

Removal of Oil and Heavy Metals from Oily Sludge using Esterquat Surfactant via Surfactant-Enhanced Oil Recovery (SEOR)



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Abstract: Oily sludge is one of the most significant solid waste materials generated by petroleum refinery plant. Due to high concentration of petroleum hydrocarbon (PHCs) that is considered as harmful and hazardous waste to human beings and environmental, the initiative was developed to minimize this issue by performing investigation on cationic plant-based esterquat surfactant for oil recovery and heavy metal removal from oily sludge via Surfactant-enhanced Oil Recovery (SEOR). Based on the research finding, oily sludge has significant amount of volatile matters (32.22 wt%) which consists of hazardous PAHs that needs to be removed from oily sludge. Cationic esterquat surfactant has been introduced in removal of heavy metal and oil from oily sludge. Result shows the CMCs of cationic esterquat surfactant obtained was about 125mg/L via conductivity method and spectrophotometric method. The percentage of oil recovery obtained was 74.62% at esterquat concentration of 125 mg/L with significant removal on chromium (Cr) and lead (Pb). This finding shows the potential of cationic esterquat surfactant in enhancing the oil and metal removal from oily sludge.

Keywords: Oily sludge, wastewater treatment, esterquat, surfactant technology.

I. INTRODUCTION

Since last decades, petroleum industries are known to be as one of valuable non-renewable source as the importance of oil is increased globally. The oil products are very significant as source of incomes for many countries in the world. However, the waste produced from petroleum processing and its derivatives leads to a serious problem worldwide. The vast amounts of oily sludge which is listed as most significant solid waste generated from a variety of sources, including on-site

wastewater treatment pond which cause a problem due to high costs in terms of treatment and disposal methods [1]

Among environmental contaminants produced from petroleum industry, petroleum accounts for approximately 70% of the total environmental pollution due to production of high volume of oily sludge during drilling process, storage, transportation or refining of petroleum to obtain crude oil products. As for Malaysia, the quantity of waste oil or oily sludge generated by 2013 for about 5277.57 metric tonnes per year and the quantity of waste of oil sludge from oil refinery accounts for about 935.81 metric tonnes per year [2]. Since the oily sludge waste is categorized as scheduled waste, the residuals generated from recovery of material or product from these oily sludge wastes intend to be treated or disposed at prescribed premises as stipulated in Environmental Quality Act (EQA) 1974 [3] by Department of Environment (DOE) Malaysia.

Since the effective remediation of oily sludge has a become global issue due to its hazardous in nature, various technologies of oily sludge treatment have been explored. Guo, Li, Qu and Liu [4] reported about 80% of total organic carbon content (TOC) in oily sludge can be converted into usable hydrocarbons via pyrolysis process. However, the ash residue produced from pyrolysis process might contain hazardous heavy metals. Apart from that, the use of chemical surfactants by Abdel Azim, Abdul-Raheim, Kamel and Abdel-Raouf [5] was able to break down the petroleum sludge emulsion and more than 80% of water can be separated from oily sludge. However, the use of chemical surfactants leads to environmental toxicity since it resists to biodegradation. Yan, Lu, Yang, Zhang, Zhang and Chen [6] found that rhamnolipid biosurfactant can recover up to 91.5% of oil recovery and functioned as effective cleaning agents for oily sludge. However, least study reported on the potential of biosurfactant in removal of heavy metals from oily sludge. Therefore, there is a need to investigate the potential of plant-based esterquat surfactant in removing oil and heavy metals from oily sludge. Utilization of esterquat surfactants possesses numerous advantages in removing organic pollutants from solid particles because of their degradability, compatibility, low toxicity and low cost. Surfactant is an amphiphilic compound in which consists of a hydrophilic head and a hydrophobic tail in its molecule [7]. Contrary to hydrophobic tail, the hydrophilic head shows more affinity to the bulk medium.

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The hydrophilic head enable the surfactant to solubilize in water due to its ionic or highly polar head group [8] whereas the hydrophobic tail tends to concentrate at interfaces [9].

The research work was investigated on the potential of cationic plant-based esterquat surfactant in removing of oil and heavy metals from oily sludge via Surfactant-enhanced Oil Recovery (SEOR) process. The study began with the characterization of plant-based esterquat surfactant and oily sludge via FTIR analysis. The proximate analysis was conducted via Thermogravimetric Analysis (TGA) to determine the quantity of volatile matters content in oily sludge sample. The investigation on critical micelle concentration (CMC) of esterquat surfactant was performed using conductivity method and spectrophotometric method. Lastly, the investigation on the effect of surfactant concentration via SEOR was observed based on performance of oil and heavy metals removals.

II. MATERIALS AND METHOD

A. Materials

The raw concentrated oily sludge (COS) waste was generated from wastewater treatment plant of petrochemical industry in Malaysia. The cationic plant-based esterquat surfactant was obtained from Stepan. Reactive Blue 4 (RB4) dye was purchased from Sigma Aldrich (M) Sdn. Bhd. The nitric and hydrochloric acid were obtained from Merck Millipore.

B. Characterization study of oily sludge and surfactant

i. Characterization of raw COS via Thermogravimetric Analysis (TGA).

The proximate analysis was conducted gravimetrically by using Thermogravimetric Analyzer (Mettler Toledo TGA/SDTA851°) to determine the moisture, volatile matter, ash and fixed carbon content in oily sludge sample [10],[11]. The purified nitrogen was used at temperature range from 30 to 600°C whereas the purified compressed air was used at temperature varied from 600 to 1000°C as purge gas. The flowrate of the purge used was 100 mL/min and the analysis were conducted at heating rate of 10°C/min⁻¹ using sample with approximate weight of 15 to 20 mg.

ii. Characterization of COS and esterquat surfactant via Fourier Transform Infrared Spectroscopy (FTIR).

The functional group of raw COS, esterquat surfactant and treated COS were analyzed using Fourier Transform Infrared Spectroscopy (FTIR) (Perkin Elmer Spectrum One).

iii. Determination the Critical Micelle Concentration (CMC) of esterquat surfactant via simplified colorimetric method.

The determination of CMC of esterquat surfactant was performed at room temperature (25°C±2°C). The simplified colorimetric method was carried out via UV-Visible Spectrophotometer (Perkin Elmer Lambda 750) in the presence of esterquat surfactant. 1 ml of 0.1 wt% RB4 solution (dye) was added into 50 ml of esterquat surfactant sample. The mixture of dye and esterquat was mixed for 10 minutes with stirring speed of 300 rpm by

using Digital Hotplate stirrer. The solution was then filtered using Whatmann filter paper to separate soluble precipitate produced during mixing process. The filtrate solution was analyzed via Lambda UV-Visible Spectrophotometer [12].

iv. Determination the CMC of esterquat surfactant via simplified conductivity method.

The concentration dependence of the specific conductivity of aqueous solution at 299K (26°C) was measured by using WTW 3420 Multimeter. The measurement of conductivity was carried out by placing 50 ml of esterquat surfactant solution in a beaker. The conductivity probe was dipped into the solution and held by using retort stand. The successive injection of deionized (DI) water was added onto the selected surfactant concentration respectively [13]. The solution was stirred by using MR Hel-Tech Digital Hotplate stirrer whereas the EKT Hei Con Temperature Control is used to maintain the temperature of surfactant solution. The conductivity value was measured and recorded after 5 minutes of injection to ensure the equilibrium attainment in the system [14].

v. Acid Digestion analysis via Inductively Couple Plasma – Optical Emission Spectroscopy (ICP-OES).

The inorganic content of oily sludge sample is analyzed via acid digestion. The acid digestion method was adopted from Hseu [15]. The mass of sludge sample used was 1g. The sludge sample was mixed with 10 mL of HNO₃ (65%) and 10 mL of HCl (37%). The mixture of sludge sample and acids were heated at 90°C for 45 minutes at the mixing speed of 100rpm. The mixture was continually heated with increment of temperature until 150°C for 10 minutes. 10 mL of HNO₃ was added periodically three times during heating and boiling to ensure the liquid remains. Then, the mixture was allowed to cool at room temperature. The sample was filtered by using vacuum filter into 100 mL volumetric flasks and filled to the mark with distilled water. The concentration heavy metals mixture in filtered solution were analyzed by using ICP-OES (iCAP 6000 Series).

C. Surfactant-enhanced Oil Recovery (SEOR)

The esterquat surfactant concentration was varied from 50 mg/L to 200 mg/L while the mass of COS was kept constant at 10 g. The raw COS was dissolved in 200 mL of surfactant solution. The mixture of raw COS and esterquat surfactant were mixed at 80 rpm for 20 minutes at ambient temperature. Then, the mixture was allowed to settle down for an hour. The sample was then filtered by using vacuum filter. The treated sludge obtained from filtration was dried in oven at 105°C for 3 hours to remove moisture from the sludge [16].

D. Analysis of oil and heavy metal removal

The absorbance of raw concentrated oily sludge and cationic esterquat surfactant were determined by using Eq. (1) at transmittance value of 1636.51 cm⁻¹ (aromatic C=C stretch).

$$A = 2 - \log_{10} (\%T) \quad (1)$$

where A is absorbance and is T transmittance, respectively.

The percentage of oil removal (%OR) of oily sludge was calculated as follows (Eq. (2)):

$$\%OR = \left(\frac{A_{raw} - A_{treated}}{A_{raw}} \right) \times 100\% \quad (2)$$

where, A_{raw} is absorbance of raw concentrated oily sludge (COS) while $A_{treated}$ is absorbance of treated oily sludge.

The percentage removal of heavy metal (HR) from COS was determined using Eq. (3):

$$\%HR = \frac{COS_{raw\ sludge} - COS_{treated\ sludge}}{COS_{raw\ sludge}} \times 100 \quad (3)$$

where, the $COS_{raw\ sludge}$ is denoted as the concentration of heavy metal in the raw sludge and $COS_{treated\ sludge}$ is denoted as the concentration of heavy metal in treated sludge.

III. RESULTS AND DISCUSSION

A. Characterization of oily sludge and surfactant

i. Characterization of raw COS via Thermogravimetric Analysis (TGA).

The proximate analysis of raw COS is tabulated in Table I. It is observed that the moisture loss from raw COS is 61.76%. According to Mansaray and Ghaly [17], higher moisture content decreases the heating values of the sample due to some energy were needed to vaporize the moisture at the beginning of the conversion process. In addition, higher moisture content yields more residue because of poor heat transfer to the inner surfaces that leads to a low average particles temperature. The percentage of volatile matter in COS observed was 32.22 wt% which indicates significant of oil content to be recovered in the raw COS sample [11].

Table I: Proximate analysis of raw concentrated oily sludge

Analysis	Moisture content	Volatile matter	Ash content	Fixed carbon
Percentage of weight (%)	61.76	32.22	4.86	1.16

Fig. 1 indicates the phases of mass loss for raw COS sample via TGA. In general, the proximate analysis of sludge can be divided into three phases which consists of drying and evaporation of light components (Phase I), devolatilization of PAHs (Phase II) and decomposition of lignin (Phase III). Phase I occurs at temperature below 200°C whereby 61.76 wt% of

water was removed from COS. The temperature range for phase II is between 200°C to 600°C involved the decomposition of 32.22 wt% of volatile matter contained in COS. The presence of high volatile matters in the sample indicates significant amount of polycyclic aromatic hydrocarbons (PAHs) to be removed in enhancing dewatering process and decreasing the cost of sludge disposal. The phase III is attained at temperature above 600°C whereby 4.86 wt% of residue yield (ash content) was observed at this phase.

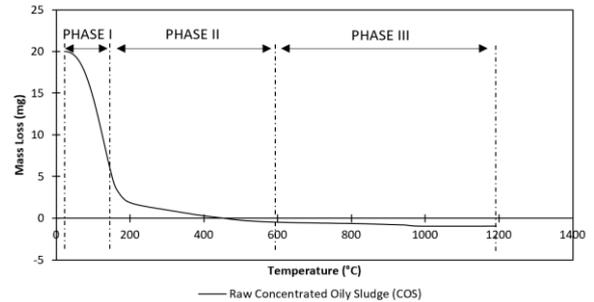


Fig. 1: TGA curve for raw COS sample

ii. Characterization of COS and esterquat surfactant via Fourier Transform Infrared Spectroscopy (FTIR).

Fig. 2 shows the FTIR spectra for raw COS and esterquat surfactant. From Fig. 2, esterquat surfactant shows a strong peak of absorption (alkanes C-H stretch) at 2922.69 cm^{-1} in which slightly similar to the raw COS of 2921.35 cm^{-1} . However, the raw COS shows a peak visible at 3336.28 cm^{-1} and 1636.51 cm^{-1} which indicates the OH and aromatic C=C stretch respectively in which notices as typical main structure exist in oily sludge. This is an indication in the presence of polycyclic aromatic hydrocarbons (PAHs) in raw COS compared to alkanes hydrocarbon in esterquat surfactants that shows the significant content of long hydrophobic chain. Besides that, the presence of C=O stretch was observed only for esterquat surfactant at wavenumber of 1740.26 cm^{-1} .

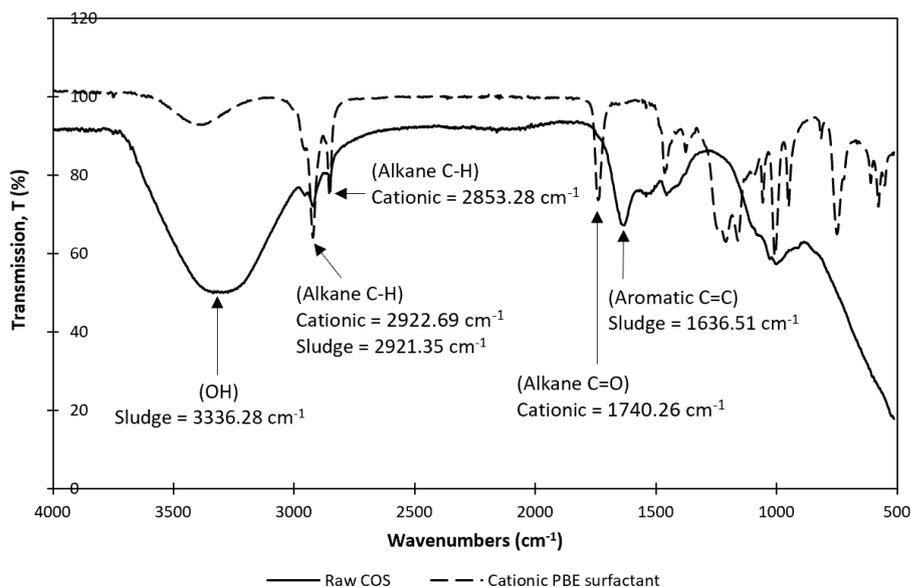


Fig. 2: FTIR spectra of raw COS and esterquat surfactant

iii. Determination the Critical Micelle Concentration (CMC) of esterquat surfactant via simplified colorimetric method.

Fig. 3 illustrates the absorbance of Reactive Blue 4 (RB4) dye at different concentration of esterquat surfactant. The maximum absorption band formed by RB4 dye in aqueous solution is 595 nm. The decrease of absorbance value at 595 nm was observed as esterquat concentration increased from 50 mg/L to 100 mg/L due to the presence of soluble precipitate during mixing of RB4 dye and esterquat surfactant. However, the absorbance of RB4 increased as esterquat surfactant concentration increased from 100 mg/L to 200 mg/L. This was due to the formation of micelles at surfactant concentration exceed its Critical Micelle Concentration (CMC). At surfactant concentration exceeds its CMC, less surfactant will form soluble precipitate with RB4 dye, hence resulted in an increase of absorbance value [18]. The minimum absorbance value which indicates CMC was observed at esterquat concentration of 125 mg/L.

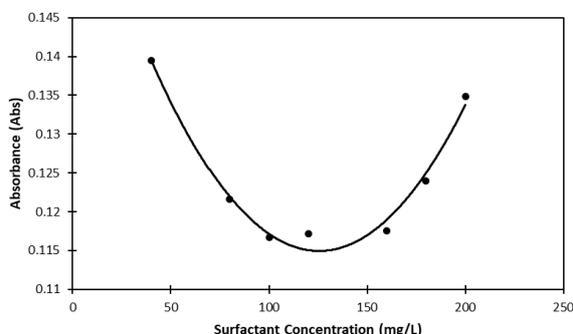


Fig. 3: Absorbance of RB4 dye at 595 nm at different concentration of esterquat surfactant.

vi. Determination the CMC of esterquat surfactant via simplified conductivity method.

The CMC is evaluated from the dependence of concentration aqueous solution using the conductivity

method. Fig. 4 shows the specific conductivity at various surfactant concentration. From Fig. 4, it is observed that the conductivity increased with surfactant concentration. However, at concentration between 100 mg/L to 150 mg/L, the conductivity increased at different manner due to formation of micelles at concentration exceed than surfactant's CMC [19]. Two straight lines were plotted whereby the CMC determined was 125 mg/L at abrupt change of slopes.

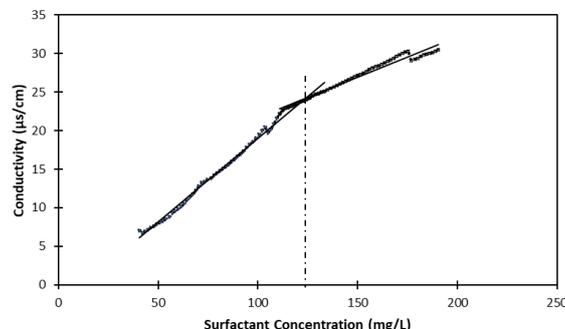


Fig. 4: Conductivity value at different esterquat concentration

B. Surfactant-enhanced Oil Recovery (SEOR)

Fig. 5 presents the analysis of FTIR spectra for raw COS and treated oily sludge at various cationic esterquat surfactant concentration. From the Fig. 5, it is observed that there is no noticeable C=O stretch at the peak of 1740.26 cm⁻¹. This result shows less deposition of cationic esterquat surfactant on the treated oily sludge. Moreover, the loss of peak occurs at 1636.51 cm⁻¹ which indicates aromatic C=C stretch indicates PAHs. The treated oily sludge also shows the loss of peak in OH stretch (3336.28 cm⁻¹) in which shows the cationic esterquat surfactant has been solubilized by most of the amounts of PAHs content in raw COS during mixing process.

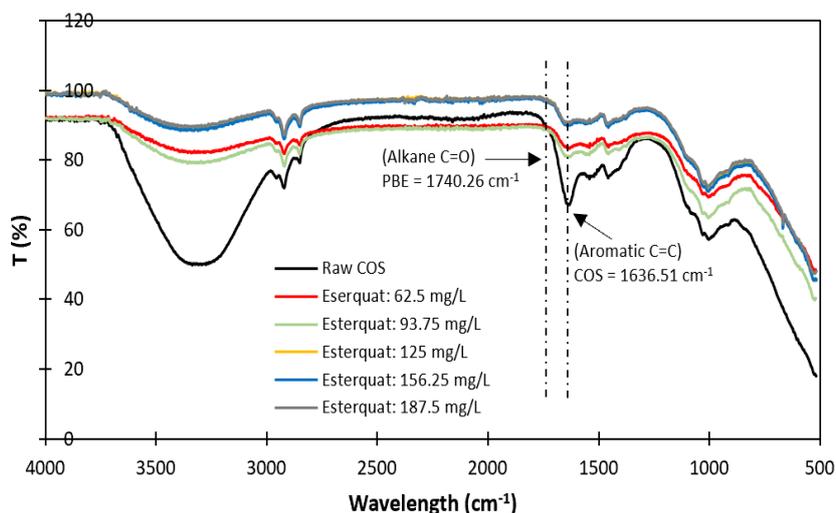


Fig. 5: FTIR spectra of raw COS and treated oily sludge at various cationic esterquat concentration

The absorbance of PAHs content is calculated according to transmission data of FTIR on aromatic C=C stretch at wavelength of 1636.51 cm⁻¹. From this data, the percentage of oil removal (OR) was determined. Fig. 6 shows the percentage of OR from treated oily sludge at different cationic esterquat surfactant concentration. It is observed from this figure that the percentage of OR was 54.20% at cationic esterquat surfactant concentration of 50 mg/L. The OR percentage decreased until 46.76% as the esterquat surfactant concentration increased to 93.75 mg/L. This was due to the less monomer present at low esterquat concentration; hence less amount of oil can be extracted from COS. The inconsistent reaction between monomer of esterquat surfactant and oil trapped in sludge resulted in fluctuate removal of oil from COS.

However, the significant increase of OR is noticeable as esterquat surfactant concentration increased from 125 mg/L (74.62%) to 187.5 mg/L (75.09%). The CMC of esterquat surfactant was determined at 125 mg/L. At CMC, the hydrophobic tail of esterquat surfactant will bind together to produce aggregates and micelles [20]. The oil trapped in sludge would solubilize in micelle's palisade layer or inner hydrophobic core of micelles and bind with hydrophobic tail of esterquat surfactant. The formation of stable micelles would avoid the tendency of hydrophobic tail of surfactant monomer to bind with the sludge. This finding was in good agreement with Zhang, Li, Thring, Hu and Song [21].

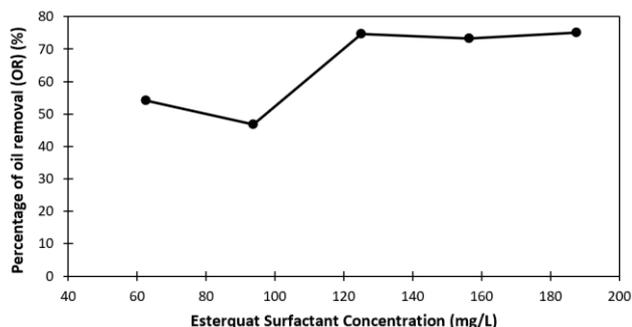


Fig. 6: Percentage of oil removal (OR) at various cationic esterquat concentration

Fig. 7 shows the effect of various esterquat surfactant concentration on heavy metal via SEOR process. It is observed that the removal of most heavy metals were

insignificant with the increase of esterquat surfactant concentration except for Lead (Pb). The Pb removal was slightly increased from 21.94% to 24.45% as the esterquat concentration increased from 62.5% to 187.5%, respectively. The presence of micelles at concentration of surfactant exceeds its CMC would slightly enhance the solubility of Pb ions into aqueous solution, hence decreased the concentration of Pb ions trapped in COS.

Generally, heavy metal ions interact with hydrophilic head of surfactant via electrostatic interaction [22]. However, the esterquat surfactant and heavy metals were consists of similar positive charge. This resulted in less removal of heavy metals via SEOR process. The removal of heavy metals may influence by solubilization of heavy metal into aqueous solution rather than in micelle's head group.

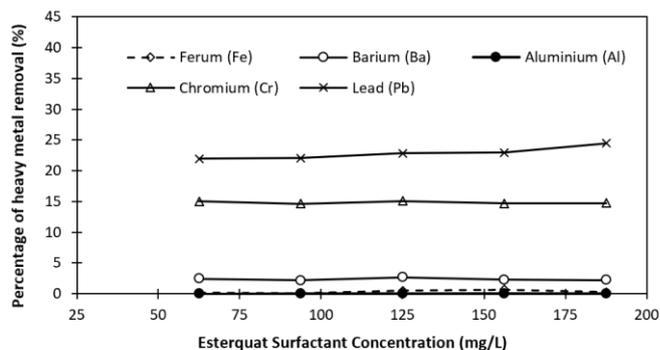


Fig. 7: Percentage of heavy metal removal (HR) at various esterquat concentration

IV. CONCLUSION

The advances in oil technology is much related to enhance oily sludge treatment using the cationic plant-based esterquat surfactant was driven well-intentioned investigation. Based on the result achieved, the treatment of COS using the cationic esterquat surfactant shows defining result in which capable to increase the removal of oil and metal in oily sludge as well as reduce the sludge weight for dumping process. It is proven that the cationic esterquat surfactant proficient in removing oil content from COS for about 46.76% to 75.09% at esterquat concentration ranging from 100 mg/L to 200 mg/L. In addition, the cationic esterquat surfactant was noticeable to attract the heavy metals from COS such as chromium (Cr) and lead (Pb) in which capable to be classified as scheduled waste SW204 and accounting for environmental-friendly of sludge disposal. These findings verify the potential of cationic esterquat surfactant to enhance the oil and metal removal from COS.

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