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## Impact of cationic-anionic surfactants on selective oil agglomeration of oil shale

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**Abstract:** Egyptian oil shale from Red Sea area is upgraded via oil agglomeration technique to achieve a clean fuel. A representative sample is characterized to identify its undesirable components and its liberation size. The quartz, apatite, calcite, siderite and anhydrite are the main gangue minerals. The sample was pulverized to less than 20  $\mu\text{m}$  for efficient liberation. The impact of anionic (Calcium dodecyl benzene sulfonate, CDBS) or/and cationic (Cetrimonium bromide, CTAB) surfactants on the zeta-potential and agglomeration process was investigated in presence of different kerosene concentrations. A concentrate of 62% kerogen with 95% recovery was obtained from feed of 29% kerogen using 0.1% CTAB/CDBS mixture in 2% kerosene emulsion.

**Keywords:** oil shale, oil agglomeration, kerogen, surfactants, zeta potential

### 1. Introduction

Oil shale is a sedimentary rock that contains insoluble kerogen as a combustible organic component. It is considered as the second largest fossil fuel deposit worldwide (Tissot and Welte, 1984; Hruljova et al., 2013; Al-Thyabat et al., 2014). Recently oil shale is assumed to serve as alternative energy due to great lack of energy (Larsen et al., 2000; Savest et al., 2009). The structure of shale is porous with a complex heterogeneous mixture of organic and inorganic components. Compared to coal, shale organic component is more susceptible to be converted into oil (Lille, 2003) Kerogen represents approximately 10-35% by weight of oil shale which is decomposed efficiently and yielded oil upon heat treatment.

Different physical and physicochemical techniques were applied on oil shale to recover kerogen due to its simplicity, flexibility and low operational costs of the process. Heavy liquid techniques upgraded shale with low kerogen recovery efficiency 14%-50% (Larson et al., 1981). Flotation and oil agglomeration techniques were applied to upgrade fine shale particles, less than 150  $\mu\text{m}$ , and a kerogen recovery efficiency of 70% was obtained (Luo and Huy, 2013; Qiao, 2013).

Oil agglomeration gets a great importance as a technology for coal and shale treatment as it is reported that oil agglomeration technique is superior to flotation for delivering higher combustible recoveries (Abdel Khalek and Parekh, 2001; Polowczyk et al., 2018). The oil agglomeration was extensively utilized for fines aggregation and minerals selective separation like coal treatment however its application on kerogen recovery from oil shale is limited (Polowczyk et al., 2018; Özer et al., 2017; Bokanyi and Csoke, 2003; Ceylan and Kucuk, 2004). Oil agglomeration could be defined as "aggregation of hydrophobic low sized particles in the existed bridging oil that is distributed into the suspended aqueous solid particles". Droplets of oil became wet and bridge hydrophobic particulates, creating aggregates. Enlargement of size for established aggregates is called "oil agglomeration" and is formed due to collision of inter particles, adhesion of particles coated by bridging liquid and creation of stable aggregate (Polowczyk et al., 2018; Özer et al., 2017).

Better agglomeration performance could be achieved via mixing two different surfactant types. This is resulted from strong electrostatic interaction between shale particles and kerosene droplets

improving their adhesion. It refers to synergism between cationic and anionic surfactants during oil agglomeration. Also, surfactant existence during bridging oil emulsification causes smaller diameter of oil droplets and resulting in larger agglomerates (Bokanyi and Csoke, 2003). Polowczyk et al. reported that the carbonaceous metal-bearing shale could be agglomerated using kerosene with cationic-anionic surfactants mixture. Generally, the oil agglomeration is governed by hydrophobicity and electrostatic interactions (Polowczyk et al., 2018).

Egypt possesses huge estimated oil shale resources at Red Sea region of approximately 15 billion tons (Yehia et al., 2017). The challenge of using these resources, as alternate source of energy, is to achieve an environmentally clean fuel with favorable characteristics in terms of combustion quality. Although, the Egyptian oil shale was concentrated using both falcon concentrator as enhanced gravity separator and column flotation, however they resulted in moderate kerogen grade and recovery. A concentrate of 42% kerogen with 94% recovery was produced using Falcon SB40 concentrator at 60 Hz and water pressure of 4 Psi (Yehia et al., 2017). A concentrate of 38% kerogen with 88.5% recovery was obtained using column flotation in presence of kerosene as a collector and MIBC as a frother (Sobhy et al., 2019).

This work aims to investigate the upgrading of Egyptian shale using oil agglomeration technique to achieve a clean fuel. A characterization study would be applied to identify its undesirable components and its liberation size. The impact of anionic (Calcium dodecyl benzene sulfonate, CDBS) or/and cationic (Cetrimonium bromide, CTAB) surfactants on zeta-potential and agglomeration process in presence of different kerosene concentrations would be studied.

## 2. Materials and methods

### 2.1. Materials

A representative shale sample from Red Sea locality, Wadi El-Nakhil deposit, was pulverized to less than 20 microns via attritor ball mill. A purified kerogen was employed for zeta potential measurements. The purification process was achieved via removing Bituminous content by solvation in benzene-methanol mixture using soxhlet apparatus. Carbonates as well as iron salts were removed by solvation in 1:1 HCl solution. SiO<sub>2</sub> was evaporated by heating residues with HF acid in platinum crucible. Finally, iron sulfide was removed by refluxing with LiAlH<sub>4</sub> for 1hr (Looney et al., 2010).

Calcium dodecyl benzene sulfonate (CDBS, C<sub>36</sub>H<sub>58</sub>CaO<sub>6</sub>S<sub>2</sub>, MW= 691.05) and Cetyl-trimethyl ammonium bromide (CTAB, CH<sub>3</sub>(CH<sub>2</sub>)<sub>15</sub>N(Br)(CH<sub>3</sub>)<sub>3</sub>, MW=364.45) as anionic and cationic surfactants were purchased from MERK. Kerosene (Sp.gr. 0.8 g/cm<sup>3</sup>) as a bridging oil was used for shale agglomeration. All reagents and surfactant solutions were prepared in ultra-pure water.

### 2.2. Methods

Complete chemical analysis of shale samples was determined using X-ray fluorescence. Kerogen was determined according to Fischer assay (Fischer and Schrader, 1920). X-ray diffraction system Cu K $\alpha$   $\lambda=1.5 \text{ \AA}$  radiation (Philips PW1730) was used to identify shale morphology.

A laser zeta meter, Malvern Zeta-sizer 2000, was used for zeta potential analysis. A suspension of 0.02 g purified kerogen with 50 ml 0.01 M KCl solution, with kerosene or/and surfactants, was conditioned at desired (pH 3-10) during 5 minutes and at room temperature. Then, it was transferred to electrophoresis cell. Kerosene zeta potential was measured via mixing a small oil quantity with 10 ml of aqueous solution of ionic surfactants or water. Zeta potential values were determined as an average of three successive measurements.

Oil agglomeration tests were carried out by adding 20 g shale sample to 50 cm<sup>3</sup> Kerosene emulsion and/or surfactant solution, in a "Kitchen" blender and agitated at 2000 rpm during 5 min. The mixing speed was then reduced to 500 rpm for another 5 minutes, to allow growing of agglomerates. The product was poured on a screen, 150  $\mu\text{m}$ , to separate the agglomerates. The effect of kerosene or/and surfactants was studied. All experiments were performed at natural pH (Uwadiale, 1996). Results of both kerogen recovery and grade were obtained as an average of duplicate experiments and error analysis was estimated as  $\pm 0.60\%$  for kerogen recovery% and  $\pm 0.15\%$  for kerogen%.

### 3. Results and discussion

#### 3.1. Characterization of oil shale

Complete chemical analysis displays that the sample has high amount of inorganic constituent (57.79%), with high loss of ignition (42.2%) due to presence of organic matter and inorganic carbonates, Table 1.

Table 1. Complete chemical analysis of oil shale sample

Constituent	SiO <sub>2</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	L.O.I	Total
Weight %	18.89	20.84	4.02	3.48	3.08	7.48	42.2	99.99

X-ray diffraction of shale sample shows that the main associating impurities are quartz (SiO<sub>2</sub>), siderite (FeCO<sub>3</sub>), anhydrite (CaSO<sub>4</sub>), calcite (CaCO<sub>3</sub>), kaolinite (Al<sub>2</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>) and apatite (Ca<sub>5</sub>(PO<sub>4</sub>)<sub>3</sub>OH) minerals, Fig. 1.

The microscopic investigations show that shale matrix is consisted of lamina. Laminations are usually flat but occasionally undulant and distorted around tests and detrital grains. The alternating thickness of lamina is ranged 10–50 μm. Lamina is consisted mainly of argillaceous material that is rich in organic matter. Other associated minerals are quartz, pyrite, carbonate and phosphate as cavity filling or dispersed within the matrix, Fig. 2.

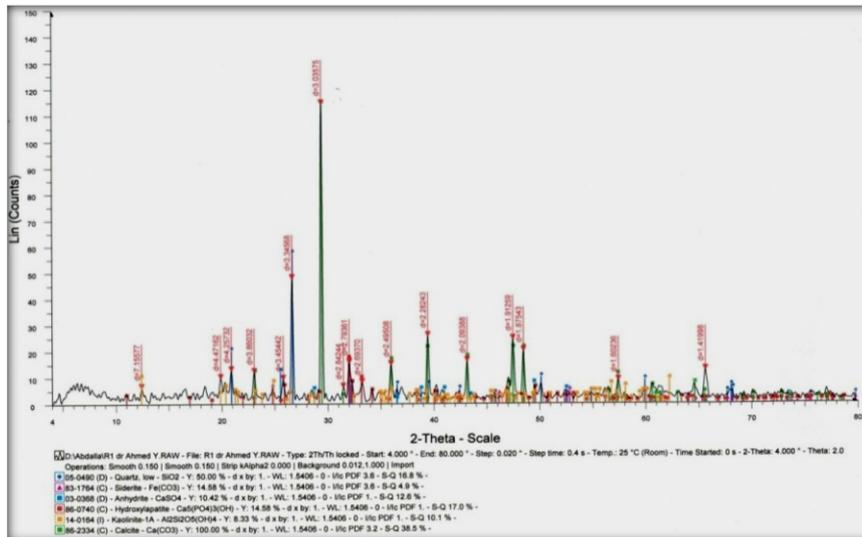


Fig. 1. XRD pattern of shale sample

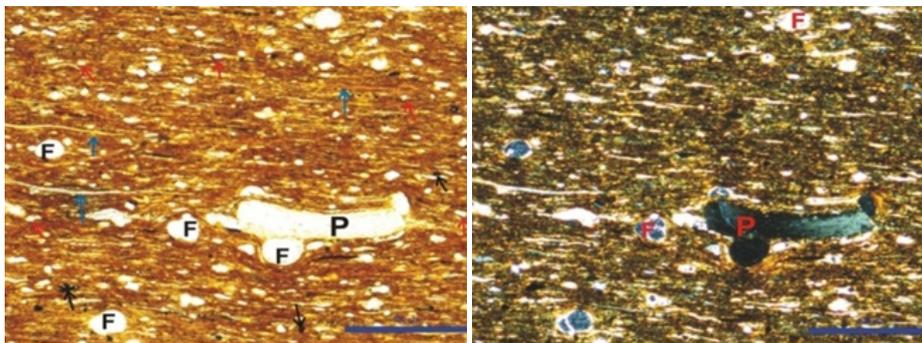


Fig.2. Microscopic images of shale sample

#### 3.2. Effect of kerosene and surfactants on zeta potential

The zeta potential (surface charge on kerogen particles) gives an expectation about the hydrophobicity which is an important factor in aggregation. Zeta potential of kerogen particles as a function of pH is represented in Fig. 3. It is showed that kerogen surface has a negative charge in water or kerosene

emulsion. The negativity is increasing with increasing alkalinity, while it is decreased in kerosene. The decrease of kerogen surface negativity is due to spread of kerosene on carbon surfaces which mask its hydrophilic character (Laskowski and Yu, 2000; Han et al., 2020).

The negativity is decreased in cationic surfactant CTAB and in its mixture with kerosene. Surfactants reduce the energy barrier in the droplet-kerogen shale particles. It turns positive over pH 6 for CTAB and over pH 4 for CTAB/Kerosene mixture. The cationic surfactant turns kerosene droplets positive (Polowczyk et al., 2018). Although adsorption of polar species on negative surfaces is occurred through many mechanisms, the adsorption of CTAB and CDBS on kerogen surface refers to the formation of H-bond or ecstatic attraction (Polowczyk et al., 2018). The adsorption of CTAB may be occurred by formation of H-bonds between its nitrogen and bi-valent cations. Also, positive  $R-NH_3^+$  groups interact with negative species in solution to form a precipitate on solid surface (Gao et al., 2015; Zhao et al., 2020).

For anionic surfactant CDBS and its mixture with kerosene, the kerogen surface exhibits negative zeta potential values over the whole range of pH. The negativity is increased with increasing pH. This is due to adsorption of sulfonate groups of CDBS by ecstatic attraction with calcium ions species on the kerogen (Gao et al., 2015). The anionic surfactant increases the negativity of kerosene droplets (Polowczyk et al., 2018).

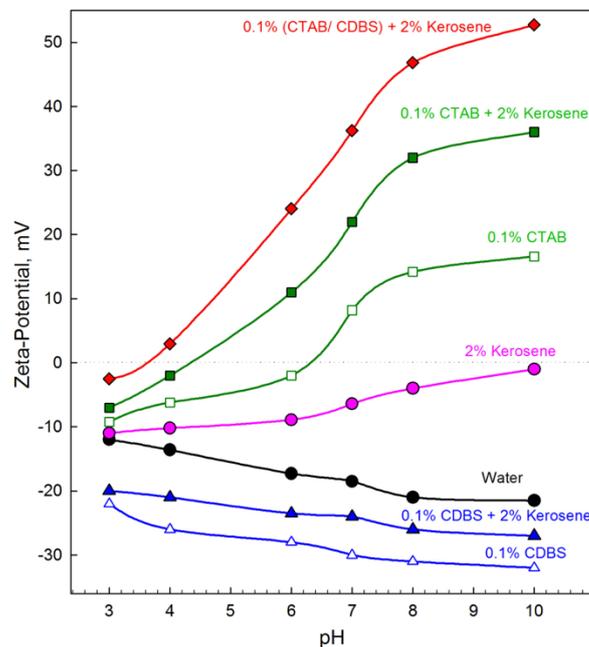


Fig. 3. Effect of surfactants on zeta potential of kerogen as a function of pH

Zeta potential is increased excessively as a result of conditioning with mixture of cationic-anionic surfactants and kerosene, Fig. 3. Mixing cationic and anionic types is expected to display higher effect on zeta potential (Rosen and Hua, 1982). It is stated that mixing of two different surfactant types arise a stronger effect than a single effect (Xiao et al., 2005; Kume et al., 2008). The resulting effect of mixing two types depends on the difference in their charge. Adsorption of surfactants on the negatively charged shale surface is a result of several interactions at the solid-solution interface and is mainly governed by the structure of surfactant molecules i.e. type of polar head, structure and length of hydrocarbon chain, but also the surface characteristics of shale. Kerogen exhibits chemical heterogeneity due to presence of many different functional groups and pore structures on its surface. Its complex mineralogy, structure, roughness, surface charge and heterogeneity have a strong impact on the adsorption density of ionic surfactants and thus hydrophobic coagulation (Piestrzyński and Pieczonka, 2012; Rahfeld et al., 2018).

### 3.3. Effect of kerosene and surfactants on agglomeration

The effect of kerosene concentration displayed an increase of recovery to 90% with increasing kerosene concentration to 4%. This could be explained as follows; at low kerosene concentration, only swinging

bridges are formed between particles and a consolidated two-dimensional flock structure exists, Fig. 4 (Hand Book, 1991). On the other hand, the grade (organic content) decreases relatively from 44% to 37% with increasing kerosene concentration, Fig. 5. This is resulted from selective adsorption of kerosene on kerogen surface at low concentration. As more kerosene is added, the flocks are grown causing impurities trap (Hand Book, 1991).

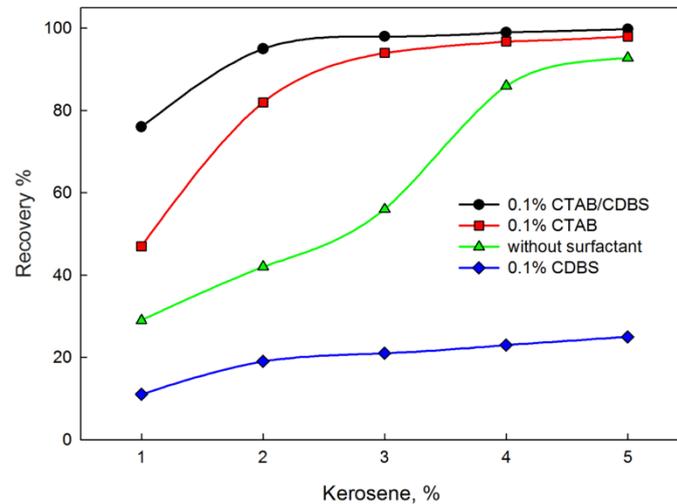


Fig. 4. Effect of surfactants addition on kerogen recovery in agglomerates at different kerosene concentrations, the error analysis was estimated as  $\pm 0.60\%$

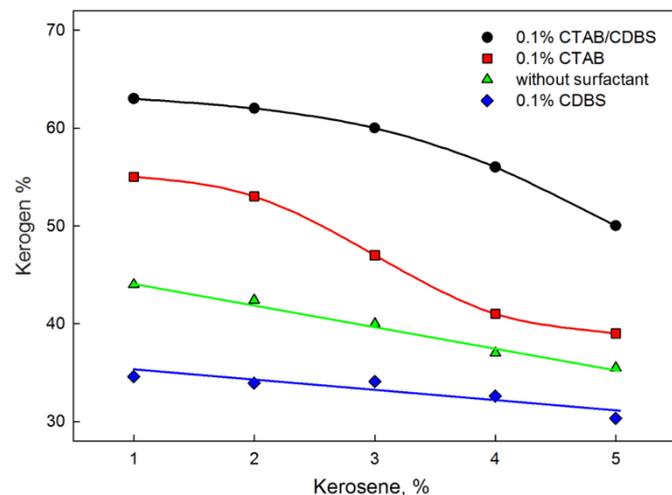


Fig. 5. Effect of surfactants addition on kerogen grade in agglomerates at different kerosene concentrations, the error analysis was estimated as  $\pm 0.15\%$

Surfactants reduce the interfacial tension lower than its critical value (Cebeci and Sönmez, 2004; Ozkan et al., 2005). Therefore, the addition of 0.1% surfactant to kerosene may affect the recovery and grade values. Agglomeration is affected by the surface charge of both kerogen particles and kerosene collides. In absence of surfactant, the negative kerogen particles cannot agglomerate easily with negatively charged kerosene collides. The presence of cationic surfactant CTAB turns kerogen into positively charged particles and hence it is attracted with negative kerosene by ecstastic interactions. Thus, stable agglomerates are expected (Polowczyk et al., 2014; 2015). Kerogen recovery is much better in the presence of 0.1% CTAB than that for kerosene only due to decease of kerosene droplets. As the kerosene consumption needed for efficient oil agglomeration is reduced (Laskowski and Yu, 1998).

The cationic surfactant gives relatively higher recovery than that of anionic one. This is may be due to oxidation of kerogen surface (Laskowski and Yu, 2000). There is no aggregation in the presence of CDBS even with its mixture with kerosene. The CDBS decreases the surface hydrophobicity and

interfacial tension. Bastrzyk et al., (2011) explained that; as a result of low interfacial tension, the agglomerates were unstable as a result of small liquid bridges strength between particles. This is evidenced by zeta potential measurements, Fig. 3. The anionic surfactant increases the negative charge of kerogen surface even in presence of kerosene as it increases the negativity of kerosene droplets (Polowczyk et al., 2018).

As stated above, the mixture of different surfactant types gives stronger effect than a single effect. (Xiao et al., 2005). Mixed cationic-anionic surfactants are often unstable and various structures are formed e.g. vesicles, coacervates, rod-like micelles and precipitates (Kume et al., 2008).

Mixed surfactant system resulted in better agglomeration performance due to strong electrostatic interactions between kerogen particles and kerosene droplets enhancing their adhesion. This is a result of synergism between cationic and anionic surfactants. As surfactant is located at the kerosene/water interface, its hydrocarbon tails are directed towards kerosene while the hydrophilic portions are facing the aqueous solution. A successful agglomeration is achieved when solid particles are being wet to some extent. Although zeta potential is positive, a reasonably good agglomeration performance is possible when the magnitude of change in zeta potential is high (Drzymala, 2007). This is may be due to increasing of Turbiscan Stability Index (*TSI*), (Drzymala, 2007; Duzyol and Ozkan, 2010).

In the presence of 0.1% CTAB/CDBS mixture, the recovery is reached up to 95% with 62% kerogen at 2% kerosene. The best recovery of 98% is obtained with 60% kerogen at 3% kerosene. While CTAB/Kerosene system resulted in a product of 53% kerogen with 82% recovery.

The chemical analysis of final product (62% kerogen) is compared with original sample (29% kerogen), Table 2. Inorganic components are reduced by 70% while ignition loss, due to organic and carbonate contents, is increased from 42.2% in the feed to 82.6%.

Table 2. Chemical analysis of the final product (62% kerogen) and original sample

Constituent, %	SiO <sub>2</sub>	CaO	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	L.O.I	Total
Feed Sample	18.89	20.84	4.02	3.48	3.08	7.48	42.2	<b>99.99</b>
Concentrate	5.65	6.27	1.62	1.05	0.89	1.86	82.6	<b>99.94</b>

#### 4. Conclusions

Egyptian oil shale, from Red Sea area, is composed of high amount of inorganic constituent (57.79%) with high loss on ignition (42.2%). It was found that the shale matrix is composed of lamina of thickness between 10–50 μm. The main associated minerals are quartz, selerite, anhydrite, calcite, kaolinite and apatite. They are filling cavities or dispersed within the matrix.

The kerogen as the main organic component of the oil shale has a negative surface charge in water or kerosene emulsion. The negativity is turned positive in presence of CTAB as a cationic surfactant. The upgrading of oil shale via oil agglomeration with kerosene gives poor concentrate even up to 5%. While the addition of 0.1% cationic surfactant give better concentrate at lower kerosene consumption.

Although, the cationic-anionic surfactants mixture with kerosene increases the zeta potential excessively, better agglomeration performance arises with mixed surfactants system. A concentrate of 95% recovery and 62% kerogen is obtained using 0.1% CTAB/CDBS surfactant mixture in 2% kerosene emulsion.

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