

Research article

Effect of Diesel on Adsorption of Coco Amine in Muscovite and Quartz

Ismail Ibrahim and Md Muzayin Alimon

Mineral Research Centre, Minerals and Geoscience Department Malaysia,
Jalan Sultan Azlan Shah 31400, Ipoh, Perak, Malaysia
Phone Number: 605-5477052
Fax number: 605-5477185

E-mail: is.ibrahim@jmg.gov.my and muzayin@jmg.gov.my



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Abstract

A study of surface properties of a muscovite source found in Malaysia was carried-out to evaluate their flotation performance. For this study, ground muscovite and quartz samples were used. The adsorption mechanism of long-chain alkyl amine (coco amine) and diesel on muscovite and quartz were studied by zeta potential and Fourier transform infrared (FTIR) measurements. Zeta-potential results showed that the isoelectric points of the muscovite and quartz were 1.80 and 2.00, respectively. The differences in surface charge between minerals at pH 2 were related to the preferential of muscovite (-4.80 mV) flotation from quartz (0.00 mV) whenever coco amine C100 was used as a collector. Then, at pH 2 FTIR analysis was used to examine coco amine and diesel adsorption on the muscovite and ground quartz surfaces by means of alkyl band characteristic between the region 3000-2800 cm^{-1} as $\nu_{\text{as}}(\text{CH}_2)$ and $\nu_{\text{s}}(\text{CH}_2)$ groups at various dosage. The muscovite floatability study was carried out by using Denver cell. For this flotation, a synthetic sample was used. The flotation test results showed that by adding diesel, the floatability of quartz was reduced and by this, it increased muscovite grade and recovery. The result showed that at 50 g/t diesel, the grade and recovery of muscovite obtained were 30.00% and 55.96% respectively and reaching to the maximum grade (64.44%) and recovery (81%) when the amount of diesel increased to 150 g/t. The optimum result was obtained when a sample containing 10 g of muscovite with d_{80} at 450 μm (80% of particle passing the size) and 190 g of quartz was used. The other parameters involved were diesel (150 g/t), pH 2, percentage of solids (20%), coco amine (170 g/t) and pine oil (150 g/t).

Keywords: muscovite; quartz; adsorption; flotation.

Introduction

Mica is grouped as an alumina-silicate mineral which is normally found in pegmatite deposit. Generally, it exists as impurities which are associated with i.e., albite ($\text{NaAlSi}_3\text{O}_8$) and orthoclase or microcline (KAlSi_3O_8). Its surface contains ions, mostly potassium and a lesser extent sodium, which can be exchanged with other inorganic or organic cations [1]. Muscovite mica has found increasing interest in both industrial and basic research because of its layered structure, chemically inert and smooth surface. Normally, this mica that may have economic values is produced as by-products of other process such as to get feldspar from pegmatite sources.

In physico-chemical separation process, the floatability of muscovite is essentially determined by their adsorption properties. Thus, when dealing with muscovite which exists with other mineral such as quartz, the adsorption behavior of a collector (e.g. coco amine) and a fuel oil (eg diesel) on muscovite and quartz need to be fully studied. The adsorption mechanism is explained by the interaction between negatively charges minerals surface to positive charges from the collector. If muscovite surface is immersed in strong acidic aqueous medium, K^+ ion will leave the lattice and it becomes negatively charged [2]. In higher pH or in alkalinity solution, the negative charge is contributed by OH^- which then will be measured as zeta potential [3].

In acidic mica flotation circuit, sulfuric acid can be used as pH controller combined with amine collector [4]. Mica flotation studies were carried out on an ore from Alabama that contained 16.5 % muscovite and 77% quartz with the particles size range of (-590+53) μm , coco amine acetate as collector (140 g/t), assisted by kerosene as promoter (200 g/t), and flotation at different pH between 4.0 and 4.5. Based on the test results, a concentrate grade of 98% with a recovery of 96% was achieved [5]. However, the addition of diesel as promoter will increase the selectivity of muscovite as compared to quartz [6]. The function of fuel oil is essentially subjected to the adsorption of hydrocarbon on muscovite. Basically, hydrocarbon or oil droplets containing molecules such as fuel oil and kerosene are non-polar. The oil droplets can interact with minerals surfaces, then spread out as oil lenses and enhance hydrophobic properties of the minerals. For this purpose, the oil droplets should be prepared with distilled water by ultrasonic reaction [7].

This paper aims at evaluating muscovite and quartz behavior during flotation process by understanding how the coco amine interacts with muscovite and quartz surfaces. Besides, the effect of diesel on the surface of muscovite and quartz was also studied. The data collected from zeta potential and FTIR techniques were then correlated in order to develop a better understanding of the adsorption of coco amine and diesel on muscovite and quartz. Thus it can provide guidance for manipulation of surface properties before proceeding to the separation between them using a real sample. Finally the performances of binary mixture minerals (muscovite and quartz) flotation by means of Denver cell were observed.

Materials and Method

Sample Preparation and Reagent. The natural pegmatite source sample containing muscovite, quartz and feldspar was obtained from Bukit Mor, Johore, Malaysia. For single mineral tests, muscovite ($\text{KAl}_2(\text{AlSi}_3\text{O}_{10})(\text{OH})_2$) and quartz (SiO_2) minerals 99.8% purity were used. For each single mineral sample, 200 g of muscovite and quartz were ground separately in a laboratory ball using a load of 5.0 cm, 3.75 cm and 2.50 cm steel balls (weighing ratio of 5 kg, 3 kg and 2 kg, respectively) for several minutes and sieved for -150+38 μm size range. The muscovite and quartz were characterized using X-ray fluorescence (XRF) to determine the chemical compositions. The chemical reagent for flotation used were (i) Coco amine C100 or cocos alkyl amine ($\text{NH}_2\text{C}_{8-18}\text{H}_{17-37}$), as a collector containing 99.0% of primary amine obtained from Clariant, Germany, (ii) Diesel ($\text{C}_{12}\text{H}_{22}$), as promoter obtained locally and was mixed with deionized water and treated using ultrasonic and (iii) Pine oil ($\text{C}_{11}\text{H}_{10}\text{BrN}_5$) as a frother.

Zeta Potential Measurements. The objective of determining zeta potential is to obtain an indication of the magnitude of the potential at the beginning of the diffused double layer around the particles. To perform the zeta potential test on single mineral for muscovite and quartz, each ground sample (-150+38 μm) was pulverized using agate mortar. This material was then mixed with deionised water (~5% solids) to form slurry. The slurry was then sieved using a sieve (-5 μm) in ultrasonic bath. The undersize fraction was then dried. The dried powder was then dispersed in deionized water at a concentration of 0.025 g/100 ml and conditioned for 30 minutes (the deionised water with constant ionic strength of 10^{-3} M was prepared by adding with KNO_3 at particular pH). After conditioning, the pH of the suspension was recorded again and regarded as the pH of the measurement. Zeta potential was determined using Brookhaven Zeta plus zeta meter [8].

FTIR Measurements. The relative adsorptions of this coco amine and diesel on muscovite and quartz were accessed by the intensity of the absorption band and were tested by Fourier transformed infrared (FTIR) spectroscopy. Thus the alkyl band characteristic, ν_{as} (CH₃), ν_{as} (CH₂) and ν_s (CH₂) as well as amine spectrum, ν_{as} (NH₂) and ν_s (NH₂) could be determined. At the same time the area under the alkyl chain and amine spectrum also can be measured. To perform the FTIR test, 0.1 g of the mineral powder (-5 μ m) was conditioned in an Erlenmeyer flask containing 100 ml collector solution at specified pH and concentration for 60 minutes. After equilibration, the suspension was filtered through millipore filter paper and the solids were air-dried overnight at room temperature. The samples were mixed with KBr matrix. The FTIR spectrums were obtained with Perkin Elmer Spectrum One, an average of 400 scans measured at 4 cm⁻¹ resolution.

Denver Cell Flotation by Binary Mixture. Binary mixture (muscovite and quartz) flotation technique was carried out in a 1.0 litre mechanical laboratory Denver flotation cell. The stirrer speed was operated constantly at 1000 rpm. Sulphuric acid or NaOH was used to control pH of the slurry. After conditioning and the pH of the slurry was fixed, coco amine with 170 g/t concentration was then added and conditioning process continued for another 5 minutes. It was followed by diesel addition. Diesel consumption was varied from 50 - 250 g/t. Pine oil was then added and the conditioning continued for another 3 minutes before applying air into the cell, from which moment the flotation started. Flotation was carried out for 2 minutes until the air flow was stopped. During the flotation process the froth product was removed by a scrapper manually. In these experimental works, a binary mixture of 95% quartz and 5% muscovite was used. In each test 200 grams of sample was used. For 5% muscovite, the percentage of -600+212 size range particle was fixed at 65%, 75% and 85%. In the investigation, the relationship between the pH of the slurry, dosage of the collector, particle size of the sample, dosage of frother and dosage of promoter with flotation performances were studied. The grade and recovery of muscovite in float product was determined through grain counting and weight analysis. The float sample was placed onto a glass slip (3x1 cm²), then the number of grains (particles) for quartz and muscovite were calculated under microscope. Based on the number of particles and their specific gravity (SG), the concentration can be estimated by multiplying the SG and the number of grains obtained.

Results and Discussions

Mineral Composition. The chemical composition of the muscovite by XRF analysis showed the major composition present were SiO₂ (45.16%), Al₂O₃ (35.35%), Na₂O (0.80%) and K₂O (11.06%).

Zeta Potential. Fig. 1 illustrates the effect of pH on zeta potential of muscovite and quartz. It shows that an increase in the pH of the suspension resulted in an increase of the negative charge of both samples until pH 10. Above this pH a charge reversal is observed. The results show that the isoelectric point (iep) of quartz is at pH 2 while the extrapolation of the curve in the case of muscovite indicated iep at about pH 1.80. At pH 2, while quartz shows potential of 0.00 mV, muscovite exhibits -4.80 mV. However, after pH 3 quartz having more value of negative charge compared to muscovite. Due to iep of quartz was at pH 2, the zeta potential as a function of collector concentration was performed at pH 2 as shown in Fig. 2. The figure shows an increase in the zeta potential of all minerals corresponding to the increasing of coco amine concentration. It also shows that muscovite posed much higher mV value as compared to quartz when the dosage of coco amine was kept increased until up to certain concentration.

Nonpolar oily organic compounds such as fuel oil have been widely used as collectors in muscovite flotation to improve the hydrophobicity of the muscovite surface [9]. The NH₃⁺ ions are the major cationic ions and adsorb onto negatively charged muscovite and quartz. The positive charge of NH₃⁺ indicates that the amine group was protonated and subjected through coulombic attraction. If the pH is increased, the zeta potential will increase due to the adsorption of individual ions through surface precipitation [10 - 11].

In this experiment, Fig. 3 depicts the effect of diesel dosage on zeta potential for muscovite and quartz. It is known that the oil-water interface is negatively charged [9]. Thus from the zeta potential result, it clearly shows that in the presence of diesel correlated higher negative values of mV for muscovite as compared to quartz. However, when the diesel increased to more than 40 ppm the zeta potential obtained is closer to positive value. This indicated that H⁺ and OH⁻ ions have less of an impact on the surface potential than the surface ions. When the concentration of diesel exceeded its limit (more than 40 ppm) some of the OH⁻ ion would be actively attracted to agglomerated diesel. Zeta potential as a function of diesel concentration for muscovite and quartz at pH 2 with the presence of 40 ppm coco amine is shown in Figure 4. It can be noted that the zeta potential for muscovite was higher than quartz and its maximum reading was seen to be 18.00 mV at diesel concentration of 30 ppm. Yet, the positive zeta potentials decreased as the dosages of diesel were increased. Indeed the role of diesel was more likely to promote flotation selectivity for muscovite at 30 ppm diesel concentration.

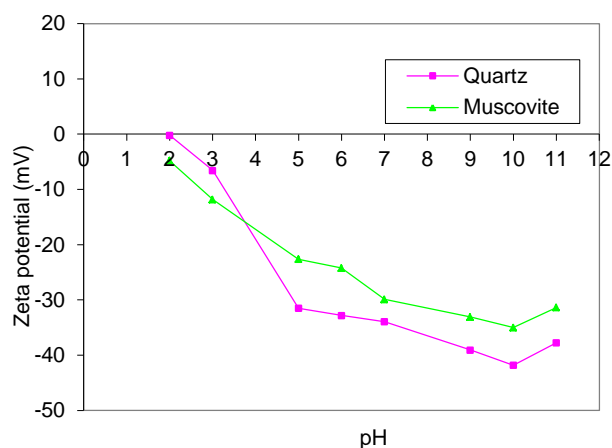


Fig. 1 : Effect of pH on zeta potential for muscovite and quartz

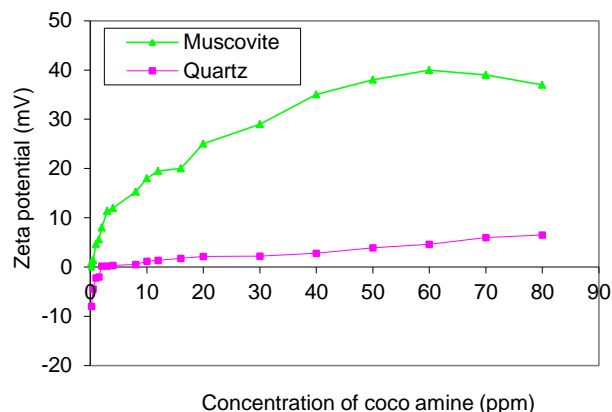


Fig. 2: Effect of zeta potential when coco amine are added on muscovite and quartz at pH2

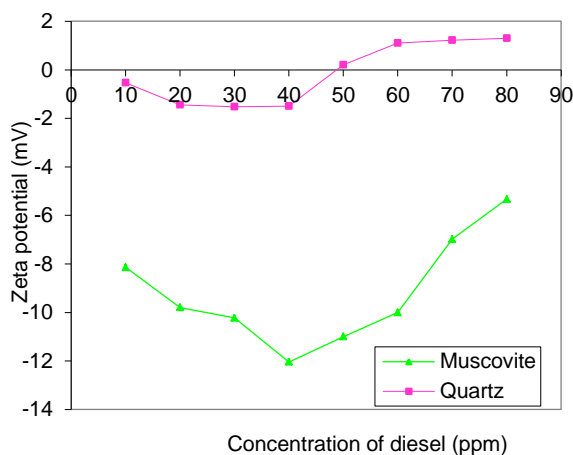


Fig. 3: Zeta potential as a function of diesel concentration for muscovite and quartz at pH 2.

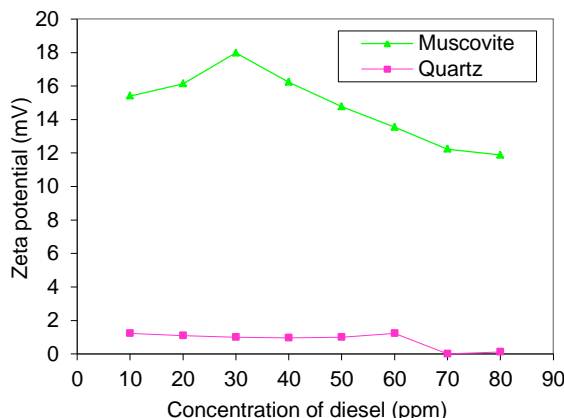


Fig. 4: Zeta potential as a function of diesel concentration for muscovite and quartz at pH 2 with the presence of 40 ppm coco amine.

FTIR Measurement. The FTIR spectroscopic studies on natural silica minerals and hydrocarbon in the frequency region of 2000 to 400 cm^{-1} carried out by other researchers [12] have shown the crystallinity of quartz and the existence of anhydrous which can be used as basis. However, in this study the FTIR spectra of natural muscovite and quartz treated with 40 ppm coco amine in the frequency region of 3000 to 2800 cm^{-1} have shown significant variations in their adsorption at different diesel dosage as shown in Figures 5 and 6. The FTIR spectra of muscovite and quartz treated with 40 ppm coco amine at pH 2 resulting quartz behaves less significant effect of coco amine and only generated the highest intensity. The bands of characteristic of alkyl chains can be seen clearly such as $\nu_{\text{as}}(\text{CH}_2)$ and $\nu_{\text{s}}(\text{CH}_2)$ groups between region 3000-2800 cm^{-1} area at 2922 cm^{-1} and 2852 cm^{-1} for muscovite, and for quartz at 2923 cm^{-1} and 2852 cm^{-1} . The FTIR spectra of muscovite and quartz treated with 40 ppm of coco amine as a function of diesel concentration significantly shown the intensity of alkyl chain bands increased with increasing diesel concentration for both muscovite and quartz. However the intensity increased more rapidly for muscovite compared to quartz with increasing initial coco amine concentration, emphasizing more coco amine adsorption on muscovite. Moreover, the total area under alkyl chain bands (3000-2800 cm^{-1}) of spectra of muscovite and quartz as a function of diesel concentration is shown in Fig. 7. It was found that the total alkyl area of both minerals increased corresponding to diesel concentration.

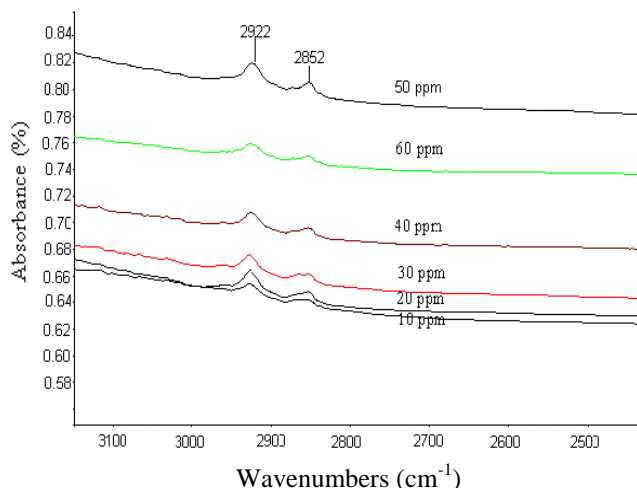


Fig. 5: Adsorption of coco amine (40 ppm) on muscovite with the presence of different concentration of diesel at pH 2.

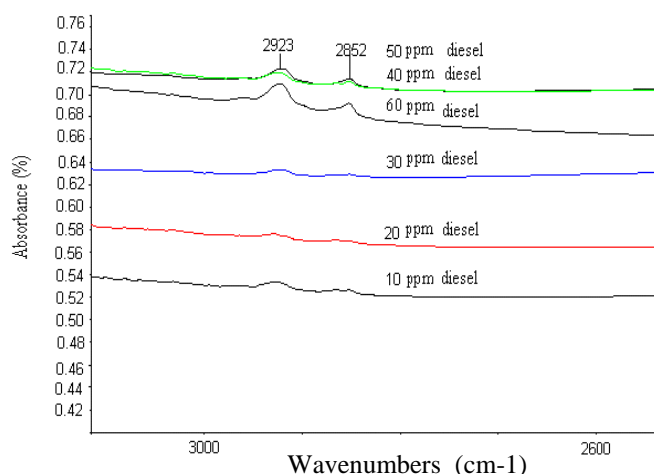


Fig. 6: Adsorption of coco amine (40 ppm) on quartz with the presence of different concentration

As overall, total area under alkyl chain bands for muscovite adsorption with coco amine were higher than quartz. In this circumstance diesel behaved to promote flotation selectivity for muscovite when adsorbed with coco amine.

Denver cell flotation by binary mixture - Effect of diesel dosage. Fig. 8 shows the effect of diesel dosage using d_{80} of individual muscovite in 10 g of 450 μm at pH 2. This pH was chosen because quartz shows 0.00 mV (zeta potential) and it is an agreement with Xua [13] stated that the recovery of muscovite ranged from 80.0% (at pH 2) to 50.0% (at pH 11) when dodecylamine acetate was used. Fig. 8 shows that at 50 g/t diesel, the grade and recovery of muscovite obtained were 30% and 55.96% respectively and reaching to the maximum grade (64.44%) and recovery (81.00%) when the amount of diesel increased to 150 g/t. To some extent diesel was capable to increase the selectivity between coco amine and muscovite. Attachment by diesel on muscovite surface will enhance the hydrophobic properties of the particles. This can be simulated by the FTIR results that at certain amount of diesel will result to increase the total area under alkyl chain bands (3000-2800 cm^{-1}) of coco

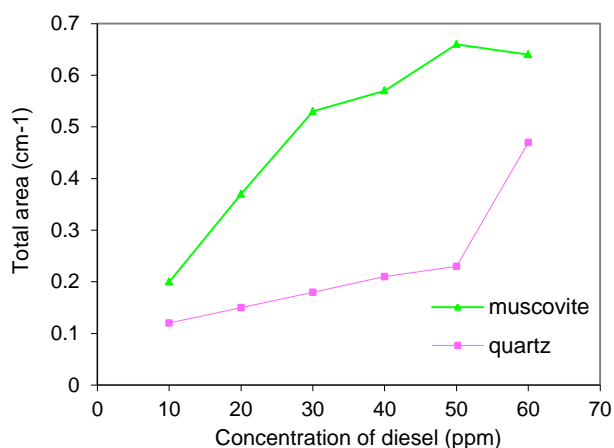


Fig. 7: Area under alkyl chain bands (3000-2800 cm^{-1}) of spectra of muscovite and quartz as a function of diesel concentration.

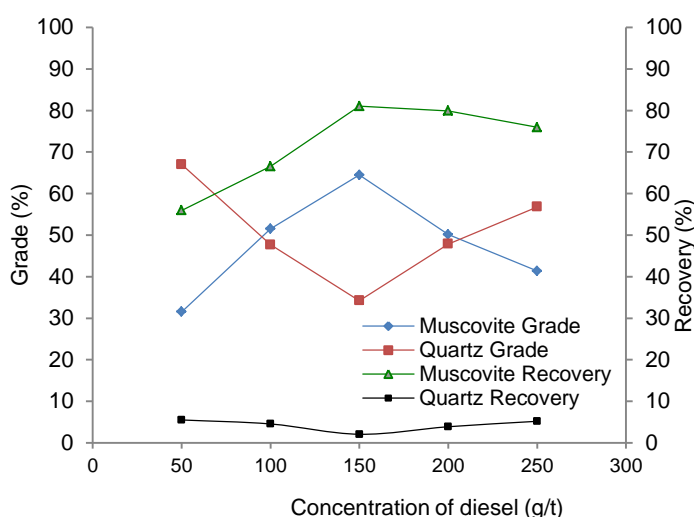


Fig. 8: The effect of diesel dosage on the flotation of muscovite at pH 2.

amine on muscovite surface. However, when the amount of diesel was further increased to 250 g/t the grade and recovery of muscovite were decreased to 50.12% and 76.00%, respectively. When the concentration of diesel exceeded 150 g/t some of the OH⁻ ion would be actively attracted to agglomerated diesel.

Conclusion

The zeta potential at pH 2 showed that the magnitude of mV for quartz was 0.0 compared to muscovite (at pH 1.8). Thus, pH 2 was applicable to be used for separation of these minerals by flotation. At pH 2 the adsorption of coco amine has shown a less significant effect on quartz resulting in lower results of intensity of the alkyl chain bands. However with increasing coco amine concentration, the adsorption of this amine on muscovite was increased significantly compared to quartz. In Denver flotation test, diesel was believed to reduce the selectivity between the coco amine and quartz but increased the selectivity for muscovite.

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