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AN INVESTIGATION INTO BENEFICIATION OF JORDANIAN EL-LAJJUN OIL SHALE BY FROTH FLOATATION

A. Y. AL-OTOOM*

Department of Chemical Engineering
Jordan University of Science and Technology
P.O.Box 3030, Irbid 22110, Jordan

Oil shale is available in Jordan in large quantities with high proportion of minerals. This study presents an investigation in order to enhance oil recovery from Jordanian El-Lajjun oil shale, which represents a major local deposit area. This enhancement was attempted through decreasing the content of minerals by the method of froth floatation using different floatation and frothing agents. The effect of the type of agents, the concentrations of these agents, and the effect of particle size were also investigated. Jordanian El-Lajjun oil shale has shown a poor separation propensity (floatability). Particle size and type of frother did not have any impact on the efficiency of oil shale recovery. A maximum recovery of 3% was achieved when using fuel oil as a collector and methyl isobutyl carbinol (MIBC) as a frother in the froth floatation process. It is believed that kerogen causes wetability of oil shale particles during preparation of oil shale hindering floatability of oil shale. On the other hand, phosphates and calcite were found to be easily separable from Jordanian oil shale using froth floatation. A degree of separation of more than 99% has been achieved when using kerosene as a collector and MIBC as a frother.

Introduction

Oil shale in Jordan is considered the most promising fossil fuel resource which is distributed throughout the country, particularly in the central and southern parts of the country. The estimated national reserve of oil shale is approximately 50 billion tonnes [1].

The future of utilizing Jordanian oil shale is still to be predicted owing to the major differences between this oil shale and oil shales around the world. Although oil shale is being used extensively in several countries, its local use

* Corresponding author: e-mail *awni_otoom@just.edu.jo*

is still in the research phases. Low calorific value of oil shale and high share of minerals make its utilization a real challenge.

Floatation is a separation process that depends on the difference in hydrophobicity and hydrophilicity scale of materials. Froth floatation is a process used to selectively separate hydrophobic minerals suspended in a solution by attaching them to gas bubbles and transferring into the froth layers. This is achieved by the use of surfactants and wetting agents. It is considered the cheapest and most extensively used method for separation of valuable minerals.

Floatation processes employ several chemicals mainly frother, collector, and modifiers. Frothers are normally surface active agents that aid in the formation and stabilization of air-induced floatation froths. Collectors are also surface active agents that are added to the floatation pulp, where they selectively adsorb on the surface of the particle and render them hydrophobic properties. Modifiers are used to modify the surface of constituents so that it does not adsorb the collector or promoter.

Froth floatation of coal has been extensively studied and found to be feasible in enhancing the quality of coal by removal of unwanted minerals such as pyrite which is easily separated. The effect of different operating parameters on froth floatation has been studied extensively. These parameters include froth depth, frother concentration, solid density, amount of hydrophobic particles in the feed on entrainment [2–4]. The effect of particle size was also investigated, it was shown by many researchers that the smaller the particle size, the better the recovery of coal [5]. Different investigators have found that kerosene is on of the best collectors that can be used for coal froth floatation [6].

Very few studies have been completed on floatation of oil shale since the floatability of shales was found to be a difficult process [7]. The present study aims to find the degree of separation of minerals from an oil-rich matrix or the floatability of Jordanian oil shale obtained from El-Lajjun deposits for the purpose of beneficiation. It is also known that Jordanian oil shale locates between phosphate beds [8], therefore, the floatability of oil shale and phosphates and calcite is also studied.

Experimental

Experimental setup

A Denver DR floatation machine was used to perform froth floatation. This is an example of a typical froth floatation unit used in the mining industry. The schematic diagram is presented in Fig. 1. The pulp is introduced through a feed box and distributed over the entire width of the cell. The pulp comes into contact with the impeller, where it is subjected to intense agitation and aeration. The compressed air enters the cell by impeller rotation.

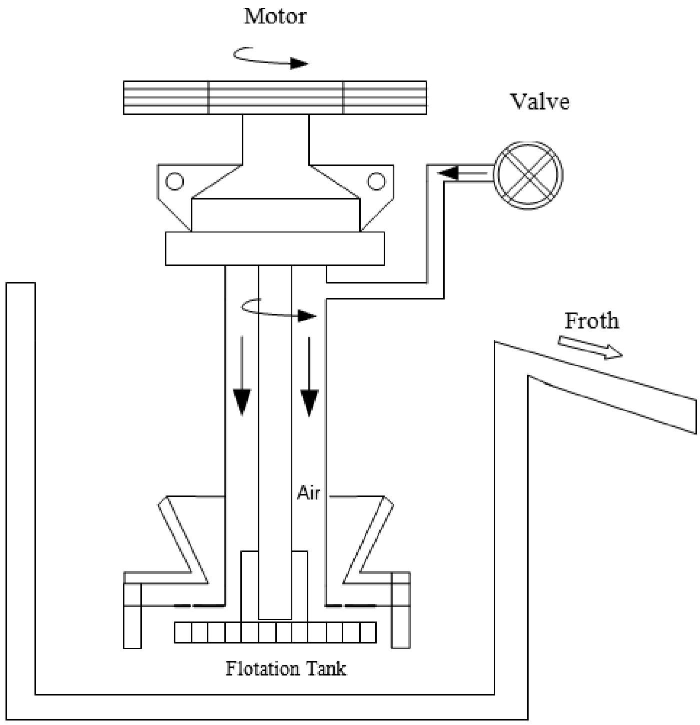

Fig. 1. Schematic diagram of the floatation cell.

Samples of El-Lajjun oil shale were provided by the Natural Resources Authority. The data of proximate analysis are as follows (wt.%): moisture – 1.1; volatile matter – 44.0; ash – 54.5; fixed carbon – 0.4. Ash of this oil shale contains (determined by XRF, wt.%): SiO_2 – 26.6; Al_2O_3 – 7.2; Fe_2O_3 – 2.9; CaO – 43.4; MgO – 0.8; Na_2O – 0.3; SO_3 – 9.1; P_2O_5 –5.9.

Floatation of pure oil shale

Experimental

A full factorial analysis was used to determine the most effective parameters influencing the floatation process of El-Lajjun oil shale (Table 1). The main factors emphasized in this analysis are the mean particle size ranging from 55 μ to 375 μ, the effect of pH, and collector to frother ratio. The effect of different floatation agents was also investigated.

Chemicals and reagents

MIBC (methyl isobutyl carbinol), a widely used frothing agent, was used. Floatation agents kerosene, DMSO, calcium lignosulfonate, sodium lignosulfonate, sulphonated naphthalene, diethyl ketone, and Fuel oil #2 were used as collectors.

Table 1. **Full factorial experimental design of floatation of pure oil shale**

Experiment	Pattern of variables	Mean particle size, μm	Collector/ Frother	pH	Mass percent of froth	Ash percentage of froth
1	– – –	375	∝	4	73.8	54.6
2	– – +	375	∝	7	72.2	54.3
3	– + –	375	0	4	74.1	54.4
4	– + +	375	0	7	73.3	53.89
5	+ – –	55	∝	4	74.5	53.95
6	+ – +	55	∝	7	73.8	53.7
7	+ + –	55	0	4	73.6	54.1
8	+ + +	55	0	7	74.6	53.87

Experimental procedure

A sample of oil shale was weighed (150 g) and placed in a beaker which contains the solution of the solvent (water), collectors and frother. Samples were kept for conditioning for a period of 10 minutes. The total volume of the solution was 1 liter.

Thereafter the solution was placed in the floatation cell, air flow rate was adjusted to 2 L/min, and the rotation speed of the impeller was kept at 1000 Rev/min. Frother was continuously collected from the cell into a beaker for a period of 30 minutes. Tailing and froth were collected, washed with distilled water, vacuum filtered and then dried in an oven at 105 °C.

Floatation of oil shale and phosphate

Samples of calcium phosphate were added to the oil shale sample (about 15 g per 150 g oil shale) and the previous procedure was repeated to find the distribution of the phosphate in froth and tailing. The froth from these experiments was floated again using the same procedure to ensure full recovery of oil shale from phosphate deposits.

Floatation of oil shale and calcite

Samples of calcium carbonate (calcite) were added to the oil shale sample (15 g per 150 g oil shale). The froth from these experiments was floated again using the same procedure to ensure full recovery of the oil shale from calcite.

Dried samples of tailing and froth were weighed, placed in silica crucibles and heated to 500 °C in 30 minutes, thereafter heated to 815 °C in 60 minutes and kept at this temperature for 3 hours until the mass remained constant. The amount of ash left was calculated basing on the mass difference. The amount of ash left is taken for indicator of the amount of enhancement or the recovery of organic material by floatation.

Results and discussion

The full factorial experimental design (Table 1) shows the major variables that are believed to affect oil shale recovery. Kerosene was used as a collector and MIBC as a frother. The effect of particle size and acidity of the solution were also studied. The results given in the table show that none of these parameters enhances oil shale recovery since ash contents in the froth and tailing are almost constant at 54% which is similar to the ash content of the original oil shale samples.

The further investigation on the type of the collector was performed to test the recovery of the organic matter. As shown in Table 2, changing the type of the collector did not change the recovery of oil shale much. Slight improvement in the oil shale recovery was noticed when using fuel oil #2 as a collector. The maximum enhancement was approximately around 3% achieved in two stages froth floatation as shown in Table 3. It is considered a low recovery rate that might not justify the investment in floatation units.

Images obtained by scanning electron microscope for El-Lajjun oil shale, as shown in Fig. 2, indicate that kerogen is encapsulated inside a shell or minerals (normally calcite). Therefore, separation of oil-rich minerals is believed not to be possible since wetting of the kerogen by the collector is hindered by the layer of the minerals that encapsulate it.

Although the recovery of oil shale using fuel oil as a collector is relatively low, fuel oil is believed to attract more of kerogen-rich particles to the froth layer.

Table 2. **Effect of different collectors on floatability of oil shale (mean particle size 55 μm, frother MIBC, pH 7)**

Experiment	Collector	Mass percent of froth	Ash percentage of froth
9	DMSO	73.9	54.1
10	Sulphonated naphthalene	73.7	54.0
11	Sodium lignosulfonate	73.0	54.1
12	Calcium lignosulfonate	71.9	54.0
13	Diethyl ketone	73.9	54.6
14	Fuel oil #2	72.9	52.8

Table 3. **The effect of multiple stages on the floatability of oil shale (mean particle size 55 μm, frother MIBC, collector – fuel oil #2, pH 7)**

Experiment	Number of floatation stages	Mass percent of froth	Ash percentage of froth
14	1	72.9	52.8
15	2	98.0	52.3
16	3	100	52.3

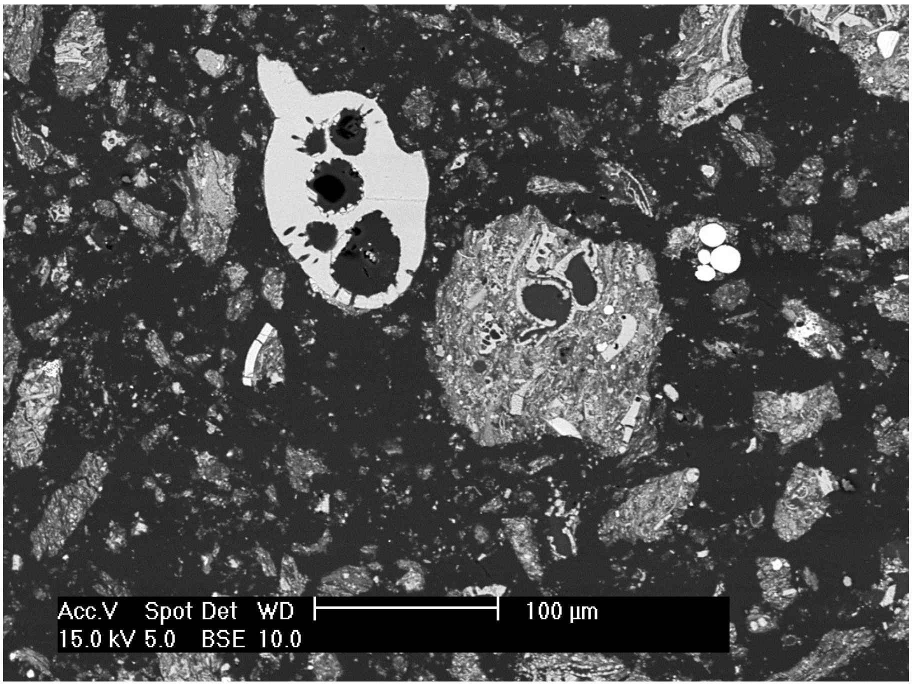

Fig. 2. Scanning electron microscope image of El-Lajjun oil shale.

It was noted that around 73% of original oil shale has moved to the froth side, while around 27% remained in the tailing part. This can be explained by the fact that during crushing of oil shale samples, kerogen has wetted most of oil shale making it hydrophobic. Since that hydrophobic material moves into the froth layer, it is expected that the most part of oil shale will behave in this way. This also can explain the low recovery of oil-rich particles in the froth.

The floatation of oil shale from a mixture of oil shale and phosphate has shown a high recovery of oil shale in the froth since most of the phosphate has stayed in the tailing part. High recovery was achieved using kerosene as a collector and MIBC as a frother. A 99.9% separation of phosphate was achieved after two stages of froth floatation. After the first stage, the mass percentage of froth was 71 and the mass percentage of phosphate in froth was 1.3. After the second stage these numbers were 98.6 and 0.1, respectively (experiments 17 and 18). Such a high recovery rate indicates the big difference in the hydrophobicity of oil shale and phosphate.

Similarly, floatation of oil shale in a mixture of oil shale and calcite has shown a high recovery of oil shale in the froth layer. Kerosene was used as a collector and MIBC used as a frother. After the first stage mass percentage of froth was 72 and mass percentage of calcite in froth was 2.8. After the second stage of floatation these numbers were 97 and 0.3, respectively (experiments 19 and 20).

Conclusions

Floatability of Jordanian oil shale has been studied and the effects of different factors (type of collector, pH level, particle size, and concentration of frother and collector) have been investigated. In addition, separation of phosphate and calcite minerals has been investigated.

Floatation of oil shale has shown a poor recovery of oil-rich components with a maximum of 3% enhancement when using fuel oil #2 as a collector. This poor recovery is believed to be due to the fact that kerogen is encapsulated in layers of minerals and that during crushing and milling kerogen wets most of oil shale particles making it difficult to adjust surface properties.

The separability of phosphate from oil shale using froth floatation has been demonstrated to be effective using kerosene, since 99.9% of the phosphate can be separated in a two-stage floatation process. Calcite can also be separated using froth floatation. The results have indicated that 99.7% of calcite can be separated in a two-stage floatation process.

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