy (AFM). The adsorption experiments were carried out at different concentrations of the collector and at diverse pH values in the presence or absence of exterior ions. The changes occurring in the surface morphology of chalcopyrite due to the collector adsorption were evaluated by measuring the contact angle of the collector and its surface coating. According to the 3D images obtained by AFM, an increase in the pH value from 7.5 to 9.5 at two concentrations of 25×10^{-3} and 50×10^{-3} g/ton of the collector would increase the number of particles adsorbed on the surface, improve the adsorption morphology, and reduce the contact angle. Moreover, at a constant pH value, increasing the collector would result in the proliferation of contact angles as well as a relative increase in the number of particles. By comparing the morphological surface changes in the tap and distilled water samples, applying tap water, owing to the presence of Cu^{2+} ions and activation of the surface through the production of CuS, the quality and quantity of adsorption would be increased. The use of tap water not only can account for an appropriate coating by the collector but also causes to reduce the consumption of collector for at least 50%.

Keywords: *Morphological Changes, Potassium Amyl Xanthate, Chalcopyrite, Atomic Force Microscopy.*

1. Introduction

As a physio-chemical process, flotation is an extremely useful way to separate the minerals with different surface properties [1, 2]. In this regard, chemicals such as collectors play a significant role in the flotation process. In the chalcopyrite flotation, collectors are usually added into the pulp to have a selective adsorption onto the target mineral and render its surface with high hydrophobicity [3] which is generally beneficial for a strong mineral-bubble attachment and a high flotation recovery [4]. The adsorption of the collector on a mineral surface is vital for a successful flotation process and it has been studied by applying various surface analysis techniques, such as IR (Infrared Spectroscopy), CV (Cyclic Voltammetry), and XPS (X-ray Photoelectron Spectroscopy) [5]. These studies have revealed a lot of information such as the reactions, products, and mechanism of the

adsorption of chemicals on the mineral surface. According to the surveys, the collector adsorption on the mineral surface has been studied through the direct and indirect methods. It is also of great interest to get directly the image of the collectors adsorbed on mineral surfaces under different conditions of pulp chemistry such as pH, redox potential, ionic strength, and chemical dosage. To this end, researchers have applied scanning tunnelling microscopy (STM) to study the changes, i.e. oxidation, reaction, and adsorption of galena surface under flotation conditions [6]. By applying the AFM method to study the adsorption of chemicals on the mineral surface, Zhang et al. [7] have reported that mineral surface morphological changes are not due to the oxidation of the mineral surface in distilled water. Rather, it occurs due to the existence of collectors. Later, they compared the effect of adsorption of

several different collectors on the chalcopyrite surface by applying the AFM imaging techniques [6]. The results showed that the potassium amyl xanthate (PAX) collector had a better performance for adsorption on the chalcopyrite surface in comparison with potassium ethyl xanthate. Unlike scanning electron microscopy (SEM), which is only capable of visualizing 2D images, the AFM imaging technique can magnify the sample surface a million times and provide 3D images [8]. This device evaluates the surface of the sample using a sharp probe with two microns long, less than 10 \AA in diameter. This probe is located at the free end of the wing with 100 to 200 micrometers long. For imaging, the reaction of the probe to the forces that the sample imposes on it can be used to create an image of 3D shape (topography) of a sample surface at a high resolution [9, 10]. This is achieved by raster scanning the position of the sample with respect to the tip and recording the height of the probe that corresponds to a constant probe-sample interaction. Thus the data is transferred to the computer and is converted to an image [9]. AFM has some advantages over other surface characterized techniques. By employing an AFM fluid cell, it is possible to study the *in situ* adsorption of chemicals in the solution. On the other hand, some other analysis techniques, with the prerequisite of taking the solid sample out of the solution, may not collect the actual adsorption information because the adsorption usually changes when the solid sample is removed from the solution [11]. Obviously, the information that is obtained in this way has many errors. Similarly, unlike methods such as STM, the AFM imaging method does not require a vacuumed environment [6], nor does the conductivity or the semi-conductivity properties of the sample in the AFM method matter. Although many studies have been carried out on the collector adsorption, there have been a few research works on the investigation of

changes in mineral surface morphology by the new AFM method under various chemical conditions and direct observation of the coated collector in the 3D area on the mineral surface. Therefore, in this work, we investigated the effect of the collector dosage on the pH and chalcopyrite surface morphology using the AFM methodology.

2. Materials and method

2.1. Materials

A high-purity chalcopyrite (CuFeS_2) sample (about 98%) was obtained from the Mazraeh mine copper deposits, located in the East Azerbaijan province (Iran). The sample was finely polished, and further cleaned by rinsing thoroughly with ethanol and water. A $3 \text{ cm} \times 3 \text{ cm}$ sample was used for the surface characterization experiments. In order to perform the sample mineralogy, an X-ray diffraction device (XRD), model PW 1800, Philips Company (Netherlands), was used. The diffraction angle (2θ) was scanned from 10° to 80° , and the scan speed was two degrees per minute. The XRD analysis was performed at 40 kV and 40 mA current. The XRD pattern and the chemical composition of the material are shown in Figure 1 and Table 1, respectively. Both sodium hydroxide and sulfuric acid (0.1 M) supplied from the Merck Corporation (Germany), were employed to adjust the pH, and PAX with the molecular weight of 203.57 mol/g was applied as a collector [9, 12]. In some experiments, pure water was deionized twice by the distillation device SDL 12L, supplied from the OES Company (USA), and in some other experiments, tap water was utilized [8, 13]. According to the chemical analysis results shown in Table 1, there were some impurities in the chalcopyrite sample. However, they were not observed in the XRD pattern due to their small amounts. As shown in Figure 1 and Table. 1, the chalcopyrite sample was highly pure.

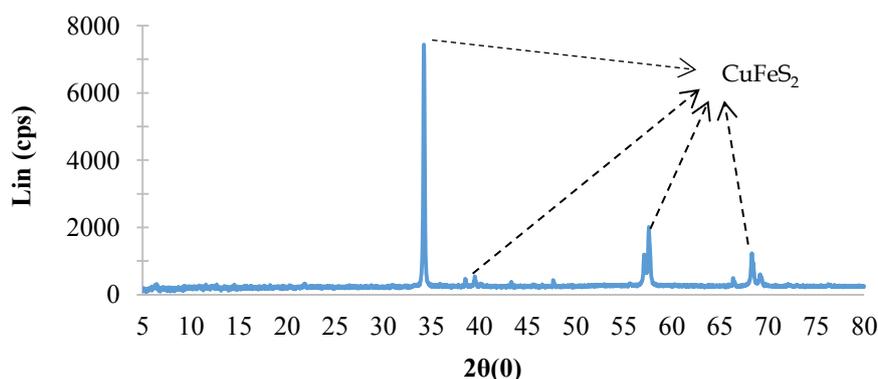


Figure 1. XRD pattern for chalcopyrite crystals.

Table 1. Sample chemical composition (mass fraction, %).

| Cu | Fe | S | SiO ₂ | CaO |
|-------|-------|-------|------------------|------|
| 34.08 | 28.22 | 34.91 | 1.49 | 1.30 |

2.2. Method

The chalcopyrite sample was mounted in resin. In order to prepare the sample for studying, a sandpaper (300 mesh and up to 4000 mesh) was employed. The final polished sample was a 3-cm diameter cylinder with a height of 3 mm. In order to prevent oxidation of the sample surface, it was kept in a bag containing pure nitrogen at -23 °C. Prior to each experiment, the surface of the sample was first washed with 98% ethanol and then with distilled water, and was placed in the AFM device reservoir. As a reference image and for ensuring the smoothness of chalcopyrite, a preliminary image was taken from the sample surface in distilled water at an approximate pH value of 6. During the AFM imaging study, PAX was flushed through the AFM fluid cell, and the AFM measurement was commenced after the chalcopyrite plate contacted the chemical solution for 15 minutes [14]. Imaging of the sample

surface was performed by the AFM device, supplied from the J. P. K. Corporation (Germany). This device was installed on an optical microscope obtained from the Olympus Company (Japan). Silicon nitrate cantilevers with the spring constants of 12-58 n/m were obtained from the ApNano Corporation (USA). The experiments were carried out at different pH values and collector concentrations, and in both the presence (tap water) and absence of external ions (distilled water). The conditions for conducting various experiments are shown in Table 2. It should be noted that during imaging, high sounds, magnetic field, electronic device signals, and turning on some electronic devices such as electrospinning machine (used for producing nanofibers) generate noise, and result in errors. Therefore, some efforts were made to remove or reduce these noises during image analysis. The AFM images reported in this communication include both height and deflection images obtained in the contact mode. All the images in the form of 2D, cross-sectional, and 3D images were obtained by scanning a mineral surface in a 10*10 μ m area.

Table 2. Collector adsorption experiments on chalcopyrite surface in different conditions.

| Experiment number (N) | pH value | Collector concentration (g/ton) | External ions |
|-----------------------|----------|---------------------------------|---------------|
| 1 | 7.5 | 25*10 ⁻³ | Absence |
| 2 | 9.5 | 25*10 ⁻³ | Absence |
| 3 | 7.5 | 50*10 ⁻³ | Absence |
| 4 | 9.5 | 50*10 ⁻³ | Absence |
| 5 | 7.5 | 25*10 ⁻³ | presence |
| 6 | 9.5 | 25*10 ⁻³ | presence |

3. Results and discussion

3.1. Initial surface image

Figure 2 shows a 3D image of the chalcopyrite surface, washed with ethanol and distilled water before contacting with the collector at pH 6. According to this image, no collector particle was observed on the chalcopyrite surface in this condition.

3.2. Effect of pH in different collector concentrations

3.2.1. Effect of pH in collector concentration of 25*10⁻³ g/ton

Figure 3 (1) shows a 2D image of the PAX particles adsorbed on the chalcopyrite surface at pH 7.5 in distilled water. A part of this figure shows a cross-section of the sample surface. The AFM software output data is both the length (average diameter) of the collector particles adsorbed on the chalcopyrite surface and the highest difference in height of these particles.

Using this data, the contact angle of the adsorbed collector on the chalcopyrite surface can be measured. The contact angle is the angle between the collector particle and the solid. A small contact angle indicates that the adsorbed collector is flat. In contrast, a high contact angle shows that the adsorbed collector is sharp [15]. A flat adsorption of the collector on the chalcopyrite surface leads to a better and more complete coverage. If in the shear diagram X = length and Y = height difference, the collector angle with the chalcopyrite surface is determined by Equation 1.

$$\theta = \tan^{-1} \frac{y}{x} \quad (1)$$

The section analysis of Figure 3 indicates that the radius of a medium size patch is 215.1 nm and the height of the cap is 38.3 nm. The angle formed by the radius and the height is 10°, suggesting that the collector adsorption on the chalcopyrite

surface is not completely flat. Figure 4 (1) depicts the morphology of the collector adsorption on the surface. According to this figure, at neutral pH (7.5) and a collector concentration of 25×10^{-3} g/ton, the amount of collector coating is not appropriate on the surface, and it is mainly seen as peaks and sharp tips. Figure 3 (2) shows the cross-sectional graph of adsorption of the PAX collector at a concentration of 25×10^{-3} g/ton on the chalcopyrite surface with an alkaline pH of 9.5 in distilled water. The section analysis of Figure 3 (2) shows that the radius of a medium size patch is 616.4 nm and the height of the cap is 47.8 nm. The angle formed by the radius and the height is 4.5° , suggesting a quite flat patch. In the 3D image of the sample surface, as shown in Figure 4 (2), the collector adsorption morphology has

progressed toward larger size and more spherical particles. The appearance of wider particles is in virtue of the increase in pH. A small adsorption indicates a lack of sufficient preparation time, which leads to an incomplete growth. Also, in the alkaline condition, the number of collector particles increases. These particles are in a quite good spherical and large shape, and furthermore, the surface coating is much better than neutral pH. As a result, at a collector concentration of 25×10^{-3} g/ton in distilled water, by changing the pH from neutral to alkaline, a greater amount of chalcopyrite is coated by the patches. Accordingly, the conditions for connecting the air bubbles to the hydrophobic chalcopyrite surface will be more appropriate.

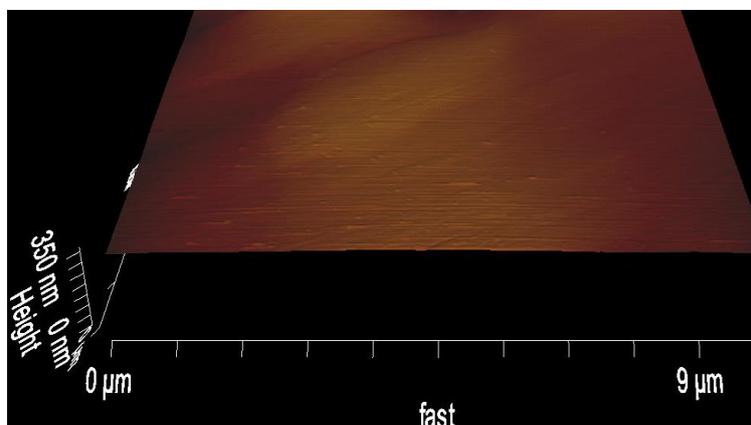


Figure 2. AFM image of the chalcopyrite surface before preparation by the collector.

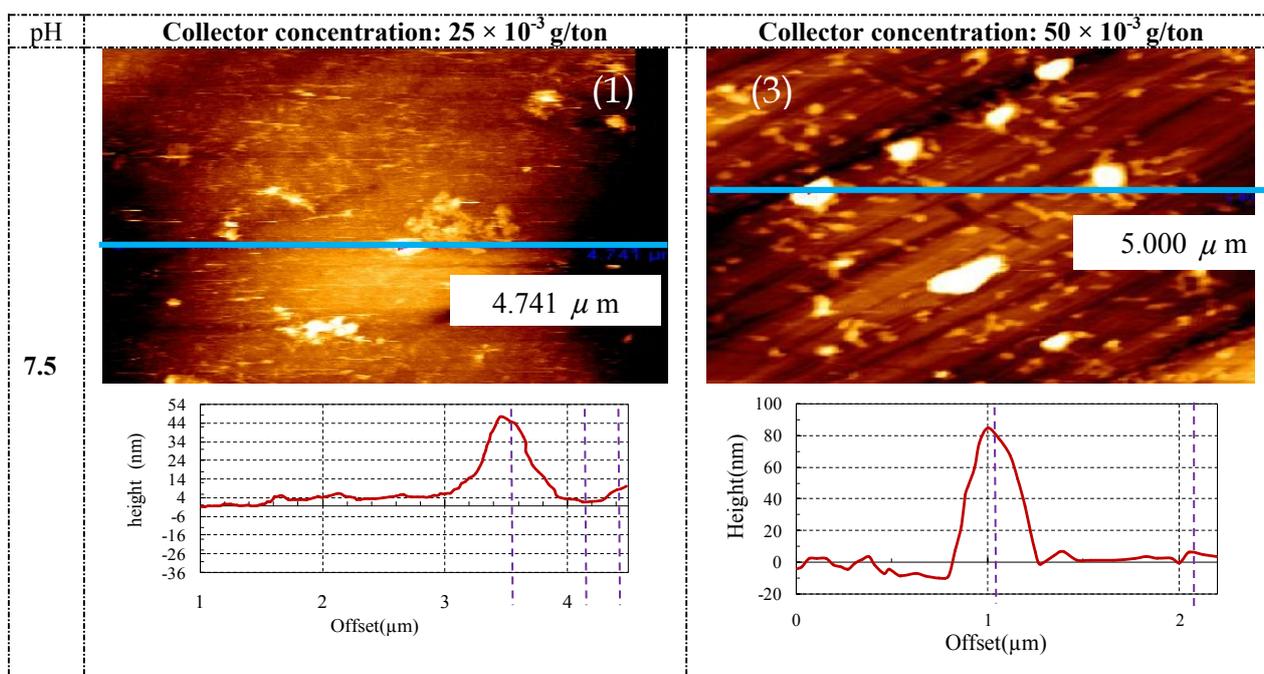


Figure 3. 2D images (up) and cross-sectional diagrams (down) of the chalcopyrite surface at different pH values and collector concentrations in distilled water.

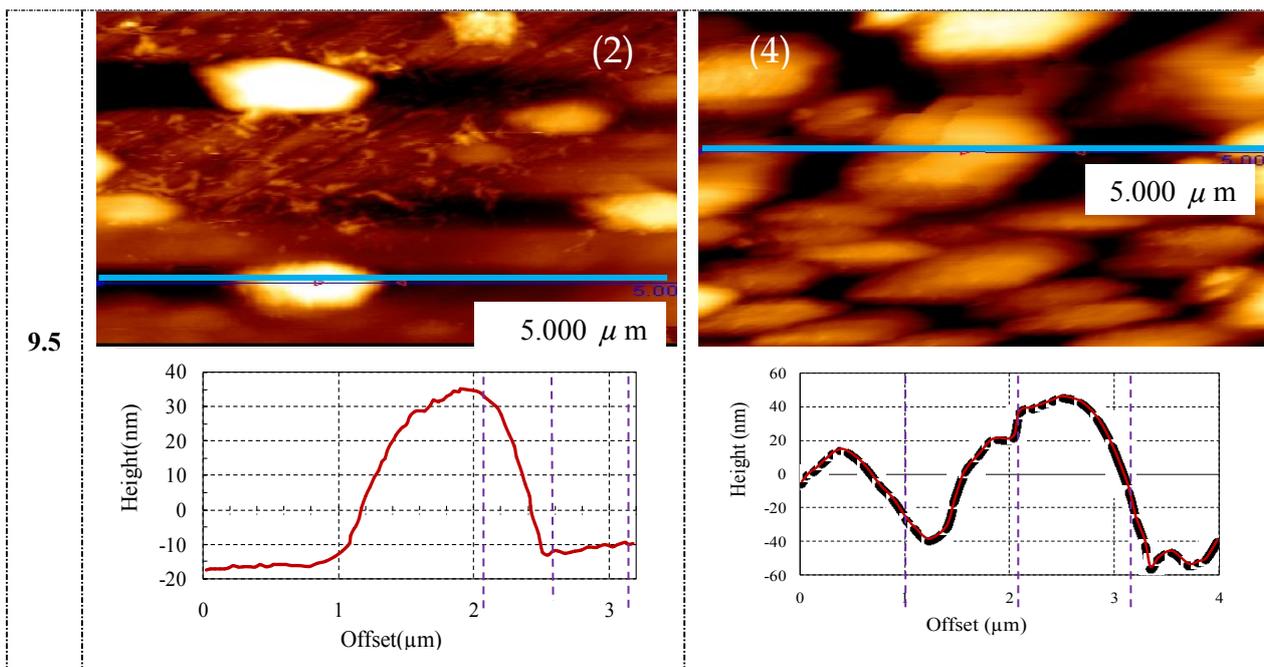


Figure 3. Continued.

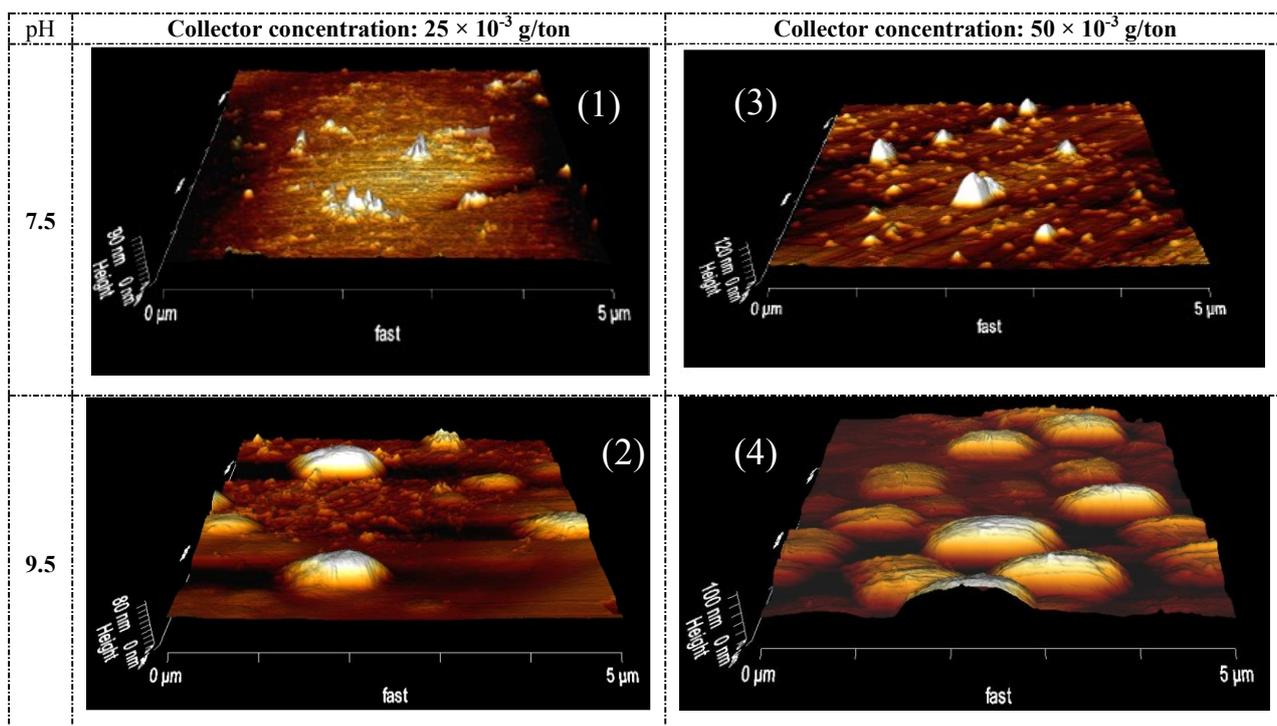


Figure 4. AFM 3D images of chalcopyrite surface at different pH values and collector concentrations in distilled water.

3.2.2. Effect of pH collector concentration of 50×10^{-3} g/ton

The effect of pH on the adsorption morphology of the amyl xanthate collector on the chalcopyrite surface in a concentration of 50×10^{-3} g/ton was also studied. The section analysis of Figure 3 (3) at neutral pH of 7.5 shows that the radius of a medium size patch is 235.4 nm and that the height of the cap is 84.4 nm. The angle formed by the

radius is 19.7° . In addition, the 3D image of the sample surface, as shown in Figure 4 (3), indicates that there are few absorbed particles with large contact angles. Figure 3 (4) shows a 2D image and a cross-sectional graph at a concentration of 50×10^{-3} g/ton in alkaline distilled water (pH = 9.5). The section analysis of Figure 3 (4) indicates that the radius of a medium size patch is 928.9 nm and the height of the cap is 98.1 nm. The angle

formed by the radius and the height is 6° , suggesting a quite flat patch. It can be observed that there are a few large spherical collector particles. Thus, the adsorption collector morphology on the chalcopyrite surface is suitable. At a collector concentration of $50 \cdot 10^{-3}$ g/ton in distilled water, increasing pH from 7.5 to 9.5 accounts for a higher adsorption, improving morphology, and reducing the contact angle (due to the flatness of the collector coating particles). In a like manner, at a collector concentration of $25 \cdot 10^{-3}$ g/t in distilled water, increasing pH from 7.5 to 9.5 does so. The high contact angle (vertical adsorption) in the neutral state causes the instability of the adsorbed particles on the surface. By increasing pH to 9.5, the collector particles become more spherical. Thus, a better coverage happens. In neutral pH, by increasing the preparation time, there is the possibility to reduce the contact angle and increase the collector surface coating. This phenomenon has been proven by other researchers [6]. Nevertheless, increasing the preparation time increases the flotation time and reduces the cost-effectiveness of the process. Based on the results obtained, the contact angle at a constant pH (whether 7.5 or 9.5) by increasing the collector concentration from $25 \cdot 10^{-3}$ g/ton to $50 \cdot 10^{-3}$ g/ton in distilled water has increased despite a more collector adsorption. This phenomenon indicates that increasing the collector concentration at a constant pH, mainly by forming the second and subsequent collector layers through the bond between non-polar hydrocarbon chains, leads to increasing growth and adsorption of the collector in a vertical direction instead of adsorption on the mineral surface. As a result, the contact angle will increase, while at a constant collector concentration (whether $25 \cdot 10^{-3}$ or $50 \cdot 10^{-3}$ g/ton), increasing the pH value causes both the reduction of contact angle (improvement of adsorption morphology) and the increase in the collector adsorption. It can be concluded that increasing the collector concentration is not the only factor involved to achieve a complete coverage but pH of pulp also affects the adsorption process. Different hypotheses have been suggested by the researchers to explain the effect of pH on the xanthate adsorption. Some researchers have reported that chalcopyrite flotation at pH between 2 and 13 is independent from changes in pH [16]. The presence and formation of elemental sulfur in acidic solution cause chalcopyrite to be hydrophobic. It has been reported that the chalcopyrite recovery will be increased by a

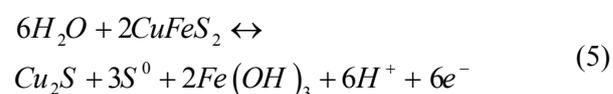
decrease in pH from 5 to 10.5 [14]. There is no investigation that reports the effect of pH on the quantity and types of PAX adsorption of chalcopyrite surface. Adsorption happens through three methods, either chemically adsorption (chemisorption) or physically (physisorption) or by complexing with atoms that solubilize somewhat at the surface (surface precipitation) [9, 17].



where X represents the xanthate molecule. Equations (2) and (3) refer to the surface precipitation and physisorption, respectively. The chemical adsorption happens as follows:



Adsorption of the xanthate collector on the chalcopyrite surface at pH values of 7, 9, and 11 has been studied by the voltammetric diagrams [9]. The results obtained from the voltammetric diagrams have shown that at pH values of 7 and 11, the CVs in the presence and absence of xanthate are the same, illustrating that xanthate chemisorption is indeed absent [18]. However, at pH 9, there appears to be a significant current flow in both the absence and presence of xanthate. According to the Eh-pH diagram (Figure 5), at -0.05 V, $CuFeS_2$ is stable at pH 7 (no current is observed), Cu_2S is stable at pH 9 (current is observed), and $CuOH$ is stable at pH 11 (no current is observed). According to the researcher's results, clearly, xanthate chemisorption does not occur at pH values of 7 and 11. This suggests that xanthate chemisorption just occurs on the Cu_2S surface, even if it is an oxidation product of chalcopyrite [19, 20]. This reaction is shown in Equation 5.



Based on the results obtained from the AFM image analysis in this work, increasing the absorbance of the collector and reducing the contact angle of the collector/surface at alkaline pH of 9.5 in comparison with 7.5 is attributed to the collector chemisorption, while at pH 7, the adsorption of xanthate is in the form of surface precipitation of CuX on the chalcopyrite surface. The chemical adsorption is far more stable and better than the surface precipitation [21].

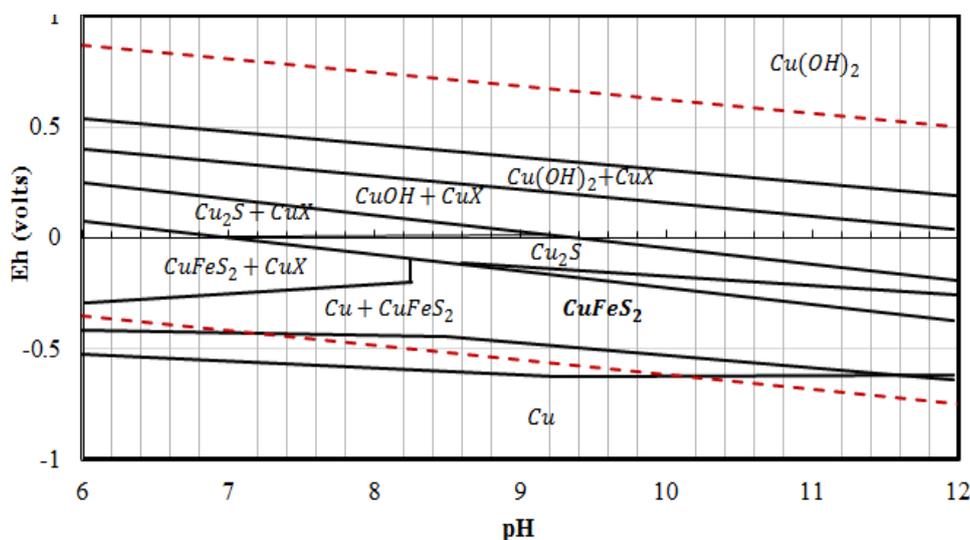


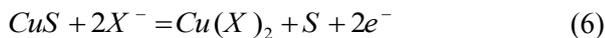
Figure 5. Eh-pH diagram for chalcopyrite/xanthate/water system [17].

3.3. Effect of exterior ions

In order to study the effect of the exterior ions on the adsorption of the xanthate collector, tap water was employed. Tap water was analyzed using atomic absorption spectroscopy. This analysis indicated the presence of Cu^{2+} , Pb^{2+} , Mn^{2+} , Fe^{2+} , and Zn^{2+} ions (Table 3). Figure 6 (1) shows a 3D image of the chalcopyrite surface with a collector concentration of 25×10^{-3} g/ton in tap water and at a pH of 7.5. Coating the surface in comparison to distilled water in a similar situation is more appropriate. In this case, the collector angle was calculated to be 6° , while under the same conditions, with distilled water, the contact angle was 10° and the collector coating was poor (Figure 6 (2)). Smaller contact angles and better collector coverage in the presence of ions improve the morphology of the collector adsorption on the surface in comparison with distilled water. Figure 6 (3) shows a 3D image of the chalcopyrite surface at alkaline pH of 9.5 and collector concentration of 25×10^{-3} g/ton in tap water. According to this image, the collector adsorption has been increased spherically and coarse, and the collector coating has significantly increased. In this case, the contact angle was calculated to be 3.2° , suggesting a quite flat patch and no sharp peak. Reduction of the contact angle at alkaline pH of tap water addition, the significant increase in the collector coverage on the chalcopyrite surface indicates an improvement in the collector adsorption morphology in the presence of exterior ions in comparison with distilled water. By examining the contact angles obtained from the tap and distilled water samples at neutral and alkaline pH values, it can be concluded that when tap water is employed, the collector contact angle

decreases from 28% to 34%, indicating the progression of morphology. According to the results obtained from the experiments by distilled water, the best morphology of the collector adsorption of PAX was gained at a collector concentration of 50×10^{-3} g/t and a pH value of 9.5. By doing so, the contact angle of the collector and the chalcopyrite surface was calculated to be 6° , while in tap water, the collector adsorption in a better coverage was only obtained at 25×10^{-3} g/t through the same pH value. Therefore, consumption of the collector decreases up to 50% by applying tap water. According to the atomic absorption spectroscopy, one of the important cations present in tap water is Cu^{2+} . Activation of the surface by the cation was investigated in relation to sphalerite, pyrite, and other minerals. However, further research works are required in this regard. This cation enters the pulp by surface solubilization of sulphide metal minerals and water. The effect of copper cation activation on collector adsorption, surface properties, and zeta potential have been investigated. However, its effect on the collector adsorption rate has not been studied directly. As shown in Figure 7, when $\text{pH} \leq 7.5$, the soluble copper is predominantly a cation of Cu^{2+} with minor amounts of $\text{Cu}(\text{OH})^+$ and $\text{Cu}(\text{OH})_2$. However, in the range of $9 \leq \text{pH} \leq 11$, copper is mostly stable as $\text{Cu}(\text{OH})_2$; in this case, $\text{Cu}(\text{OH})_2$ is more than 95%. The effect of copper ions on the adsorption of sulfide minerals depends on the environmental pH. In an acidic environment, a substitution reaction takes place between copper ions and the surface of sulfide minerals. In contrast, at neutral and alkaline pH values, copper ions are absorbed or deposited in the form of copper hydroxide on the surface to

produce CuS. The presence of CuS on the chalcopyrite surface leads to an increase in the adsorption of the xanthate collector [22-24]. The formation of CuS is as follows:



This phenomenon reduces the contact angle and improves the adsorption morphology of the collector on the chalcopyrite surface in the

presence of copper(II) cations. It seems that the other cations present in tap water, i.e. Fe^{2+} , Mn^{2+} , Zn^{2+} , and Pb^{2+} cause an increase in the collector adsorption through this mechanism [25].

Table 3. Chemical composition of tap water (ppm).

| zn | Pb | Mn | Fe | Cu |
|------|------|------|------|------|
| 0.03 | 0.01 | 0.02 | 0.03 | 0.09 |

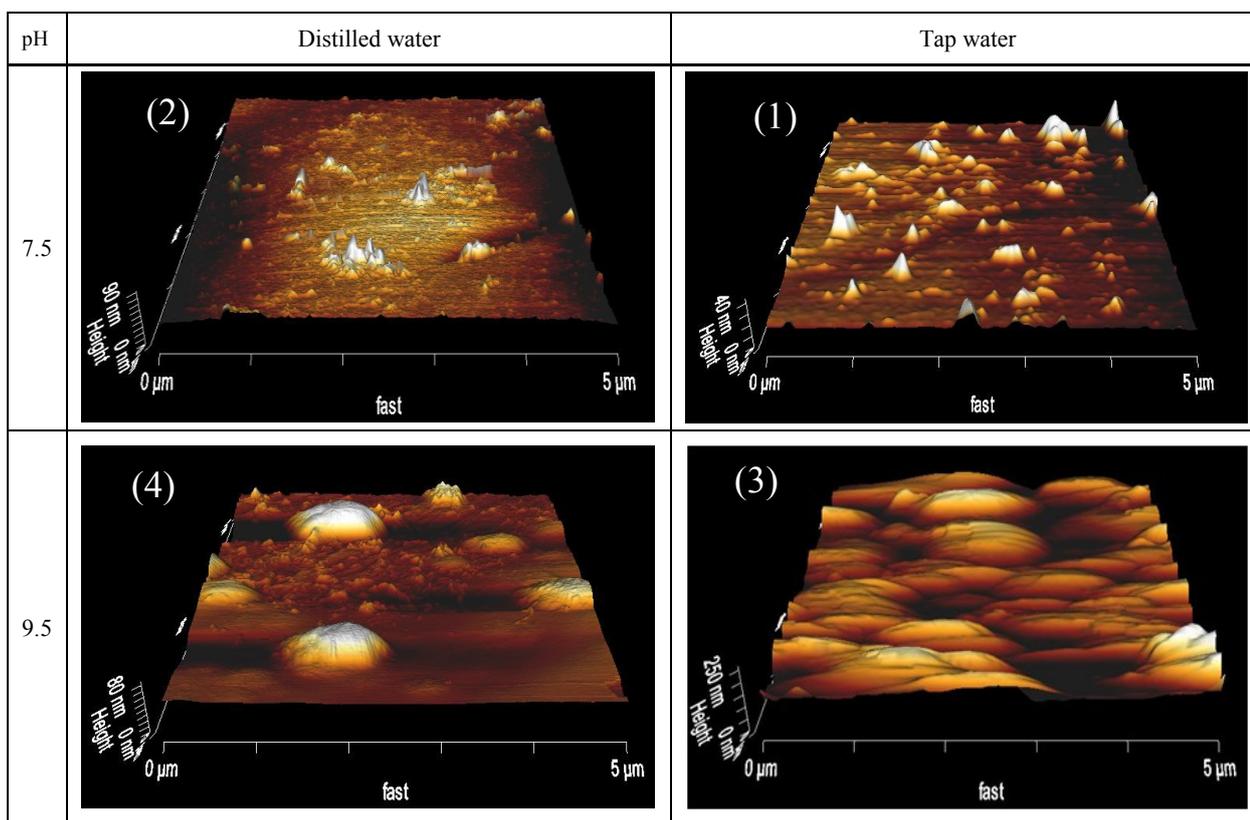


Figure 6. Comparison of morphological changes in both tap water and distilled water at a collector concentration of $25 \cdot 10^{-3}$ g/ton at various pH values.

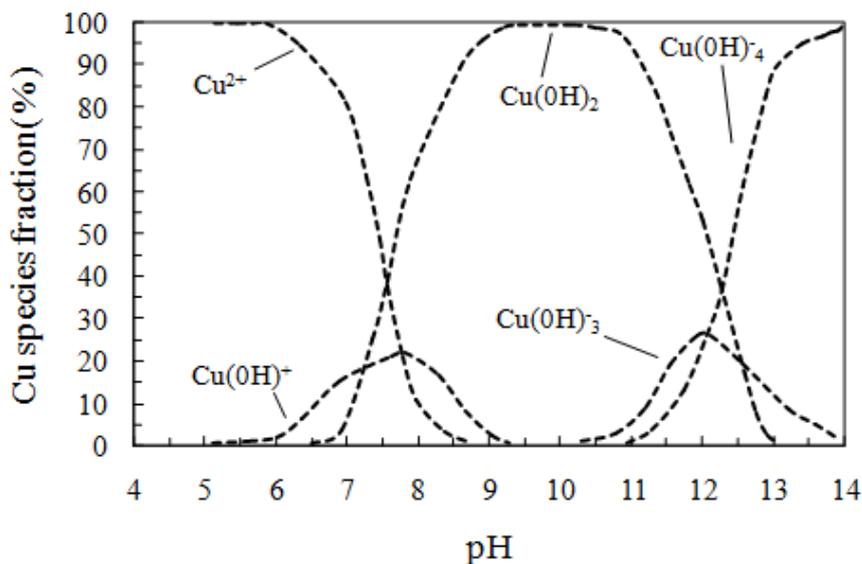


Figure 7. Copper species fraction vs. solution pH [22].

4. Conclusions

The AFM images obtained show the morphology of mineral surface changes by the change in the solution chemistry such as collector dosage, pH values, and presence or absence of exterior ions. According to the results obtained, increasing the pH value from 7.5 to 9.5 improves the collector coverage on the chalcopyrite, suggesting that a better morphology through flat patches appears. In addition, at constant pH values (either neutral or alkaline), the collector concentrations increase from 25×10^{-3} to 50×10^{-3} g/ton, mainly by forming the second and subsequently collector layer exposes. This phenomenon makes higher contact angles and patches. It can be concluded that a surface adsorption depends on another main factor, i.e. pH. Employing tap water instead of distilled water causes a better adsorption in quality and quantity, mainly due to the presence of Cu^{2+} cations. Moreover, applying tap water can reduce the collector consumption up to 50%.

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بررسی مکانیسم جذب زنتات بر روی سطح کالکوپیریت: مطالعه به وسیله AFM

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چکیده:

در این پژوهش، جذب کلکتور پتاسیم آمیل گزنتات بر روی سطح کالکوپیریت خالص توسط روش AFM مورد مطالعه قرار گرفته است. آزمایش‌های جذب، در غلظت‌های مختلف کلکتور در مقادیر مختلف از pH در حضور و عدم حضور یون خارجی انجام شده است. تغییرات مورفولوژی سطح کالکوپیریت که بر اثر جذب کلکتور پدید آمده است، به وسیله اندازه‌گیری زاویه تماس کلکتور و سطح ارزیابی شده است. با توجه به تصاویر سه‌بعدی به دست آمده از روش AFM، افزایش مقدار pH از ۷/۵ به ۹/۵ در دو غلظت از کلکتور 25×10^{-3} و 50×10^{-3} گرم بر تن باعث افزایش تعداد ذرات کلکتور جذب شده بر سطح، بهبود مورفولوژی سطح و کاهش زاویه تماس شده است. علاوه بر این، در یک مقدار pH ثابت، افزایش غلظت کلکتور باعث بهبود زاویه تماس و یک افزایش نسبی در تعداد ذرات شد. با مقایسه تغییرات مورفولوژی سطح نمونه در آب شهری یا مقطر، به کارگیری آب شهری به دلیل وجود یون Cu^{2+} و فعال‌سازی سطح از طریق تولید CuS، کیفیت و کمیت جذب را افزایش داده است. به کارگیری آب شهری نه تنها باعث پوشش مناسب به وسیله کلکتور شده است بلکه کاهش مصرف کلکتور به مقدار ۵۰٪ را به همراه دارد.

کلمات کلیدی: تغییرات مورفولوژی، پتاسیم آمیل گزنتات، کالکوپیریت، AFM.
