



Comparative study on sulphur reduction from heavy petroleum - Solvent extraction and microwave irradiation approach

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Abstract

Sulphur- containing compounds in heavy crude oils are undesirable in refining process as they affect the quality of the final product, cause catalyst poisoning and deactivation in catalytic converters as well as causing corrosion problems in oil pipelines, pumps and refining equipment aside environmental pollution from their combustion and high processing cost. Sulphur reduction has being studied using microwave irradiation set at 300W for 10 and 15minutes and oxidative- solvent extraction method using *n*- heptane and methanol by 1:1, 1:2 and 1:3 crude- solvent ratios after being oxidized with hydrogen peroxide, H₂O₂ oxidants. Percentage sulphur removal with *n*- heptane solvent by 1:1 and 1:2 are 81.73 and 85.47%; but extraction using methanol by different observed ratios gave less sulphur reduction. Indeed when microwave irradiated at 300W for 10 and 15minutes, 53.68 and 78.45% reduction were achieved. This indicates that microwave irradiation had caused oxidation by air in the oven cavity and results to formation of alkyl radicals and sulphoxide from sulphur compound in the petroleum. The prevailing sulphur found in the crude going by FT-IR results is sulphides which oxidized to sulphoxide or sulphones. It is clear that sulphur extraction with heptane is more efficient than microwave irradiation but economically due to demands for solvent and its industrial usage microwave irradiation can serve as alternative substitute for sulphur reduction in petroleum. Sulphur reduction by microwave radiation should be up- scaled from laboratory to a pilot plant without involving extraction column in the refining.

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1. Introduction

Heavy petroleum having been dense with high viscosity contains many varieties of complex hydrocarbon molecules along with organic impurities containing sulphur, nitrogen, polycyclic aromatics and asphaltenes, heavy metals and metal salts [1]. However, most of heavy petroleum processed in our refineries is often foreign crude imported from either Middle East or Russian country. It has been known that heavy crude impurities are in concentrated form. Sulphur has been recognized as one of the major impurity which affects the quality of the final refined product and often contributes to high processing cost. Parallel to this, sulphur containing compounds in the crude oil creates many operational issue and

also pollutes air when products burn. Sulphur in petroleum poisons the catalyst and causing catalyst deactivation [1, 2].

According to Erikh *et al.* [3], higher sulphur products leads to increased emissions of sulphur dioxides and resultant acid rain thus giving poor performance of vehicle emissions control systems. Refining processes encountered corrosion problems when processing crude oil with appreciable amount of sulphur and sulphur- containing compounds. Sulphur compounds are highly objectionable in commercial products on account of their unpleasant smell or bad odour [4]. Each crude oil has different amounts and types of sulphur compounds, but as a rule the proportion, stability, and complexity of the compounds are greater in heavy crude oil fractions. The combustion of petroleum products containing sulphur compounds produces undesirables such as sulphuric acid and sulphur dioxide [5]. Conventionally, most refineries adopted hydrotreating processes or hydrodesulphurization (HDS) reaction which involves catalytic conversion of various sulphur compounds to nonsulphur containing materials in the presence of hydrogen. The production of low levels of sulphur- containing compounds product therefore required the application of severe operating conditions, the use of active catalyst [6] and possible innovative technology.

Microwave irradiation is attracting researcher's attention as a tool for facilitating chemical reactions. Indeed, microwave energy, with a frequency of 2.45GHz, is well known to have a significant effect on the rates of a variety of processes. The number of reported applications, especially in the food industry, is increasing rapidly [7, 8]. Microwaves interact strongly with some (*polar*) materials and weakly with others. Energy absorption varies depending upon microwave operation frequency, sample composition, dielectric properties of the materials and temperature of operation. Dielectric properties of a material or combination of materials affect how the transmitted microwaves react with the materials in the system. The dielectric properties of crude oil vary according to the type and composition since crude oil has a low dielectric constant (2 to 3) and thus weakly absorbs microwaves. The dielectric factor and the loss factor are not constant because they vary with temperature, frequency (power), and moisture content [9]. Several literatures reported that microwaves heating has effectively shortened the reaction time and dramatically probe optimum production conversion to about 84- 86.3% [2, 6, 10].

Microwave heating has an incontestable place in analytical and organic laboratory practices as a very effective and non-polluting method of activation and heating. Two major effects are responsible for the heating, both resulting from interaction of electric field component of the microwaves with the sample [11, 12]. Dipole interactions occur with polar molecules. The polar ends of a molecule tend to align themselves and oscillate in step with the oscillating electrical field of the microwaves. Collisions and friction between the moving molecules result in heating. However, the more polar a molecule, the more effectively it will couple with (and be influenced by) the microwave field. Ionic conduction is only minimally different from dipole interactions. Ions can couple with the oscillating electrical field of the microwaves [13, 14]. The effectiveness or rate of microwave heating of an ionic solution is a function of the concentration of ions in solution [15]. The application of microwave heating to desulphurization reactions has precedent in the patent literature [6].

2. Sulphur- containing compound

Sulphur compounds are highly polar molecules with sulphur at the negative end, and hydrogen and carbon at the positive end. Zuloaga *et al.*, [16] reported that the more polar a solvent, the quicker its temperature shoots up. According to Erikh *et al.* [3] about 70 to 90% of sulphur containing compounds is concentrated in the fuel oil. Mercaptans and disulphides are chiefly contained in the gasoline and kerosene fraction whereas cyclic sulphides and polycyclic sulphur compounds are contained in the heavier molecular weight hydrocarbons. These get into the kerosene and oil fractions in the distillation of petroleum. The sulphides and mercaptans from C₂ to C₇ have low boiling points (37-150°C) and easily oxidized or decomposed on heating above the specified temperature range to form sulphides or sulphoxides and with the evolution of H₂S [3].

In addition to this, even air or weak oxidizing agents oxidized sulphides and mercaptans to disulphides. Many researchers observed that the most probable types of sulphur- containing compounds like polycyclic sulphur compounds are more complex to isolate completely but more often than not can be reduced to an acceptable minimum. The aim of the study is to find out the possibility of using microwave irradiation to reduce sulphur compounds in imported foreign crude petroleum; hence sulphur- containing compounds can thermally decomposed (or oxidized) on exposure to a low temperature conditions.

3. Methodology

3.1 Equipment and materials

- Sulphur- in Oil Analyser (model number XLSA-20), Thermo Nicolet IR Spectrometer (100 FT-IR) System, Magnetic heater and Stirrer with temperature regulator, Thermometer and Sample bottles, Pyrex 25ml Measuring Cylinder and 250ml separating funnel and Domestic Microwave oven (Model: HH7206R).
- Urals heavy crude oil (Russian), Methanol (BDH- Chemical, Poole, England), *n*- Heptane (Kemie Labs, India), Ethanoic acid (BDH- Chemical, Poole, England) and Aluminium oxide, Al₂O₃ and Hydrogen Peroxide (May & Baker, Dagenham, England).

3.2 Procedure for oxidation of crude oil

50ml of crude oil sample was poured into a 250ml conical flask with 5ml of organic acid (ethanoic acid) and a magnetic bar was placed inside for proper stirring. The mixture was heated at 90°C, the 5ml solution of H₂O₂ aqueous was added in drop wise and with constant stirring over 15minutes to oxidize sulphur compounds in the sample.

3.3 Procedure for sulphur compounds extraction

The oxidized crude oil sample was mixed in a 250ml separating funnel with each solvent (methanol and *n*- heptane) at room temperature and atmospheric pressure to obtained biphasic mixture in the ratio 1:1 (20ml of crude oil sample to 20ml of solvent), 1:2 (20ml of crude oil sample to 40ml of solvent) and 1:3 (20ml of crude oil sample to 60ml of solvent). Thereafter, the mixtures were left to achieve extraction equilibrium for 30minutes to form two- phase separation. The crude oil phase was decanted carefully from the solvent- sulphur phase and finally analyzed for total sulphur content.

3.4 Procedure for microwave desulphurization

50ml of crude oil sample was poured into a 250ml pyrex glass with aluminium oxide, Al₂O₃ (microwave absorber) and placed in the domestic microwave oven (Model: HH7206R). The microwave power was adjusted to 300W. The timer was turned on first to its maximum (60minutes) then back to the desired microwave irradiation time 10minutes. The operation began as soon as the oven door was firmly closed to activate the sample energy. The procedure was repeated by setting microwave irradiation time to 15minutes. After the set times elapsed, microwave irradiated crude oil samples were analyzed respectively.

3.5 Procedure for sulphur- in- oil analyzer

The sample was poured into a disposable sample container to three quarter of its capacity. This is to ensure the X- ray passes through the test sample in order to give accurate sulphur counts. The sample was then covered with X- ray transparent plastic film window. The power was switched on and light up the X- ray lamp within seconds. The analyzer gives three different readings at 30seconds intervals. The readings were recorded and average sulphur content was determined in percentage sulphur by weight.

3.6 Procedures for thermo nicolet IR spectrometer

A drop of desulphurized crude oil sample obtained was placed between two plates of potassium bromide, KBr salt (transparent to infrared light) and squeeze to remove any trapped air in order to form a thin film (sand witched) between the plates otherwise light cannot pass through. The plates were placed in the sample holder and positioned in the standard sample compartment of the spectrometer and then FT-IR spectra were obtained under 2minutes data collection period with a spectral range from 4000.00 to 406.75cm⁻¹ at a resolution of 4cm⁻² [2, 17].

4. Result and discussions

Table 1 shows the average sulphur content in the crude oil before and after extraction using *n*- heptane solvent by 1:1, 1:2 and 1:3 ratios as well as under microwave irradiation. Sulphur is the other parameter focused for determining the price of crude oil and refining processing cost. The total sulfur content is expressed as a percentage of sulfur by weight, and it varies from less than 0.1% to greater than 5% depending on the type and source of hydrocarbons respectively [18]. The total sulphur content in the untreated crude oil was 1.494wt%. After oxidation with oxidants, H₂O₂, it reduces by 25.23% to

1.117wt%. This is evidence that sulphur- containing compounds in the crude oil oxidized to sulphoxide or corresponding sulphone.

However, sulphur extraction with heptane by 1:2 gave high percentage reduction (85.47%) to 0.217wt%; this was closely followed by 1:1 ratio where 81.73% reduction to 0.273wt% was achieved. As observed, approximately the same 79.0% (to the nearest whole number) sulphur reduction were achieved by being irradiated at 300W for 15minutes and by using *n*- heptane to crude oil 1:3 ratio. This inferred that microwave has the potential to reduce sulphur in the crude oil. Most of sulphur- containing compounds found in the crude oil such as thiol (mercaptans), thiophenes, benzothiophenes, dibenzothiophenes, sulphoxide or sulphone among others are polar compounds. Sudipa *et al.* [19] observed that, much greater polarity of the sulphoxide groups moves from oxidized component to the asphaltene.

Indeed, according to Zuloaga *et al.* [16], microwave can turned to heat if they interact with a polar material and causes it to rotate and the quicker its temperature shoot up. There is every possibility some low- boiling sulphur compounds in the crude when heated can oxidized by air in the oven cavity to form sulphoxide and corresponding sulphur oxide, SO₂ as well as hydrogen sulphide, H₂S due to radicals formation and recombination [20]. It is clear that heptane extraction by 1:1 and 1:2 ratios are efficient in sulphur extraction than microwave irradiation treatment.

Table 1. Average sulphur content using *n*- heptane solvent and microwave irradiation

Sample Identity	Sulphur Content (wt%)	Percentage Reduction (%)
Untreated Crude Oil	1.494	-
Oxidized Crude Oil	1.117	25.23
EOSC- 1:1 (Heptane)	0.273	81.73
EOSC- 1:2 (Heptane)	0.217	85.47
EOSC- 1:3 (Heptane)	0.318	78.79
Mw (300W-10mins)	0.692	53.68
Mw (300W-15mins)	0.322	78.45

Table 2 presents the average sulphur compounds in the untreated, oxidized and sulphur extracted crude oil with methanol solvent. On comparison, about 24.03, 31.12 and 30.72% sulphur was removed by using 1:1, 1:2 and 1:3 ratios; whereas microwave treated at 300W for 10 and 15minutes gave 53.68 and 78.45% sulphur reduction. These prove that methanol extraction is less effective in sulphur removal than *n*- heptane as well as being microwave irradiated.

Table 2. Average sulphur content using methanol solvent and microwave irradiation

Sample Identity	Sulphur Content (wt%)	Percentage Reduction (%)
Untreated Crude Oil	1.494	-
Oxidized Crude Oil	1.117	25.23
EOSC- 1:1 (Methanol)	1.135	24.03
EOSC- 1:2 (Methanol)	1.029	31.12
EOSC- 1:3 (Methanol)	1.035	30.72
Mw (300W-10mins)	0.692	53.68
Mw (300W-15mins)	0.322	78.45

Figure 1 presents a spectrum of untreated oil. A very broad IR absorption at 3445cm⁻¹ centered between 3328-3448cm⁻¹ was not used; hence it corresponds to -OH stretching vibration which may be attributable to intermolecular hydrogen bonding from water molecules, H₂O in the crude oil. Most of all spectra are similar but band 3445cm⁻¹ those not appeared in spectrum of oxidized and irradiated crude oils.

Antoniali *et al.* [21] observed that aliphatic saturated C-H stretching bands occur within 2850 - 3000cm⁻¹ range with intensities proportional to the number of C-H bonds. However, a strong infrared bands at 2924 and 2860cm⁻¹ are responsible for methylene C-H and methyl -CH₃ asymmetric stretching vibration from saturated aliphatic compounds. Peak located at 1715cm⁻¹ in the spectrum of untreated crude oil is carbonyl group, C=O due to connection to an aromatic ring [22]. IR absorption bands at 1457 and 1603cm⁻¹ are typical of methyl, C-CH₃ asymmetric bend stretching from aromatic compound and carbon -carbon, C-C stretching showing skeletal vibrations on the ring.

John [22] observed in a practical approach that molecules containing nitro groups, -NO_2 exhibits vibration at $1260\text{-}1390\text{cm}^{-1}$. It worthy to note that peak at 1375cm^{-1} and bands at 1302cm^{-1} are due to aliphatic nitro compounds, -NO_2 or nitrate ion. Absorption at 811 and 732cm^{-1} are due to 1,4 disubstitution (*para*) on aromatic determined from C-H vibrations and methylene, $(\text{CH}_2)_n$ rocking from aliphatic saturated compound. These observation, indicate that sample crude oil composed largely paraffinic and aromatic.

It is worthwhile to mention that sulphur and its compound structural information and identification is our priority in this study. Weak absorptions at 1032cm^{-1} is assigned to anhydride responsible for overlapping vibration of C-O-C stretching of ether and S=O of sulphoxide. The weak band at 617cm^{-1} in the untreated crude spectrum is assigned to disulphide, S-S stretching vibration due to catenation (unique chemical characteristic) where the formation of S-S bonds in extended C-C chain is common. Indeed, Further study by Al-Zahrani [18] revealed that oxidation process using oxidants, H_2O_2 convert sulphur compounds and there methyl and high alkyl derivative into sulphone which are removed by solvent extraction as sulphoxide. The peak located at 1017cm^{-1} is due to anhydride (ethers), C-O-C. However, focusing on sulphur compound identified, the strength of the absorption (or transmittance) is proportional to the concentration. These bands indicate the presence of sulphur and its group in the crude oil.

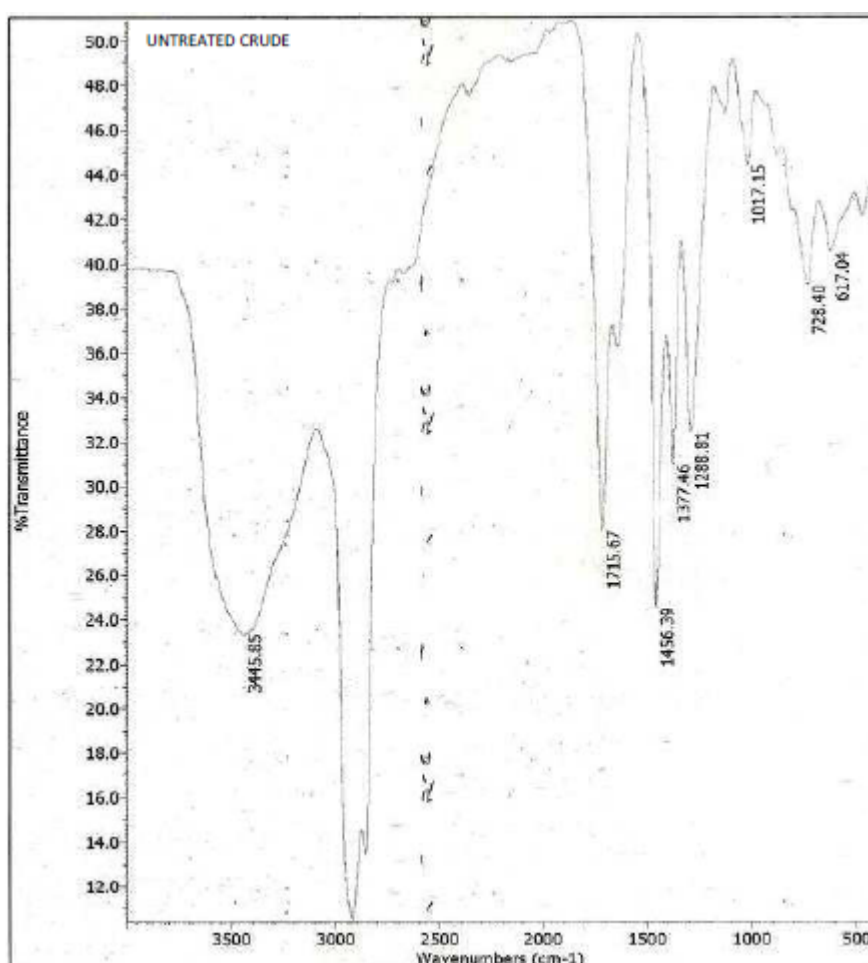


Figure 1. FT-IR spectra of untreated heavy crude oil

Figure 2 presents the spectra of oxidized crude oils using hydrogen peroxide, H_2O_2 . A spectrum pattern of oxidized oil is similar to that observed in Figure 1. Absorption bands at 2924 , 1457 and 1375cm^{-1} assigned to methylene, C-H, methyl, C- CH_3 bend and aliphatic nitro compounds, NO_2 vibrations appeared in the spectra of untreated and oxidized crude oil. Whereas, there are bands at 2860 , 1603 and 733cm^{-1} which does not appeared in some spectrum. These peaks are due to methyl, CH_3 , carbon- carbon, C-C stretching exhibiting skeletal vibration on the ring. The weak band located at 617cm^{-1} assigned to disulphide, S-S stretching vibration does not appear completely in treated samples spectra. This indicates

effectiveness of oxidation process with low sulphoxide formation based on transmittance, 69.74%T at 1029cm^{-1} assigned to sulphoxide.

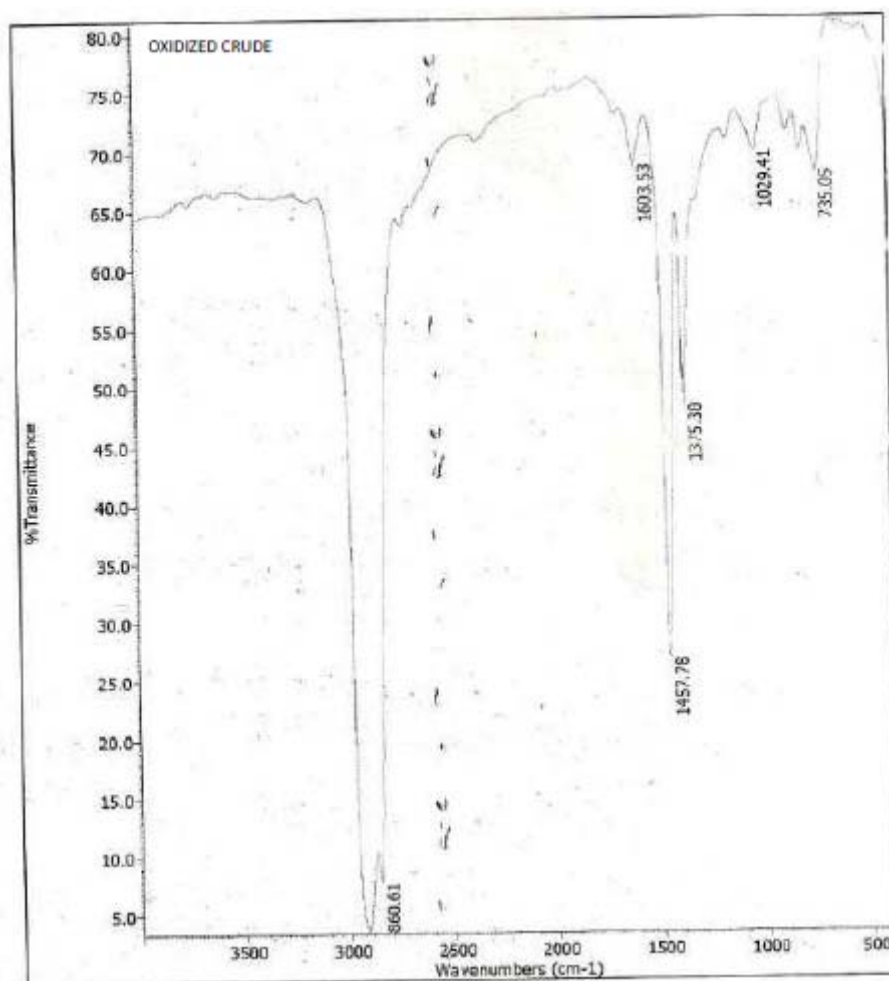
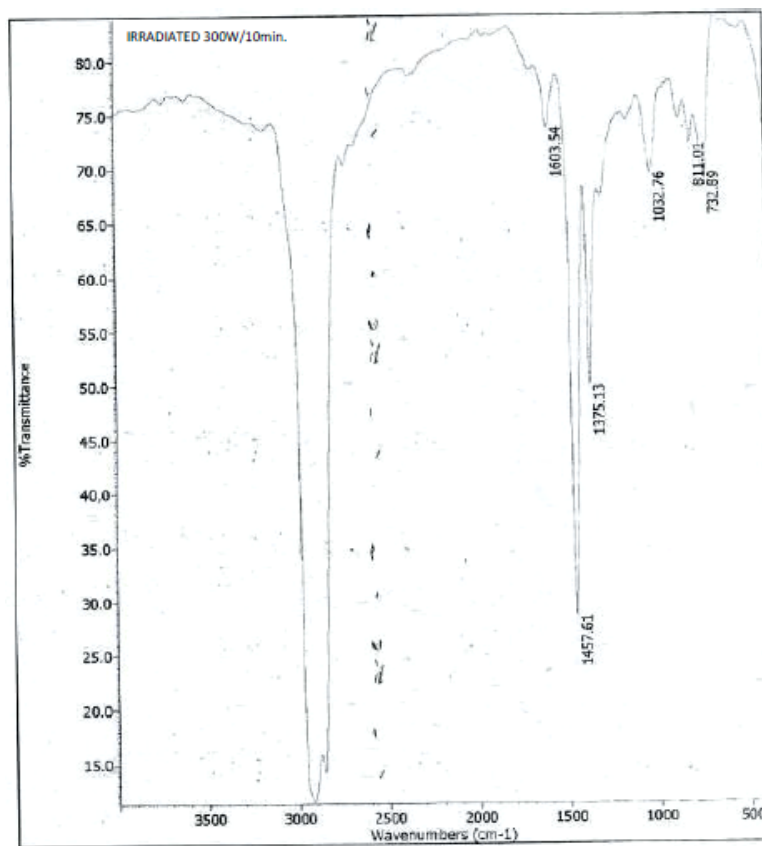


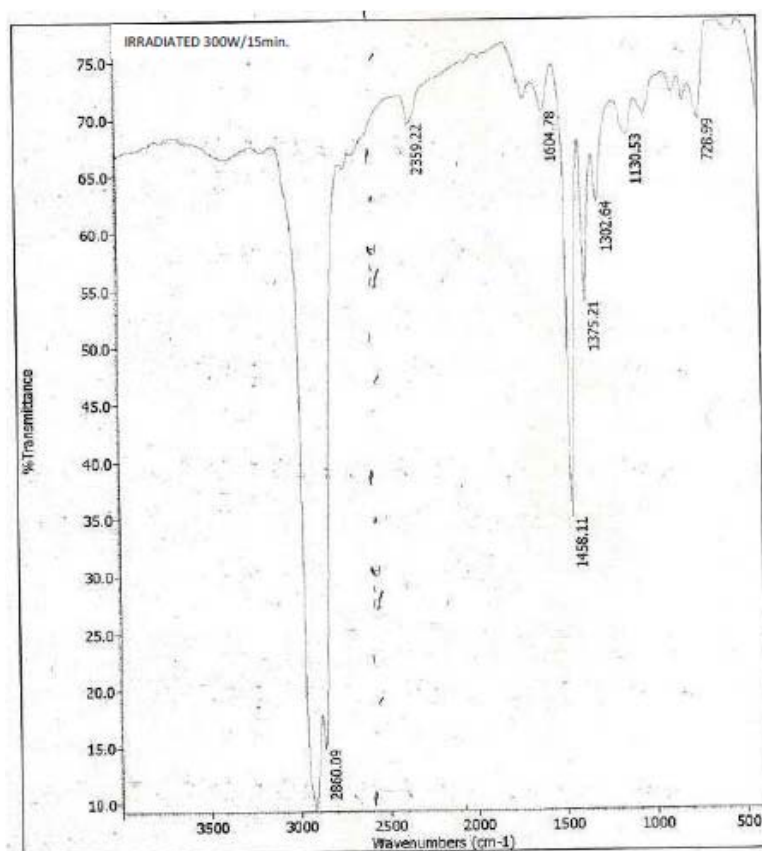
Figure 2. FT-IR spectrum of oxidized heavy crude oil

Figure 3(a) and (b) depict the spectra of irradiated at 300W for 10 and 15minutes. It is obvious the spectra are similar and showed the same pattern. Absorption bands at 2924 , 2860 and 2359cm^{-1} were assigned to methylene, C-H, methyl, C-CH₃ bend and carbon dioxide; CO₂ compounds vibrations by [22]. Peaks located at 2860 and 2359cm^{-1} do not appeared in the spectra of untreated and irradiated crude for 10minutes. Whereas, bands at 1604 , 1458 and 1375cm^{-1} could be assigned to carbon- carbon, C-C stretching exhibiting skeletal vibration on the ring, methyl, C-CH₃, and nitro, NO₂ compounds in the asphaltenes fraction of the oil.

In view of the weak- to- moderate bands associated with sulphur bonds as seen at absorption band located 1033cm^{-1} do not appeared in the untreated crude oil and irradiated crude for 15minutes spectra but it is obvious in the oxidized and solvent treated crude by using 1:1, 1:2 and 1:3 ratio and irradiated crude for 10minutes spectra respectively. These are indicative of sulphide, S-S functional group oxidation in the presence of H₂O₂ to sulphoxide, S=O prior to solvent extraction. Frequency band at $\sim 735\text{cm}^{-1}$ is due to methylene, (CH₂)_n aliphatic long chain. In comparison, peaks located at 617cm^{-1} assigned to sulphides (S-S) seen not to appear in the solvent treated and microwave irradiated crude oil has it is been oxidized.



(a)



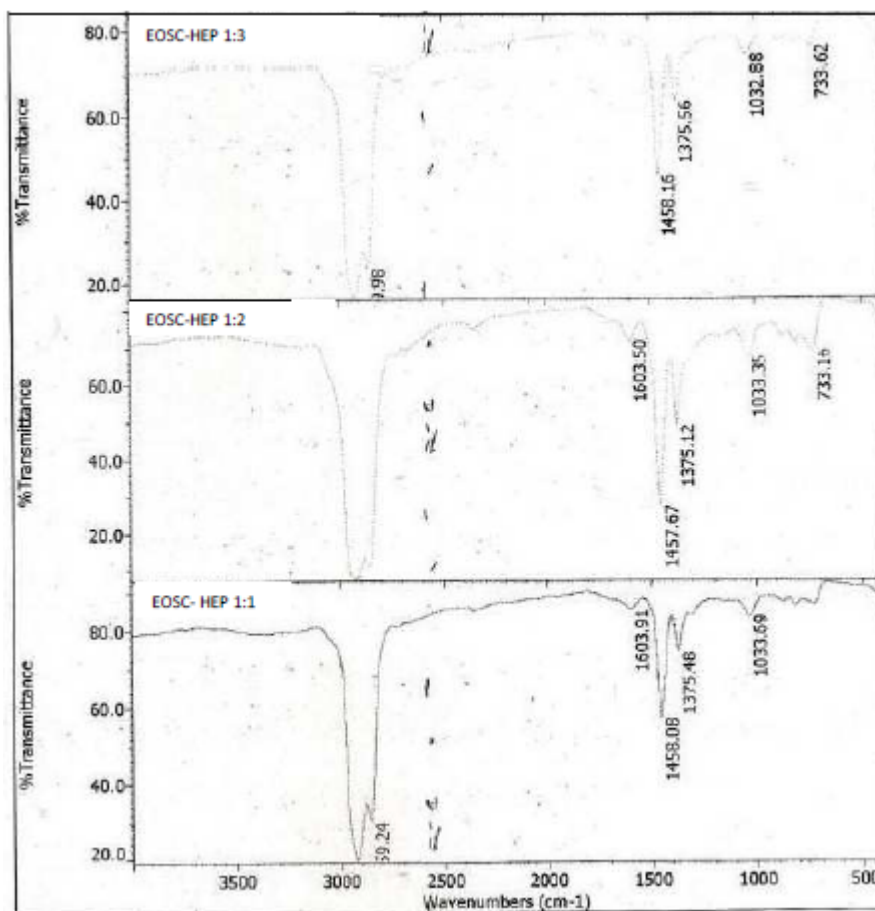
(b)

Figure 3. (a) FT-IR spectrum of irradiated heavy oil at 300W-10mins; (b) FT-IR spectrum of irradiated heavy oil at 300W-15mins

In Figure 4(a) the weak- to- moderate absorption band associated with sulphoxide bonds located at 1029 and 1033 cm^{-1} does not appear in the untreated crude oil spectrum but it is obvious in the oxidized and *n*- hexane solvent treated crude by using 1:1, 1:2 and 1:3 ratio. These are indicative of sulphide, S-S functional group oxidation in the presence of H_2O_2 to sulphoxide, S=O and anhydride, C-O-C had occurred prior to solvent extraction. Interestingly, peaks located at 617 cm^{-1} seen not to appear in the solvent treated crude oil due to its oxidation. Another important observation, the intensity of peak at 1029 cm^{-1} assigned to sulphoxide using *n*- heptane extraction by using 1:1, 1:2 and 1:3 ratio are 83.52, 67.11 and 73.71%T respectively. It is obvious that extraction using 1:1 and 1:3 ratios gave high percentage sulphur reduction seen as sulphoxide compared to sulphur concentration in the oxidized crude oil. These results show that the solvent *n*- heptane have a high capacity to reduce the sulphur- containing compounds in the crude oil to a lesser content. It is quite clear that FT-IR analytical tool cannot determine the total sulphur content in weight percent of the oil rather their percentage concentration.

Figure 4(b) similarly presents spectra of desulphurized crude oils using methanol solvent extraction by 1:1, 1:2 and 1:3 ratio. Absorption bands located at 2924, 1603, 1456, 1375, 1033 and 735 cm^{-1} due to methylene, C-H, carbon- carbon, C-C, backbone skeletal stretch, methyl, C- CH_3 , nitro, NO_2 , anhydride, C-O-C and sulphoxide, S=O and methylene, $(\text{CH}_2)_n$ aliphatic long chain were observed. In contrast, bands at 1304 and 1131 cm^{-1} due to aliphatic nitro compound, NO_2 in the asphaltene fraction of the oil and secondary amine, C-N stretching.

However, comparing the spectra outputs in Figure 4(a) and (b) with untreated crude spectrum (Figure 1), it is observed that *n*- heptane and methanol solvents reduced the total sulphur respectively. Going by the intensity of absorption, methanol solvent on the average using extraction by 1:1 and 1:3 ratios proves less sulphur content reduction than using *n*- heptane. It is worthwhile clear that *n*- heptane solvent and microwave irradiation are suitable for the extraction of sulphur in the crude oil than methanol solvents.



(a)

Figure 4. (Continued)

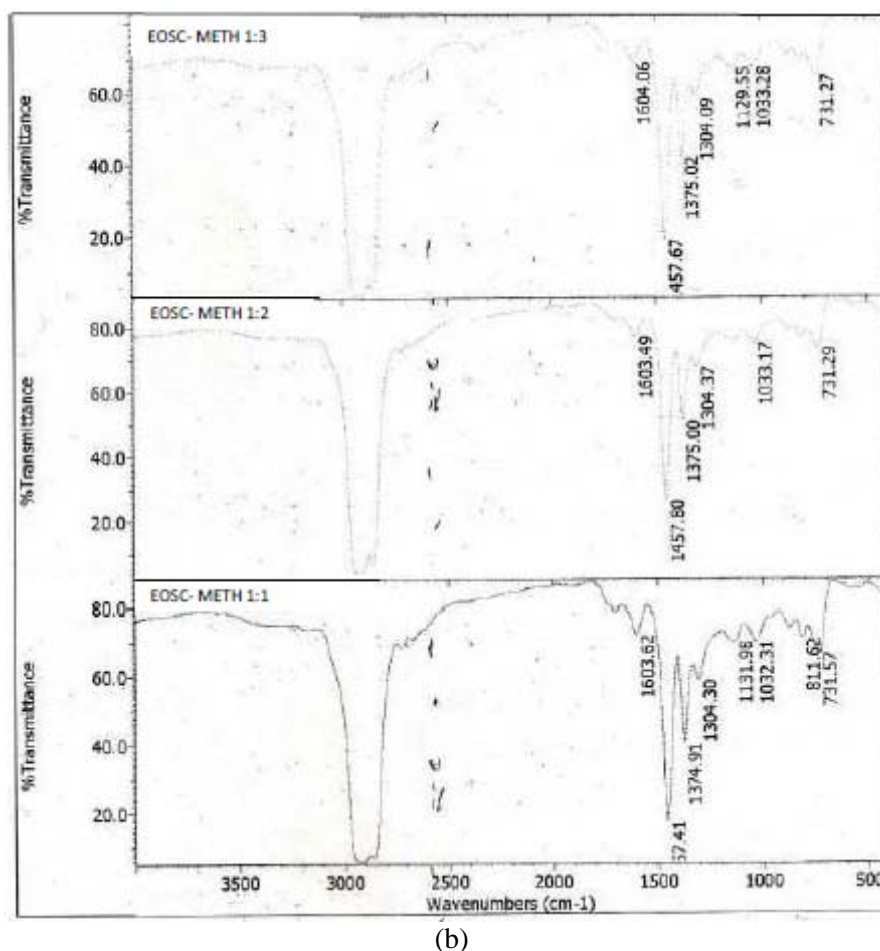


Figure 4. (a) FT-IR spectra of desulphurized crude oil with *n*- heptane at different ratio; (b) FT-IR spectra of desulphurized crude oil with methanol at different ratio

5. Conclusion

Sulphur reduction has been studied using microwave irradiation at 300W for 10 and 15 minutes, *n*-heptane and methanol solvents extraction by 1:1, 1:2 and 1:3 ratios respectively. The sulphoxide is the oxidized form of sulphur compound found in the crude oil as sulphides. Sulphur removal with *n*-heptane solvent by 1:1 and 1:2 are 81.73 and 85.47%; whereas when irradiated at for 10 and 15 minutes, 53.68 and 78.45% sulphur reduction was achieved respectively. Comparing extraction using methanol by different ratios gave less sulphur reduction (24.03- 31.12)% range to what was achieved under microwave treatments. Due to fragmentation of high molecular weight hydrocarbons caused by microwave irradiation in addition to air oxidation results to formation of alkyl radicals and sulphoxide from sulphides. In view of microwave results, 300W for 15 minutes gave a remarkable sulphur reduction; this indicates microwave irradiation power rate is another factor that influence sulphur removal. The prevailing sulphur found in the crude going by FT-IR results is sulphides which oxidized to sulphoxide or sulpones. It is clear that *n*-heptane is more efficient than microwave radiation for sulphur removal but economically microwave treatment will be encouraged. However, on economic consideration, hence demands for *n*-heptane solvent is increasing as it also required, microwave irradiation can serve as alternative substitute in the extraction of sulphur from petroleum.

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