Flotation separation of dravite from phlogopite using a combination of anionic/nonionic surfactants

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Abstract: In the present study, the effect of a mixture of anionic collector (sodium oleate)/ non-ionic surfactant (sorbitan monooleate) on the flotation separation of dravite from the main associated gangue phlogopite was investigated in comparison of using single sodium oleate as a collector. The flotation experiments were conducted on pure single minerals of dravite and phlogopite. The flotation results demonstrated that adding sorbitan monooleate to Na-oleate at a dose of 5.64 kg/Mg Na-oleate with 0.43 kg/Mg sorbitan monooleate in the presence of methyl isobutyl carbinol (MIBC) as a frother (0.12 Kg/Mg) at pH 7.7 improved the flotation efficiency of dravite from 73.5% by using sodium oleate as a single collector to 96.9% using the mixture. Whereas, the flotation recovery of phlogopite in the same conditions reached 26.3%. The zeta potential measurements and Fourier transform infrared (FTIR) spectroscopy analysis were also implemented to investigate and predict the mechanism of collector adsorption on the mineral surfaces. As a result of flotation experiments and the zeta potential measurements of FTIR analysis, high chemisorption of the mixed collector on the surface of dravite was obtained, on the contrary, a little amount of the collector mixture adsorbed on the phlogopite surface.

Keywords: dravite, phlogopite, anionic/nonionic mixture, flotation, zeta potential, FTIR

1. Introduction

Dravite, a sodium-magnesium-rich black variety of tourmaline, is a ring-structured borosilicate mineral, and has a chemical composition of NaMg3Al6Si6O18(BO3)3(OH)3(OH), and contains approximately 10% boron oxide (Hawthorne and Henry, 1999). It is commonly found in metamorphosed limestones and mafic igneous rocks. Less frequently, it also occurs in pegmatites and as authigenic overgrowths on other tourmalines in sedimentary rocks (Henry and Dutrow 1996). Minerals of tourmaline group are the primary source of boron that distinguishable by its radiation-absorbing effects, therefore, boron and its compounds have extensive applications in the nuclear industry as shielding, control, and safety systems of nuclear reactors (Matkovich, 1977). Tourmaline minerals also have attracted widespread attention because of their piezoelectric and pyroelectric characteristics that when temperature or pressure conditions change, the tourmaline crystal generates an electric potential difference that results in the ionization of the surrounding air and conversion of the adjacent water and oxygen molecules into negative oxygen ions. The oxygen ions move in the air and transfer negative charges to dust particles, smoke particles, and water droplets, resulting in the purification of the air (Wang et al., 2006). Therefore, as it possesses excellent functional and environmental properties, tourmaline has been applied as a coating material in interior paints, furniture, and electrical appliances to purify indoor air (Jiang et al., 2006; Leonard et al., 2011).

The present study was carried out on raw material from Wadi El-Gemal district, South Eastern Desert of Egypt. Wadi El-Gemal district includes four tourmaline areas; Um El-Debbaa, Um Slimate, Wadi Sikait, and Wadi Abu Rusheid. One of the most common and abundant associated minerals of dravite in Wadi El Gemal district is phlogopite. Phlogopite is a potassium-magnesium-rich aluminosilicate mica mineral. Phlogopite is used commercially in plastic composite body parts for automobiles; also ground phlogopite is used as a substitution material for asbestos in automobile brake

linings and clutch plates. Phlogopite is added to industrial coatings used to increase strength and stiffness as well as to improve resistance to heat, chemicals, and ultraviolet rays.

Phlogopite and dravite have similar physical properties, for example, they are paramagnetic minerals, and the specific gravity is about 3 for the dravite and 2.8 for phlogopite. Therefore, conventional mineral separation methods (gravity and magnetic) are not satisfactory especially at fine sizes. Flotation is the most widely used beneficiation method for fine dravite at present. In the research field of dravite flotation process, few studies have been carried out (Zhifeng et al., 2013; Fernando Pita, 2017) while there no enough information in the literature regarding dravite flotation from phlogopite. A comparison test of magnetic and flotation separation of tourmaline was studied by Zhifeng et al. (2013), and this study proved that the flotation separation achieved better results than magnetic separation, and also manifested that the flotation results were better when closed-circuit use the anionic collector (sodium oleate) than the cationic collector. The adsorption of the ionic surfactant (anionic or cationic) on the mineral surface increased in the presence of the nonionic surfactant and chain-chain interaction between the adjacent ionic and nonionic surfactants on the mineral surface was proposed to be responsible for the enhanced adsorption (Qun et al., 1991; Wang et al., 2015; Fawzy, 2018; Fawzy, 2021).

Therefore, this study aimed to investigate for the first time, the flotation separation of dravite from the associated gangue (phlogopite) and also the comparison between a mixture of anionic collector and a nonionic surfactant was studied versus using the anionic collector separately for the separation of the two minerals. Zeta potential experiments and Fourier transform infrared spectroscopy (FTIR) analysis were also performed for explaining the adsorption mechanism of collectors on dravite and phlogopite surfaces.

2. Materials and methods

2.1. Materials

Macroscopic black hexagonal dravite crystals and yellowish-brown phlogopite flakes were obtained from Wadi El Gemal district by hand selection after crushing, as shown in Fig. 1. Selected pure mineral samples were prepared for the flotation experiments and analyses by further crushing and grinding using an agate mortar to obtain a pure mineral powder with a diameter of d_{90} 75 μm.

The reagents used in the dravite flotation process were sodium oleate (Na-Ol) (anionic collector, extra-pure 99%) obtained from Oxford laboratory-Reagent India and sorbitan monooleate (SMO) (non-ionic surfactant, span 80, 99%) obtained from LOBA Chemie-India. Sodium hydroxide (99%) and hydrochloric acid (35-37%) analytical grade obtained from Alpha Chemicals were used as pH modifiers. Methyl isobutyl carbinol (MIBC) obtained from Merck Schuchardt OHG- Germany was used as a frother.

2.2. Methods

2.2.1. Mineral characterization

Mineral identification of dravite and phlogopite samples was confirmed using X-ray diffraction (XRD) unit Philips PW-3710 with generator PW-1830, Cu target tube, and Ni filter at 40 kV and 30 Ma. While the chemical composition and particle size of the mineral samples were checked using a scanning electron microscope (SEM) which was provided with a Philips XL 30 energy-dispersive spectrometer (EDS) unit.

2.2.2. Flotation experiments

Small scale flotation experiments were performed using a micro flotation column (125 cm³, 30 cm high × 3 cm³ cross-section). In each test, 5 g of pure mineral (dravite or phlogopite) of -75 μm particle size was conditioned in 50 cm³ of distilled water for 3 min. Sodium oleate and MIBC (0.12 Kg/Mg) were added at the prefixed pH in the absence or presence of sorbitan monooleate, after the conditioning process, the suspension was then transferred to a flotation column which had a stopper closed to prevent solution filtration. Air was introduced at a flow rate of 50 cm³/min, and flotation was performed

Fig. 1. Macroscopic hexagonal crystals of dravite and the associated phlogopite flakes in their ore material from Wadi El Gemal district

was performed for 3 min. The froth was taken away from the top of the flotation column into a beaker with the help ofa clean plate of glass. At the end of the experiment, both the froth and sink products were filtered, dried, and weighed. The mineral flotation recovery was calculated after the float and sink drying and according to Eq. 1:

$$R(\%) = \frac{F}{F + S} \cdot 100$$

where R represents the flotation recovery, F and S represent the mass of float and sink products, respectively. Each flotation experiment was repeated three times under the same conditions, and the final average was reported.

2.2.3. Zeta-potential measurements

Zeta-potential measurements for single pure mineral samples of dravite and phlogopite were carried out using a UK Malvern zeta sizer nano series- zs to determine the surface ionization behavior as well as the isoelectric point (IEP) for the studied minerals. Each zeta-potential measurement was performed using 0.1 g (- 75 µm) of material that was agitated in 50 cm^3 of 0.01 M NaNO$_3$ solution as a background electrolyte. Sodium hydroxide and hydrochloric acid were used for adjusting the pH values of the suspension. The zeta-potential experiments were carried out at pH range 2-11, and each measurement was repeated three times to ensure repeatability and provide a standard deviation of the isoelectric point pH value (± 0.1 mV).

2.2.4. FTIR analysis

Fourier transform infrared (FTIR) analysis for the surfactant mixture (Na-oleate and sorbitan monooleate) was performed and dravite and phlogopite dried pure samples before and after treatment with the mixture were carried out to determine the availability of collector mixture adsorption on the mineral surface. Using FT-IR Model Cary 630 FT-IR spectrometer for both qualitative and quantitative (for liquid samples) analyses produced by Agilent technologies Company, the samples were investigated in spectral range (wavenumbers cm-1) from 4000 cm^{-1} to 400 cm^{-1} without any treatment.

3. Results and discussion

3.1. Sample characterization

Dravite and phlogopite samples were mineralogically confirmed using the XRD analyses, and the results are seen in Fig. 2. The lines of diffraction are in accordance with reference code no. 86-1572 for

dravite and reference code no. 10-459 for phlogopite. The major peaks of dravite were observed at 2θ angles of 20.66°, 31.43°, 33.35°, 38.41°, 45.48°, 52.70°, and 73.20° while phlogopite major peaks were observed at 13.07°, 39.98°, 54.23°, 69.45°, and 86.25° 2θ angles. The values of degree 2θ characteristic peaks were quite in accordance with dravite and phlogopite studied samples that confirming their purity.

The SEM and EDS analyses for the samples were also employed to investigate the mineral composition of dravite and phlogopite, also the particle sizes of the samples. As seen in Fig. 3 that dravite and phlogopite particles have a diameter mainly less than 75 μm. The mineral composition data confirm the purity of the minerals.
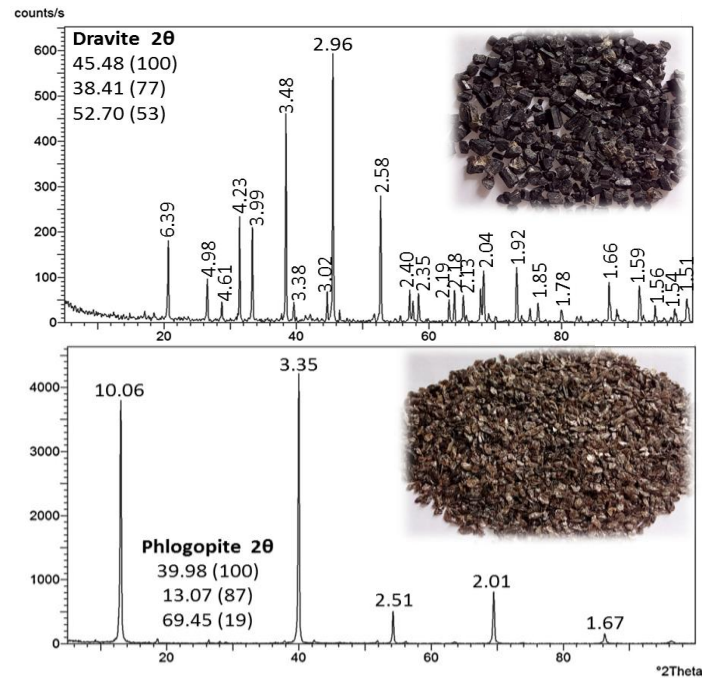

Fig. 2. XRD spectra and 2θ characteristic peaks of dravite and phlogopite pure samples
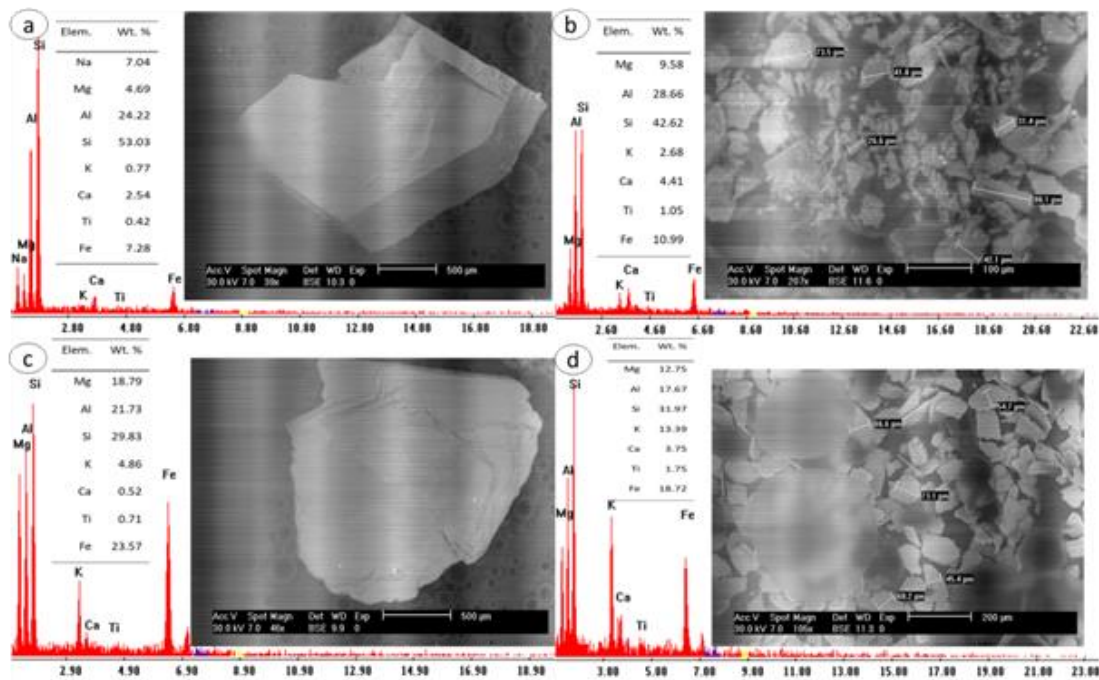

Fig. 3. SEM images and EDS analyses for (a) Dravite crystal, (b) Ground dravite, (c) Phlogopite flakes, and (d) Ground phlogopite

3.2. Zeta potential measurements

Understanding the zeta-potential of a mineral as well as its isoelectric point (*IEP*), combined with knowledge of a collector's ionization behavior at various pH levels in aqueous conditions, allows one to predict the mechanism of collector adsorption on the mineral surface (Pope and Sutton, 1973; Cheng et al., 1993; Kosmulski, 2009). The zeta potential measurements for pure untreated dravite and phlogopite powder (less than 75 μm) were carried out, and the results are shown in Fig. 4. The zeta potential-pH profile of the dravite sample showed that the isoelectric point (*IEP*) occurred at pH~2.8, andthis result is agreed well with measurements from the previous studies as Houchin (1986) and Fuerstenau (2001).

Fuerstenau, (op. cit.) concluded that the ring-structured silicate mineral's isoelectric point was supposed to be within 2.5~3.5 while Zhang and Wu (2013) made a comparison between the measured (pH$_{PZC}$=3.4) and calculated (pH$_{PZC}$=5.38) dravite isoelectric point, and they found that there was a difference between them, but they concluded that the measurement value was more accurate.

Zhang and Wu (op. cit.) also concluded that the broken tourmaline caused several metal ions like Na$^+$, Mg^{2+}, and Fe^{2+} to be exposed, and those metal ions are preferred to be attached by polar water molecules and leave the crystals of dravite. As a result, the dravite surface is negatively charged as the shortage of positive ions.

Phlogopite, like most other mica minerals, exhibited negative zeta potentials throughout the practical pH above 2. The isoelectric point of the studied phlogopite was below pH 2 as shown in Fig. 4. This is in line with the IEP of many mica minerals including muscovite (Scales et al., 1988; Mehrotra and Shekhar, 1995). This result is consistent with the results in the literature, as the phlogopite isoelectric point is below pH 2 (Hanyu et al., 2020), while Bai et al. (2019) concluded that the *IEP* of phlogopite was 2.35. The zeta potential measurements of the studied minerals revealed that both dravite and phlogopite surfaces showed negative charges throughout the pH range between 2.8 to 12.
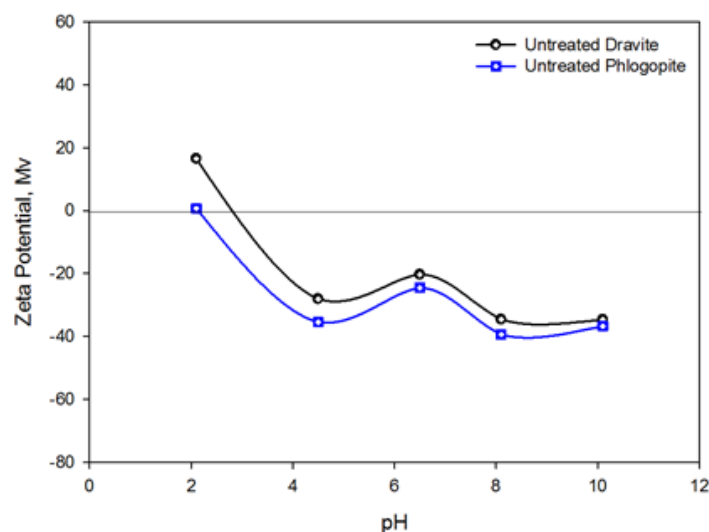

Fig. 4. Zeta potential-pH profile for dravite and phlogopite untreated pure samples

3.3. Floatation experiments

Fig. 5 shows the effect of pH on the flotation recovery of pure single dravite using sodium oleate (Na-Ol) as an individual collector at two different dosages (11.28 kg/Mg and 22.56 kg/Mg) in comparison with flotation recovery of dravite using a mixture of sodium oleate with sorbitan monooleate (NaOl-SMO) at a dosage of 5.64 kg/Mg Na-Ol with 0.43 kg/Mg SMO. The flotation recovery of dravite reached a maximum value at the pH ranges from 6 to 8, whether the sodium oleate as a single collector or as a mixture with sorbitan monooleate. When sodium oleate was used as a single collector at a dose of 11.28 kg/Mg, the flotation recovery of dravite reached a maximum of 73.6% at a pH of 7.7, whereas when the dose of sodium oleate increased to 22.56 kg/Mg, the flotation recovery of dravite increased slightly 76.2% at the same pH level. The flotation recovery of dravite using a mixture of sodium oleate with

sorbitan mono (NaOl-SMO) at a dose of 5.64 kg/Mg Na-Ol with 0.43 kg/Mg SMO reached the highest value of 96.9% at pH 7.7. It was concluded that adding a little non-ionic surfactant to the anionic collector increased the recovery rate of dravite significantly.

The flotation recovery of phlogopite as a function of pH within sodium oleate as a single anionic collector at doses of 11.28 kg/Mg and 22.56 kg/Mg is shown in Fig. 6 by comparison with flotation recovery of phlogopite with a mixture of sodium oleate and sorbitan monooleate at doses of 5.64 kg/Mg Na-Ol with 0.43 kg/Mg SMO. In general, the flotation recovery of phlogopite increased with the increasing pH, and the highest phlogopite recovery rate reaches pH 10, which is about 16% at a concentration of 11.28 kg/Mg of sodium oleate, then slightly increases to about 22% with increasing concentration of sodium oleate to 22.56 kg/Mg then reached the highest recovery value using a mixture of sodium oleate and sorbitan monooleate which is about 28%.

It is quite clear from Fig. 4 that the surface of dravite at pH range from 6 to 8 had a negative charge and at the same pH values, the mixture of anionic collector and non-ionic surfactant absorbed on the dravite surface, and this indicated that the high adsorption values on dravite surface may be due to chemisorption, not physisorption because both of mineral surface and collector mixture had the same negative charges (Somasundaran and Ananthapadmananthan, 1986; Abdel Khalek, 2001). The data of this study are compatible with the study of Yingmo and Xue (2012) that confirmed the presence of chemical   bonds between   te hydroxyl groups at the surface of tourmaline and sorbitan monooleate to
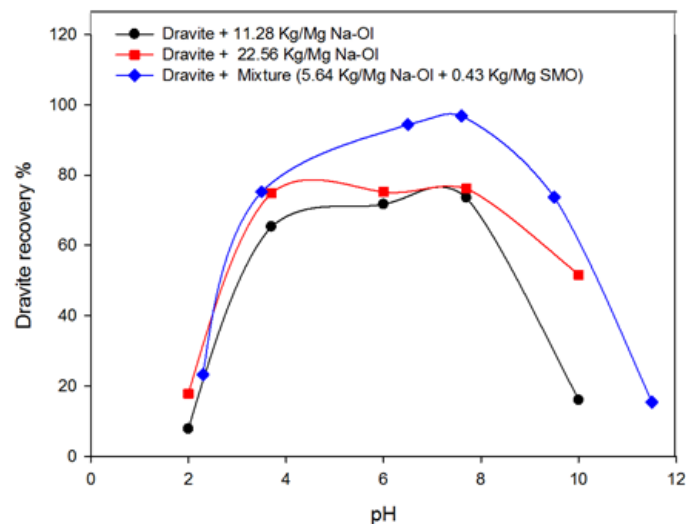

Fig. 5. Dravite flotation recovery as a function of pH that treated with Na-Ol and mixture of Na-Ol and SMO
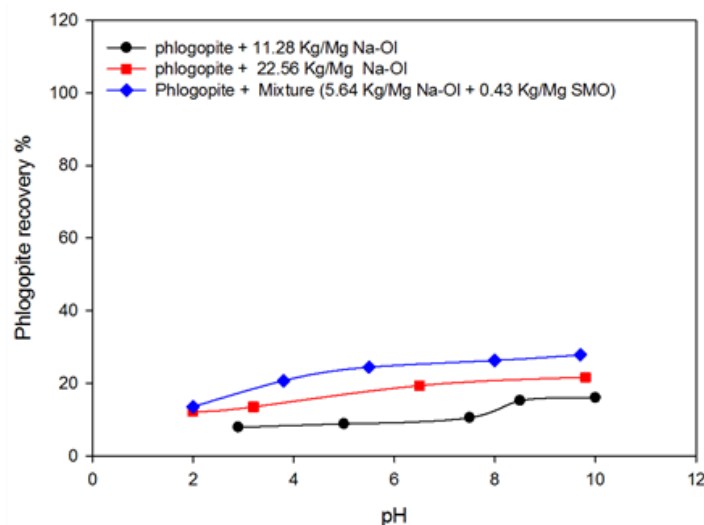

Fig. 6. Phlogopite flotation recovery as a function of pH that treated with Na-Ol and mixture of Na-Ol and SMO

produce tourmaline stearate and also confirmed that the contact angle of modified tourmaline increased to 125° from 0° for unmodified tourmaline, which indicates excellent hydrophobic properties for modified tourmaline.

Conversely, the decrease in collector adsorption on the phlogopite surface could be due to the strong repulsion between the negative phlogopite surface and the collector mixture (anionic collector with the non-ionic surfactant) (Abdel Khalek, 2001).

3.4. FTIR analysis

A Fourier transform infrared spectrum of untreated dravite is shown in Fig. 7, and the result exhibited that its main infrared absorption range was in 1400 to 400 cm^{-1} (Shen et al., 2011) and the characteristic absorption bands are 1350 cm^{-1}, 1256 cm^{-1}, 970 cm^{-1}, 776 cm^{-1}, 705 cm^{-1}, 603 cm^{-1}, 550 cm^{-1}, and 482 cm^{-1}. The absorption bands between 1250 cm^{-1} and 1350 cm^{-1} are attributed to the B-O group. As dravite is a ring silicate mineral, a strong band has presented at 970 cm^{-1} of Si-O stretches and Si-O-Si stretching vibration modes. Many vibration modes are observed at the absorption bands in the range between 800 cm^{-1} and 350 cm^{-1}, as B-O between 800 and 600 cm^{-1}, symmetric Si-O-Si band at ~700 cm^{-1}, and vibration bands of Mg-O, Al-O, and Si-O were observed below wavenumber 600 cm^{-1} (Robert et al., 1996 and Shen et al., 2011).

An infrared spectrum of the collector mixture of sodium oleate with sorbitan monooleate is shown in Figs. 7 and 8. The band at 3258 cm^{-1} is due to the stretching vibrations of the O-H groups while bands at 2001 cm^{-1}, 2291 cm^{-1}, and 2318 cm^{-1} are characteristic of the C-C triple bond of alkynes. The strong band at 1633 cm^{-1} is attributed to carbonyl groups (Coates, 2000).

Comparison of the infrared spectrum of untreated and treated dravite to collector mixture depicted new absorption bands corresponding to methyl (2926 cm^{-1}), methylene (2855 cm^{-1}), and carbonyl groups at 1633 cm^{-1}, which confirmed that the collector mixture reacts chemically with the hydroxyl groups on the surface of dravite. Other peaks that corresponding to B-O, Si-O, Mg-O, and Al-O were shifted by a few wavenumbers and broadened. These results confirmed that long-chain alkyl groups were introduced on the surface of the dravite by the reaction of the collector mixture with dravite powder.

The IR spectrum of treated phlogopite in comparison with the collector mixture and untreated phlogopite spectra is seen in Fig. 8. The untreated phlogopite spectrum revealed that its main IR absorption range was between 1100 to 400 cm^{-1} and its characteristic absorption band at 951 cm^{-1} is attributed to the Si-O group, while absorption band at 762 cm^{-1} (Al-O group), 667 cm^{-1} (Al-O-Al), 532, and 459 cm^{-1} (Si-O group) (Anton Beran, 2002). The comparison revealed that no new absorption bands appeared at the phlogopite surface after treatment with the collector mixture, which demonstrated that the weak adsorption between the collector mixture and phlogopite surface.
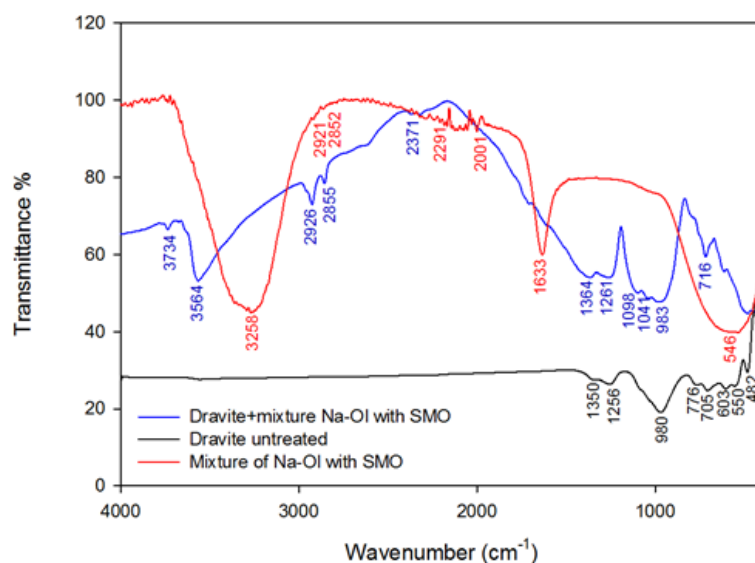

Fig. 7. Infrared spectra of untreated dravite, mixture surfactant of sodium oleate (Na-Ol) and sorbitan monooleate (SMO), and dravite treated with a mixture of Na-Ol and SMO
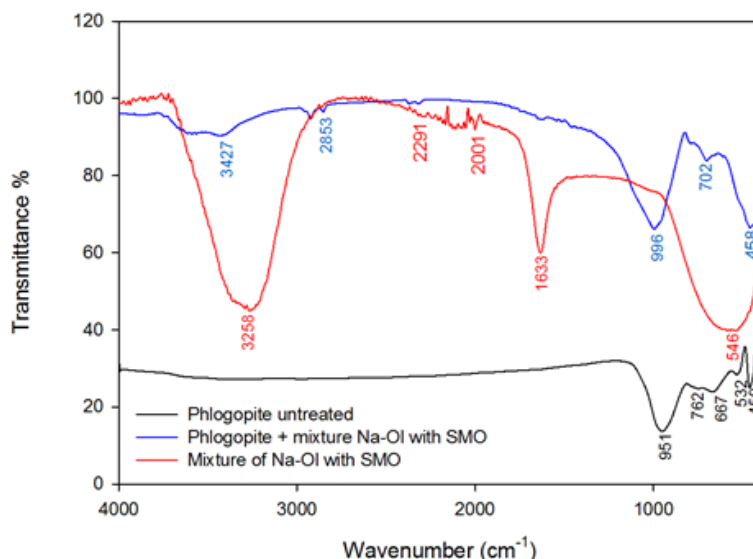
Fig. 8. Infrared spectra of untreated phlogopite, mixture surfactant of sodium oleate (Na-Ol) and sorbitan monooleate (SMO), and phlogopite treated with a mixture of Na-Ol and SMO

4. Conclusions

In this study, the results of flotation separation experiments for single minerals of dravite and the associated gangue (phlogopite) demonstrated that the collector mixture of sodium oleate with sorbitan monooleate showed higher selectivity for dravite than phlogopite compared to using sodium oleate as a single collector in the presence of MIBC as frother and at pH 7.7. The high adsorption values of the mixed collector on the dravite surface than phlogopite surface at an optimum pH value of 7.7 may be the product of the chemisorption process because both the dravite surface and collector mixture have the same negative charges.

FTIR measurements of untreated and treated dravite in addition to the collector mixture demonstrated the presence of new absorption bands on its surface and these results confirmed that long-chain alkyl groups were introduced on the surface of the dravite by the chemical reaction of collector mixture with dravite powder. While the treated phlogopite surface showed no noticeable absorption bands on its surface, indicating poor adsorption between the collector mixture and phlogopite surface.

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