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Implication and collecting mechanism of emulsified sodium vegetable oleate on fluorite flotation

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Abstract: The emulsified sodium vegetable oleate (ESVO) was prepared with low-cost vegetable oleate. Using ESVO as a collector, the flotation performance of fluorite had been investigated comparing with sodium oleate at a temperature of 20 ± 2 °C. The results of flotation showed that ESVO had better collecting performance than the sodium oleate. The interaction mechanism of these two collectors with fluorite was studied by the zeta potential, FTIR spectra and laser grain-size tests. Both ESVO and sodium oleate changed fluorite zeta potential by electrostatic attraction and chemical adsorption, and generated calcium carboxylate on the fluorite surface. Moreover, it was concluded that higher fluorite recovery was a consequence of ESVO smaller surface tension comparing to sodium oleate.

Keywords: *emulsified sodium vegetable oleate, collector, fluorite flotation, sodium oleate*

1. Introduction

Fluorite is widely used for producing hydrofluoric acid, making glass and as flux in steel, etc. These industries have different requirements for fluorite grade. For example, CaF_2 contents in fluorite is needed at least 80% and 93% in the glass industry and hydrofluoric acid industry respectively, while it is required to be more than 65% in steel smelting process. Therefore, to achieve the actual applied request, froth flotation is most commonly used as it can improve fluorite concentrates purity up to 99%. In practice, fatty acids (oleate, oxidized paraffin sodium soap and sodium naphthenate etc.) are widely used as collectors at a high pH (Crozier, 1992; Ayhan et al., 2013). If the silicate, calcite and iron oxides gangues, beside fluorite, are present, the depressants such as water glass is necessary for improving flotation selectivity (Zawala et al., 2007; Raju, 2000; Sun, 2012).

In addition, fatty acids are not only used as anionic collectors in flotation of fluorite but also in other minerals because of its strong foaming and collecting ability (Fan and Rowson, 2000; Liu et al., 2015; Satur et al., 2016). The mechanism of interaction between fatty acids and minerals is very complex based on unique solid physicochemical properties. Fatty acids can be adsorbed on the solid surface by the electrostatic, adhesion (Van der Waals) and chemical force and enhance the mineral floatability - hydrophobic particles aggregate and float together (Drzymala, 1995). There were reported the chemisorptions of fatty acids on minerals even with low fatty acids concentration (Hu et al., 1986). Nevertheless, the fatty acids use is limited with the greater dosage and easily influenced by temperature and ions presence (Sis and Chander, 2003). Therefore, it is imperative to find out an economical, effective and environment-friendly substitutes for fatty acids.

The fatty acids are the grease extracted from vegetable oils and animal fats. As important source of fatty acids, the vegetable based oils and its hydrolysis product have been developed as collectors in mineral flotation since 1930s, such as in flotation of coal, sulfide and non-sulfide ores (Floyd, 1935). In

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recent years, the vegetable oils have caused a new wave of research because of its availability, renewability and nonpolluting characteristic. Ajayi (2005) used alkaline salts of vegetable oils for galena and sphalerite recovery from complex sulphide ores. The results showed that the alkaline salts of groundnut and palmkernel oils had good ability for collectors and frothers. The nut oil, a typical palm tree in Brazil, has been saponified first, then characterized and utilized in apatite flotation, and the results showed that highest recoveries were reached at pH 8 and 9 (Silva et al., 2015). Mixing the vegetable oil with surfactant is also the way to extent its effectively application, as the presence of associated surfactant species in solution, which could also synergistically adsorb at the solid/liquid interface, increasing the hydrophobic character of the mineral surface (Filippova et al., 2014; Wang et al., 2015).

Despite many advantageous, the vegetable oils have not been used as the fluorite collector in literature, so its application in fluorite flotation need to be tested and optimized, and its collecting mechanism need to be explained. In this study, the sodium vegetable oleate, which is produced from the waste straw, is considered as the basic collector, because of its low price and environment-friendly properties. Furthermore, in our attempt to get better flotation performance of fluorite in room temperature, the sodium vegetable oleate is emulsified using the surfactant. The effect of emulsified sodium vegetable oleate on fluorite flotation was investigated through the micro-flotation test, zeta potential and FTIR measurement etc.

2. Experimental

2.1 Mineral

The pure fluorite was obtained from Shangluo of Shaanxi province, China. The mineral samples were ground into -74 µm size in a porcelain mill after initial crouching and selection by hand picking. Then, -74 µm fraction samples were cleaned by distilled water and collected for tests. The chemical multiple analysis results measured by X Ray Fluorescence (XRF) of pure fluorite are listed in Table 1.

Table 1. Chemical multiple analysis results of pure fluorite sample

Components	CaF_2	$CaCO_3$	SiO_2	Al_2O_3	MgO	Fe_2O_3	Others
Grade (mass fraction) (%)	98.57	0.26	0.93	0.07	0.05	0.09	0.03

2.2 Reagents

The mixed collector was made up by the anionic surfactant and sodium vegetable oleate. The anionic surfactant was obtained from Linyi, China. The vegetable oleate was obtained from Xuzhou, China. Chemically pure sodium oleate was tested for comparison. Analytically grade reagent HCl and NaOH were used as pH modifiers in all tests. Distilled water was used in all the experiments unless otherwise stated.

2.3 Preparation of ESVO

The ESVO is the product of the vegetable oleate through saponification and emulsification process. During saponification, the vegetable oleate was mixed with sodium hydroxide with the mass ratio of 100:13.5 in a 1 dm^3 beaker, and then about 300 cm^3 distilled water was added to the mixture. The beaker was placed in a water bath for heating and, when the temperature reached 90 ℃, saponificated for one hour. In the final process, before the obtained mixture cool to the room temperature, the anionic surfactant was added, and then the mixture was emulsified by mechanical agitation for 3 hour. The final product was ESVO.

2.4 Flotation tests

The micro-flotation tests were carried out in a XFG micro-flotation machine with 40 cm^3 plexiglass cell (Fig. 1) with impeller speed of 1600 rpm (Sun et al., 2010) at a temperature of 20±2 ℃. The pulp was prepared by adding 2.0 g of minerals to distilled water. The pulp pH was adjusted by adding 0.1 M HCl and NaOH, and the pulp was conditioned for 3 min. Then, the collector was added into the pulp and stirred for 3 min. After 3 min of flotation, the froth and products were filtrated, dried, and weighed. The flotation recovery was calculated based on the weights of the dry products.

Fig. 1. XFG micro-flotation machine

2.5 Surface tension tests

The surface tension was measured based on pulling escape method that pulled the platinum plate or ring with known circumference out of the liquid. According to the value of pulling force and circumference of either plate or ring, the surface tension is calculated by the following formula:

$$F = \alpha D \tag{1}$$

where, *F* is the pulling force and *D* is the circumference of platinum plate or ring.

The tests were carried out using K100 automatic surface tensiometer (Kruss, Germany). In the tests, the samples with different concentration were made at a temperature of 20±2 ℃, and the plate measurement method was used. About 30 cm^3 of measured samples were poured into the beaker, and then platinum plate was fixed on the balance hook, at the distance 2 mm from the liquid level. After that the tests began. Each measurement can gave 10 values, and the reported result was the mean value for at least 5 independent runs.

2.6 Zeta potential tests

Zeta potential tests were carried out using a Zeta Plus Zetameter (Brookhaven, USA). The mineral samples were ground to -2 μm in an agate mortar. A 20 mg of sample was first conditioned with 35 cm^3 of the desired solution by mechanical agitation for 15 min. After conditioning, the pulp was left overnight. Each zeta potential was measured three times and the average value was calculated. All zeta potential measurements were carried out at 20 ℃.

2.7 FTIR measurements

The characterization of collectors and the fluorite surface were studied by VERTEX 80v Fourier Transform Infrared Spectroscopy (Bruker, Germany). The infrared spectra of collectors were measured after extraction and purification in ether solution. Before and after collector adsorption, the infrared spectra of fluorite were measured with a KBr disc technique. The samples were made by adding fluorite to the prepared distilled water. The solution was conditioned according to the flotation tests and stirred for 1 hour, then filtered and dried at 60 ℃.

2.8 Particle-size distribution measurements

The particle-size distribution measurement was used for analyzing the hydrophobic agglomerate variation before and after flotation process by Master Sizer S3500 (Microtrac Instruments). The samples were conditioned according to the flotation tests and gently moved to the instruments by pipette. In order to prevent flocs breaking during the measurements, turbulence intensity index was set as zero.

3 Results and discussion

3.1 Characterization of ESVO

The infrared spectra of the ESVO and sodium oleate are shown in Fig. 2. The bands observed at 2854 cm^{-1} (a), 2924 cm^{-1} (a), 2852 cm^{-1} (b) and 2921 cm^{-1} (b) represent C-H stretching vibration of CH_3 and CH_2 groups. The peak at 720 cm^{-1}(a) (b) represents the CH_2 rocking vibration (Dréau et al., 2009). The peak at 1642 cm^{-1} (a) and 1645 cm^{-1} (b) is due to the C=O stretch from the carboxyl group. The peak at 1547 cm^{-1} (a) and 1559 cm^{-1} (b) is due to the $-COO^-$ asymmetrical stretching vibration, while the peak at 1408 cm^{-1} (a) and 1424 cm^{-1} (b) is due to the $-COO^-$ symmetric stretching vibration. The strong absorption peak of ESVO observed around 3359 cm^{-1} (a) is due to the presence of associated O-H group (Muruganantham et al., 2009). We conclude that the main functional groups of ESVO is carboxyl, which is similar to sodium oleate.

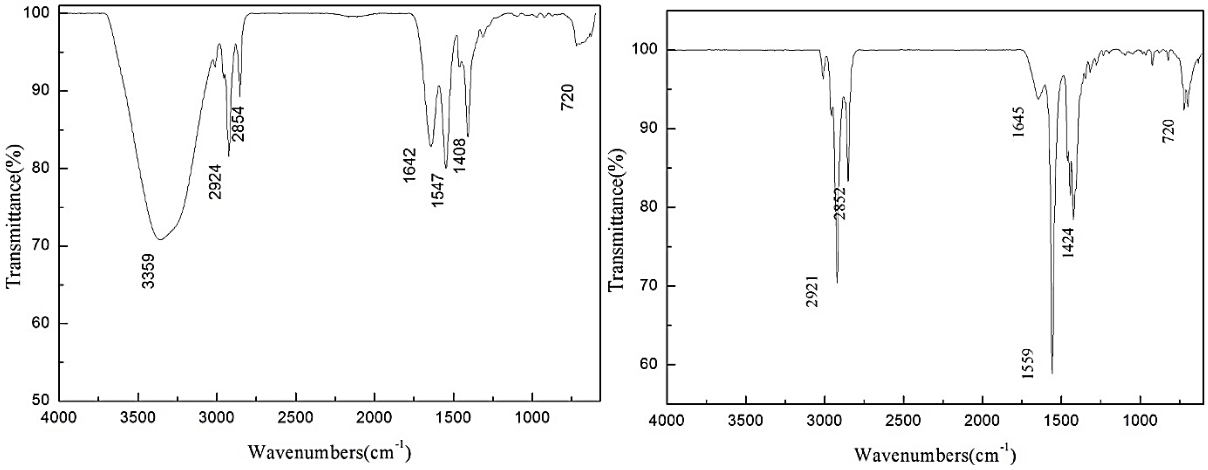

Fig. 2. FTIR spectra of ESVO (a) and sodium oleate (b)

3.2 Single mineral flotation results

In order to investigate the performance of ESVO in fluorite flotation in room temperature, the contrast experiments were applied when ESVO and sodium oleate were used as collector respectively.

The relationship between the recovery and the pulp pH was shown in Fig. 3, where concentration of ESVO and sodium oleate was 600 g/Mg and 900 g/Mg respectively. It was quite obvious that the ESVO had better collecting performance than the sodium oleate during all the tested pH except between 7-9. At the sodium oleate concentration of 900 g/Mg, fluorite showed good floatability at pH 5-8, with recovery varied between 79.85% and 86.93%. When pH > 8, the flotation recovery of fluorite decreased with pH. As illustrated in other work, the ion-molecular complexes with high surface activity had beneficial effects on flotation using oleate as the collector (Somasundaran, 1976). By calculating the equilibrium diagram of oleate variations as a function of pH, the concentration of ion-molecular complexes (RCOOH and $RCOO^-$) reached the maximum value at pH around 8.0 (Soly et al., 2015). So fluorite can get a better recovery around pH 8 when sodium oleate worked as the collector. When ESVO was used as the collector, the recovery of fluorite increased in the pH range 3-6 and exceeded 94.90% at pH 6, above which started to decrease.

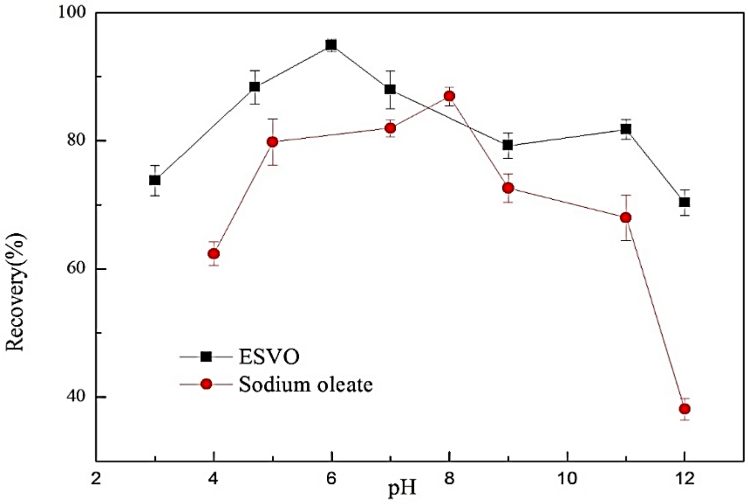

Fig. 3. Recovery of fluorite as a function of pH using ESVO and sodium oleate as collector respectively (ESVO 600 g/Mg, sodium oleate 900 g/Mg)

The relationship between the recovery of fluorite and the collector dosage was examined under pH 6 (ESVO) and pH = 8 (sodium oleate), respectively. The results are shown in Fig. 4. Comparison of the recovery for ESVO and sodium oleate revealed that both collectors had the same tendency for recovery modification with increasing dosage – recover initially increased, and then decreased. When the concentration of sodium fatty acid was large enough to reach the critical micelle concentration, multilayer adsorption was likely to be occurred at the interface, leading to the decrease of the recovery (Wang and Hu, 1988). For ESVO, the fluorite recovery reached 94% at a dosage of 600 g/Mg, while the sodium oleate results in 86% fluorite recovery at a dosage of 900 g/Mg. The flotation results indicated that ESVO had a better performance in fluorite flotation than sodium oleate, most probably because ESVO had a better dissolution ability.

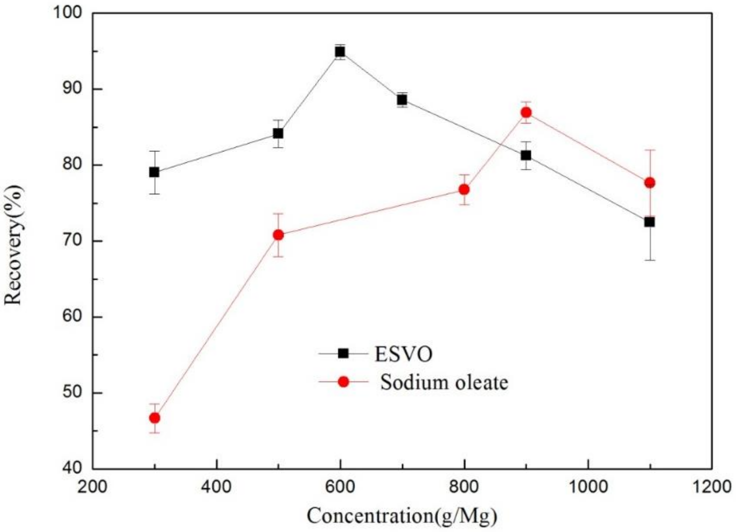

Fig. 4. Relationship between the recovery of fluorite and the collector concentration (pH_{ESVO}= 6, $pH_{sodium\ oleate}$ = 8)

3.3 Surface tension of collectors

The surface tension of the sodium oleate, sodium vegetable oleate and ESVO with different concentrations were tested, and the results are shown in Fig. 5. The surface tension of the three collectors decreased with the concentration increase, and finally reached a plateau. Critical micelle concentration (CMC) corresponds to the concentration of inflexion in the curves, which can explain the flotation performance presented in Fig. 4. It can be seen from Fig. 5 that the surface tension of the ESVO reached the plateau at lower concentration than for other two substances. Moreover, when reaching the CMC value, ESVO has the smallest surface tension among the three collectors, while the sodium oleate has the largest value.

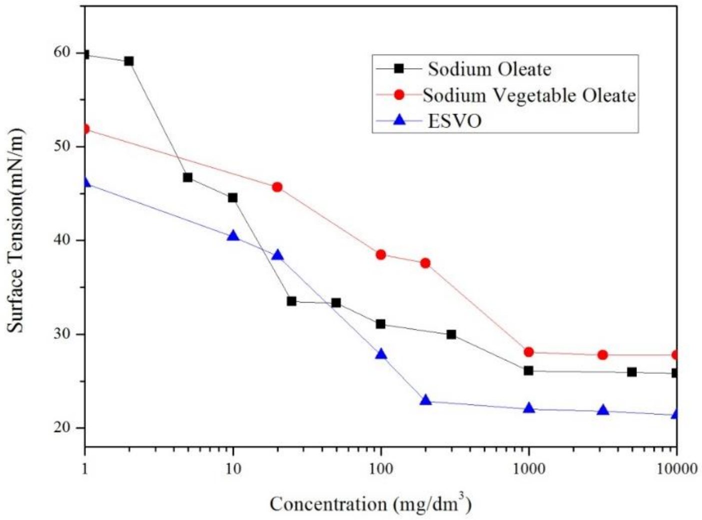

Fig. 5. Surface tension of collectors with different concentration

3.4 Zeta potential of fluorite

Fluorite is a typical ionic mineral, and surface charge at fluorite can be generated by the lattice ion hydration theory (Fuerstenau, 1962). The values of zeta potentials of fluorite particles, evaluated as a function of pH in the absence and presence of collectors, were shown in Fig. 6. It can be seen that the point of zero charge (PZC) of the fluorite is at around pH 5.5, and slightly different from the values reported in the literature, where the PZC was reported to be within the range of 6 - 10 (Miller and Hiskey, 1972; Miller et al., 2004; Zhao et al., 2012). This can be mainly a result of different origin of the fluorite samples used, caused different interfacial properties.

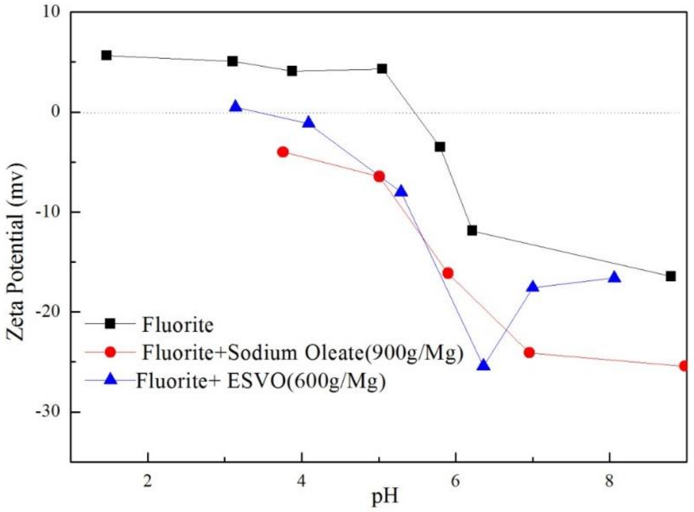

Fig. 6. Zeta potentials of fluorite as a function of pH

It can be seen also in the Fig. 6 that with addition of the sodium oleate, the zeta potentials of fluorite decreased significantly at whole pH range, indicating that sodium oleate can be adsorbed on the surface of fluorite intensively. When pH < 6, the presence of ESVO causes the zeta potential of fluorite to become more negative with the increase of pH, showing that anionic group of ESVO can be adsorbed on the positive charged fluorite surface. Moreover, the results indicate that electrostatic attraction is the driving force of the adsorption of ESVO on fluorite at pH < PZC, while the chemical adsorption mechanism become dominant between pH = PZC and pH = 6. When pH > 6, the zeta potential of fluorite with ESVO increased with pH. At pH around 8, the zeta potential of fluorite with ESVO was equal to that without ESVO. In alkaline solution, the adsorption of $RCOO^-$ or $(RCOO)^{22-}$ group on negative solid surface can reduce the concentration of OH^- which increased the adsorption of H^+ ions on the fluorite surface.

3.5 FTIR analysis on interaction of collectors and fluorite

In order to understand the interaction of collectors and fluorite, infrared spectroscopic analysis was studied and the results are shown in Fig. 7. The characteristic absorption peaks of fluorite were found around 1084 cm^{-1}. Comparing the results from Figs. 7(b) and 7(c), we found that there were similar peaks. C-O stretching vibration peaks appeared at around 1557 cm^{-1} (b) and 1674 cm^{-1} (c). CH_2 symmetrical stretching vibration peaks are observed at 2852 cm^{-1} (b) and 2850 cm^{-1} (c), and CH_3 antisymmetric stretching vibration peaks are observed at 2925 cm^{-1} (b) and 2922 cm^{-1} (c). The peaks around 1455 cm^{-1} (b) and 1551 cm^{-1} (c) corresponds to $Ca(RCOO)_2$ precipitate, confirming the chemical adsorption of collectors on fluorite. The peak at 2357 cm^{-1} (c) may be attributable to S-H stretching vibration. The new peak around 3359 cm^{-1} (c) is due to the associated O-H stretching vibration, indicating ESVO adsorbed on the fluorite surface by the hydrogen bond force.

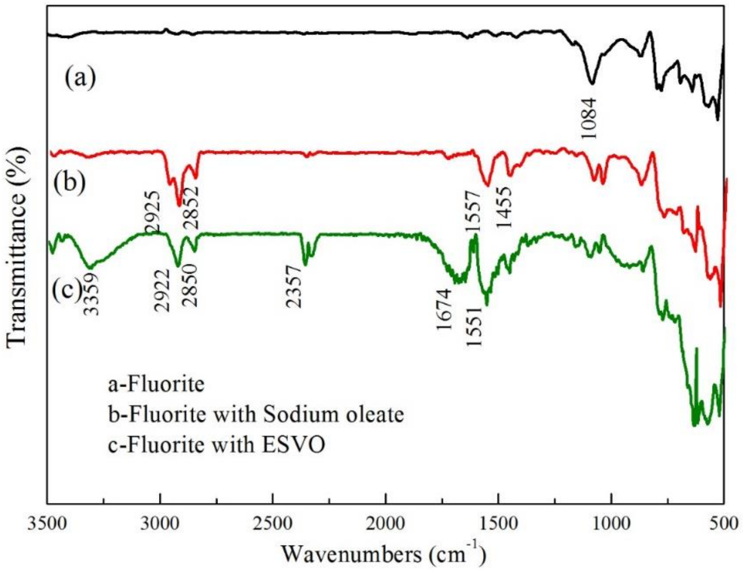

Fig. 7. FTIR analysis of fluorite absorbed with ESVO and sodium oleate

3.6 Particle-size distribution

Enhancement of the particle size has been proved to be effective in increasing the ultrafine mineral particles flotation (Cebeci, 2003; Yang et al., 2015; Shahbazi and Chelgani, 2016). As ESVO has the similar functional group as sodium oleate, and sodium oleate can be used as bridging reagents and stick hydrophobic particles together to form agglomerates (Sönmez and Cebeci, 2003a; Sönmez and Cebeci, 2003b), therefore the interaction between collectors and mineral can be expressed by size distribution of agglomerates. The effect of collector concentration on cumulative content of particles pass 20 µm and average size (d50) of fluorite agglomerates are studied. The results are shown in Fig. 8. We can see from the results, that with concentration increase from 0 to 900 g/Mg, the average size of agglomerates (d50) increased from 17.69 µm to 30.29 µm when sodium oleate was used as the collector. With the presence of ESVO d50 increased to 31.91 µm, i.e. the difference is only slight. However, in presence of ESVO, the cumulative content of fluorite particles agglomerates decreased faster comparing to sodium oleate.

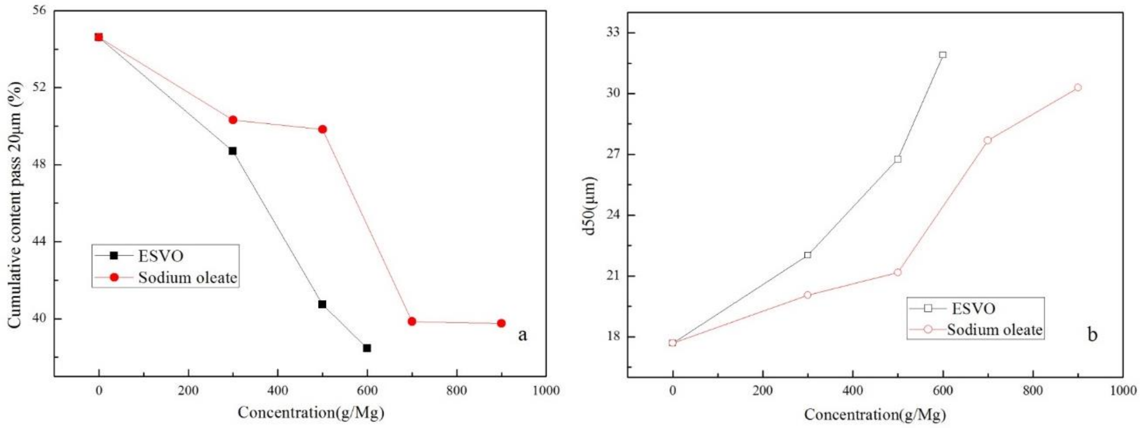

Fig. 8. Effect of collector concentration on cumulative content pass 20 μm (a) and d50 (b) of fluorite agglomerates

4. Conclusions

In this paper, the emulsified sodium vegetable oleate (ESVO) has been proven to be a good alternative to the sodium oleate collector used in fluorite flotation at room-temperature. The flotation recovery of fluorite as a function of pH and collector concentration was studied. Flotation results showed that the recovery of fluorite can reach 94% at pH 6 using ESVO at the concentration 600 g/Mg. In the case of sodium oleate, the recovery was 86% at pH 8 and the collector concentration equal to 900 g/Mg. The FTIR analysis indicated that ESVO main functional group is $-COO^-$, similar to sodium oleate. The surface tension results demonstrated that ESVO had smaller surface tension values than sodium oleate, which can be the reason for flotation recovery increase. Moreover, the mechanism of collector interaction with the fluorite surface was measured by the zeta potential, FTIR spectra and laser grain-size test. Results of zeta potential tests demonstrated that ESVO can change the fluorite surface potential through electrostatic attraction and chemical adsorption. Meanwhile, the ESVO can be adsorbed on the fluorite surface in the form of $Ca(RCOO)_2$ precipitate, which was demonstrated by FTIR tests. The laser grain-size measurement showed the cumulative content of particles pass 20 μm decreased faster using ESVO as the collector than sodium oleate. Since the vegetable oleate was produced from the waste straw, the application of ESVO in flotation had significant influence on the environment protection, recycling of waste and flotation cost reduction.

Acknowledgements

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