

## Experimental Analysis of the Effect of Microwaves on the Oil Properties in the Presence of Different Minerals

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### Highlights

- Microwave is more effective in the presence of carbonate rock powder;
- Microwave reduces the sulfur present in asphaltene compounds;
- Depending on the irradiation time, microwaves can reduce or increase oil viscosity;
- The microwave heats the oil compounds selectively;

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### Abstract

This research aims to investigate the effect of microwaves on the physical and chemical properties of heavy crude oil in the presence of different minerals. In this regard, the physical and chemical changes of the oil and rock powder (sand and carbonate) mixture are investigated by microwave radiation. Viscosity and temperature changes of two samples are measured. IP143 and elemental analysis (carbon, hydrogen, nitrogen, and sulfur) are used to extract and identify the composition changes of asphaltene. The viscosity and temperature changes show that for both samples at the beginning of microwave radiation, there is a decrease in viscosity due to heavy hydrocarbon particle cracking, such as asphaltene, and converting them into lighter ones. Light compounds evaporate by continuing the radiation and temperature increase; finally, the viscosity increases. The evaporation process in the carbonate powder sample starts earlier than in the sand powder. From elemental analysis, it is concluded that the sulfur and nitrogen in asphaltene decrease almost the same for both samples, and this decrease is more evident for sulfur; thus, the rock powder combined with oil does not have a significant effect on the reduction of these elements. The increase in IFT is also observed due to the evaporation of light oil compounds, and IFT increases further due to the higher temperature of the sample containing carbonate rock powder.

**Keywords:** Asphaltene, Carbonate rock, Heavy oil, Microwave, Sand rock

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## 1. Introduction

Today, fossil fuels are still considered the primary sources of energy supplements. The increasing demand for energy and the lack of available resources have led to the demand for the exploration and exploitation of heavy and extra-heavy oil reservoirs. The modern and optimized methods to produce heavy oil from reservoirs are called enhanced oil recovery (EOR) (Sikiru Rostami et al., 2021; Vakhin Khelkhal et al., 2021; Li Gao et al., 2022; Vakhin Khelkhal et al., 2022). Generally, EOR uses chemical, thermal, miscible gases, or physical methods (Jelmert Chang et al., 2010; Al-Muntaser Varfolomeev et al., 2021; Tirado Yuan et al., 2022). Chemical EOR methods are generally based on injecting chemicals into the oil well to increase the oil mobility. Thermal EOR methods, such as cyclic steam or in situ combustion, are used to increase the reservoir temperature, inject hot water steam and burn some of the oil in the reservoir, respectively (Khelkhal Eskin et al., 2019; Mukhamatdinov Salih et al., 2020). Moreover, physical EOR methods use electrical methods such as electromagnetic (EM) heating to crack heavy oil particles and reduce the oil viscosity (Rehman and Meribout 2012). These new methods and techniques play an essential role in increasing the exploitation of oil reservoirs and can provide a large part of the needed energy. In reservoirs with low permeability and heat transfer, using EM is more effective for reducing viscosity and increasing oil mobility than other methods (Vakhin Khelkhal et al., 2021). EM waves with a frequency of 300 MHz to 300 GHz are called microwaves. These waves create a field with their propagation, and materials with a high dielectric coefficient absorb energy by being in the direction of this field. In the oil industry, microwaves are primarily used for oil shale reservoirs (Butts Lewis et al., 1983; Chanaa Lallemand et al., 1994). The microwave heating is performed using an antenna near the pay zone. For using microwaves as an EOR method, an antenna is placed in a drilled hole close to the producing well. The microwave energy heats the oil in the reservoir, moving toward the producing well (Chhetri and Islam, 2008; Karami Dehaghani et al., 2020; Yazdani and Saeedi Dehaghani, 2022). Hence, microwaves are used for oil upgrading, viscosity change, increasing temperature, and improving wettability and EOR (Li Hou et al., 2014; Mozafari and Nasri, 2017; Taheri-Shakib Shekarifard et al., 2017; Hanyong Kexin et al., 2018; Taheri-Shakib Shekarifard et al., 2018). Energy absorption varies depending on sample composition and dielectric properties (Taheri-Shakib Shekarifard et al., 2018). Therefore, microwave radiation causes certain parts inside the oil to start heating up. Elements in crude oil with a high dielectric coefficient, such as sulfur, nitrogen, and oxygen, can better absorb microwaves. The dielectric properties of materials lead them to be aligned with the MW field pole in the same way that polarity does (Taheri-Shakib Shekarifard et al., 2017; Taheri-Shakib Shekarifard et al., 2017; Taheri-Shakib Shekarifard et al., 2018; Taheri-Shakib Shekarifard et al., 2018; Zhang Adam et al., 2018). Sulfoxides, amines, ketones, and aldehydes are the most common functional groups in asphaltene structures (Sheu, 2002; Joonaki Hassanpouryouzband et al., 2020; Karami and Dehaghani, 2022). Thus, microwaves increase the temperature of oil parts containing asphaltene particles more than other parts. Asphaltene is generally one of the heaviest components in crude oil and causes many problems in production and transportation in the oil industry, and it is essential to find out its behavior. Asphaltene precipitation has high polarity properties, including sulfur, nitrogen, and oxygen elements. Hence, by affecting these particles, microwaves cause them to crack and turn into lighter hydrocarbons (Leontaritis and Mansoori, 1988; Yazdani and Saeedi Dehaghani, 2022). Conditions such as mixing fluids with different compositions and temperatures, gas injection, electrical charges, and pressure and temperature changes due to chemical material cause the

formation of asphaltene deposits (Taheri-Shakib Shekarifard et al., 2018). In recent years, studies have been conducted on heating using microwaves and its effect on asphaltene.

Taheri-Shakib et al. (2017,2018) investigated the effect of microwave duration on heavy oil, compared this method with conventional heating, and concluded that, unlike conventional heating, radiation duration played a key role, and unpredictable changes occurred in different time intervals (Taheri-Shakib Shekarifard et al., 2017). They also investigated the effect of microwaves on crude oil upgrading and observed that compounds containing sulfur, nitrogen, and oxygen elements had better microwave absorption properties due to their high dielectric coefficient. Their research concluded that asphaltene particles were cracked by the microwave and turned into lighter hydrocarbons (Taheri-Shakib Shekarifard et al., 2017; Taheri-Shakib Shekarifard et al., 2017; Taheri-Shakib Shekarifard et al., 2018; Taheri-Shakib Shekarifard et al., 2018; Taheri-Shakib and Kantzas, 2021). By examining the effect of microwave on rock wetting, Karimi et al. (2020-2021) concluded that the higher intensity of microwave radiation on the samples, the more significant change in rock wettability. Microwave radiation changes the rock surface's wettability to water-wet (Karimi Dehaghani et al., 2021). By examining the FTIR spectra results, they also concluded that microwave heating had more effect on heavier oil components (Karimi Dehaghani et al., 2020). Bjorndalen et al. (2004) reported that microwaves increased the temperature of oil, asphaltene, and paraffin oil, but the effect of waves on asphaltene was more significant due to the high polarity of these particles.

This research aims to compare the effect of microwaves on oil in the presence of minerals such as carbonate and sands rock powder. Therefore, in the presence of these minerals, changes in oil temperature and viscosity and changes in asphaltene precipitation are examined and compared. Further, the wettability of the two rock samples used in this research is investigated and compared. The results obtained in this paper can help compare the advantages of using the microwave heating method on carbonate and sand reservoirs and compare them.

## **2. Methods and materials**

This work used oil from a reservoir in southwest Iran and two samples of sand and carbonate powder. The size of powder particles of the two rock samples was made uniformly by 80–130 mesh. Fifteen grams of rock powder was mixed with 50 mL of oil and placed in a 70 °C oven for 5 days. Then, they were exposed to microwave radiation for 30 min at 5 min intervals. The changes in oil viscosity and temperature of both samples were measured every 5 min of microwave radiation. The IP143 test was used to extract asphaltene from the oil sample before and after microwave radiation, and the changes of two asphaltene samples were then examined using elemental analysis (CHNS).

### **2.1. Heavy oil properties**

Heavy crude oil from a reservoir in the southwest of Iran with a viscosity of 871 mPa·s (API = 30.95) at 25 °C was used in the experiments. According to the IP143 test results, the total amount of crude oil asphaltene was 16% before microwave.

### **2.2. Microwave oven and thermometer**

The microwave oven used in the experiments had a maximum power of 1300 watts, and the device dimensions were 20 cm × 17 cm × 10 cm. A digital thermometer was used to record the temperature changes in the oil up to 350 °C.

### **2.3. Elemental analysis**

Elemental analysis (CHNS) was used to determine the molar concentration of carbon, hydrogen, nitrogen, and sulfur atoms in asphaltene using Vario Max-CHNS elemental analyzer. It is possible to find the changes in sulfur and nitrogen atoms of samples under microwave radiation using this analysis.

### **2.4. IP 143**

IP 143 was used to extract asphaltene from crude oil. For this purpose, oil was added to normal heptane with a volumetric ratio of 1:20, and the solution was heated for 20 min. After cooling, it was kept in the dark for 12 h until the oil was dissolved entirely in normal heptane. Asphaltene is insoluble in normal heptane, so it remains as solid particles in the solution (Dehaghani and Badizad, 2016). Then, the solution was passed through the Whatman-42 filter paper; the solid particles contained large amounts of asphaltene, resin fraction, and wax crystals. The filter paper was placed in a Soxhlet and washed with normal heptane in the next step. After that, the filter paper was rewashed with toluene to dissolve the asphaltene. Solid particles, such as remaining rock powders, were trapped in Whatman paper. Then, toluene was placed under the hood to dry, and the black deposit in the container was asphaltene and was used for CHNS analysis (Taherian Dehaghani et al., 2021).

### **2.5. Viscosity**

An Anton par rheometer measured the oil viscosity in each microwave radiation interval. CC27 spindle was used to calculate the desired viscosities with a maximum rotation speed of 1200 rpm and a shear rate of 1400 s<sup>-1</sup>. Moreover, the device was set in a temperature range of 0–80 °C. When the fluid was placed inside the rheometer, the spindle in the device started to rotate by its motor, and the machine calculated the resistance force caused by the viscosity of the fluid and displayed it on a monitor.

### **2.6. Interfacial tension**

Both tested oil samples were analyzed before and after radiation to check the changes of interfacial tension (IFT) by microwave. IFT analysis by the pendant drop method measured the interfacial tension between oil and water. To perform this analysis and obtain the interfacial tension, we first connected a syringe filled with oil to the bottom of the glass chamber of the device. The chamber was filled with distilled water, and by pressing the syringe, a drop of oil was injected into the chamber; the drop was photographed using a high-resolution camera. The density of two samples before and after microwave radiation was calculated by a pycnometer to calculate IFT. Then, the interfacial tension was measured using MATLAB software.

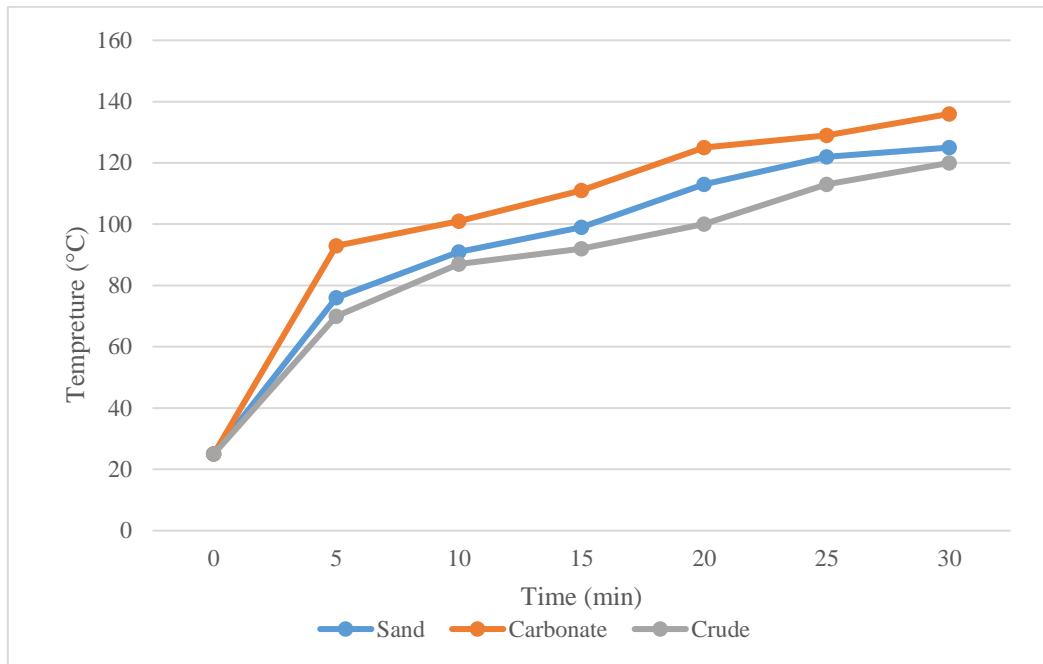
### **2.7. Pycnometer**

The oil density of the tested samples was obtained for IFT calculations at 25 °C using a pycnometer with a volume of 10 mL.

## **3. Results and discussion**

### **3.1. Temperature**

As we mentioned before, temperature changes are one of the results of using microwaves. The temperature changes of the two samples can be seen in Figure 1. It can be concluded that the increase in temperature for the oil sample combined with carbonate rock powder occurred faster. Temperature changes can be attributed to the presence of rock powder in the oil. The results of temperature changes are further discussed in the following sections.

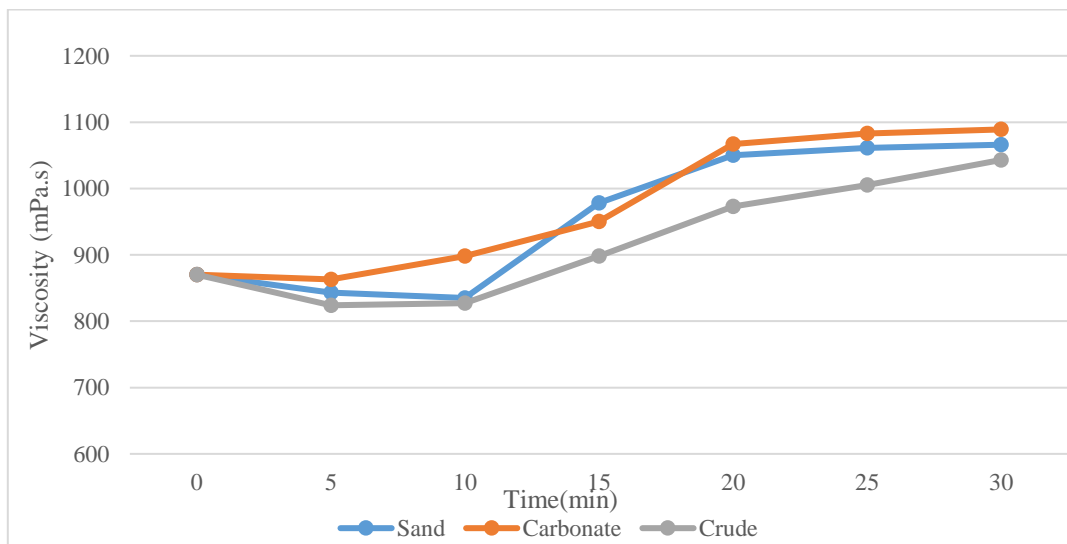


**Figure 1**

The variation in the temperature of the oil samples containing sandstone and carbonate rock powder with microwave radiation time.

### 3.2. Viscosity

By heating and changing the heavy oil compounds, microwave causes changes in oil viscosity. Cracking of heavy compounds of oil change oil viscosity. Figure 2 shows the changes in the viscosity of oil mixed with carbonate rock powder and sandstone.



**Figure 2**

The variation in the viscosity of the oil samples containing sandstone and carbonate rock powder with microwave radiation time.

Microwave radiation causes a decrease in viscosity in the first 10 min for the sample containing sandstone powder. After 10 min, an increase in viscosity is observed, and the increasing viscosity slope decreases over time. For the sample containing carbonate rock powder, the viscosity changes are almost constant in the first 5 min, and after this period, the changes are similar to other samples; an increase in viscosity is also observed. With microwave radiation, certain parts containing compounds with a high dielectric coefficient, such as asphaltene, start to heat up inside the oil, forming hot zones inside the oil (Taheri-Shakib Shekarifard et al., 2017). The asphaltene particles in these areas are cracked by the absorption of microwaves and turn into lighter compounds (Taheri-Shakib Shekarifard et al., 2018). This process reduces the viscosity of oil in the initial stages of radiation. After asphaltene particles crack, the temperature of other oil parts also rises. Thus, by the temperature increase, the light compounds of the oil are liberated from the surface, which causes an increase in viscosity (Taheri-Shakib Shekarifard et al., 2017; Taheri-Shakib Shekarifard et al., 2017). A similar process is observed for crude oil. In the first 10 min of radiation, the crude oil is associated with a decrease in viscosity due to the increase in temperature and the cracking of heavy oil particles. The viscosity increases with the continued radiation and evaporation of light hydrocarbons. As shown in Figure 1, the rate of increase in the temperature of the oil samples containing sand and carbonate rock powder is higher than that of the crude oil sample, so their viscosity increases later. With the continuation of radiation for up to 30 min, the viscosity of oil increases less due to the lower final temperature of the crude oil sample than the two other samples; as a result, less evaporation of light hydrocarbons is observed. Comparing the changes in the viscosity of the two tested samples shows that the increase in viscosity starts much faster in the sample containing carbonate rock than in the sample containing sand rock powder. It can be attributed to the faster increase in temperature and faster removal of light oil hydrocarbons of the sample containing carbonate rock. The temperature changes can be seen in Figure 1.

**Table 1**

The deviation and standard error of the viscosity change over time.

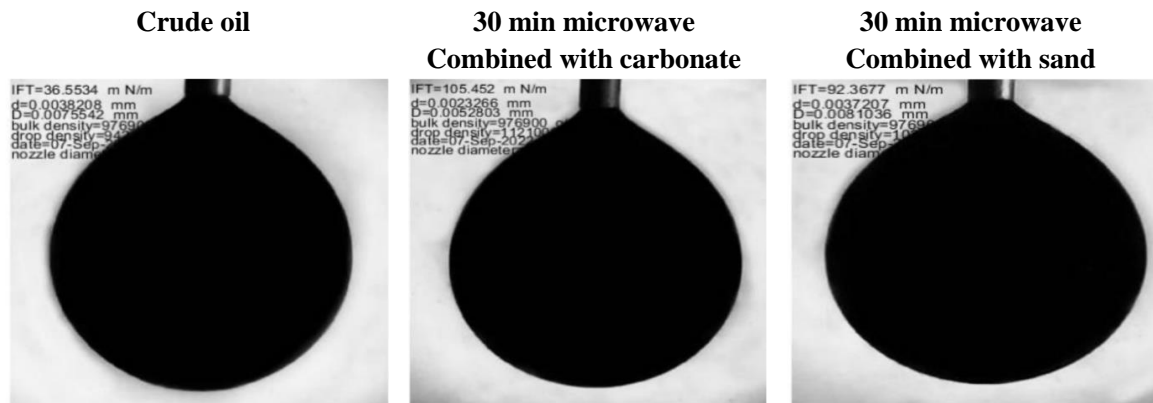
Time (min)	deviation	Standard Error
0	0	0
5	15.92342788	9.193395375
10	31.75251521	18.3323232
15	33.14614105	19.13693346
20	40.89824772	23.61261433
25	32.83629428	18.95804334
30	18.77942136	10.84230398

**Error! Reference source not found.** presents the standard error and standard deviation of viscosity change over time. From these two parameters, it can be concluded that the data dispersion increases with time and then decreases again. The most changes in data dispersion are in the first 10 min, which can cause a sudden increase in the temperature of the sample. As the radiation continues, the standard error decreases due to the evaporation of most light hydrocarbons in all the samples.

### 3.3. Interfacial tension

**Error! Reference source not found.** shows the images taken for IFT calculation. In this research, the pendant drop method measured the IFT of two oil samples combined with carbonate and sand rock

powder, which were exposed to 30 min of microwave radiation. For this purpose, after microwave radiation, the density of the samples was measured by a pycnometer after cooling, and IFT was obtained using MATLAB software.



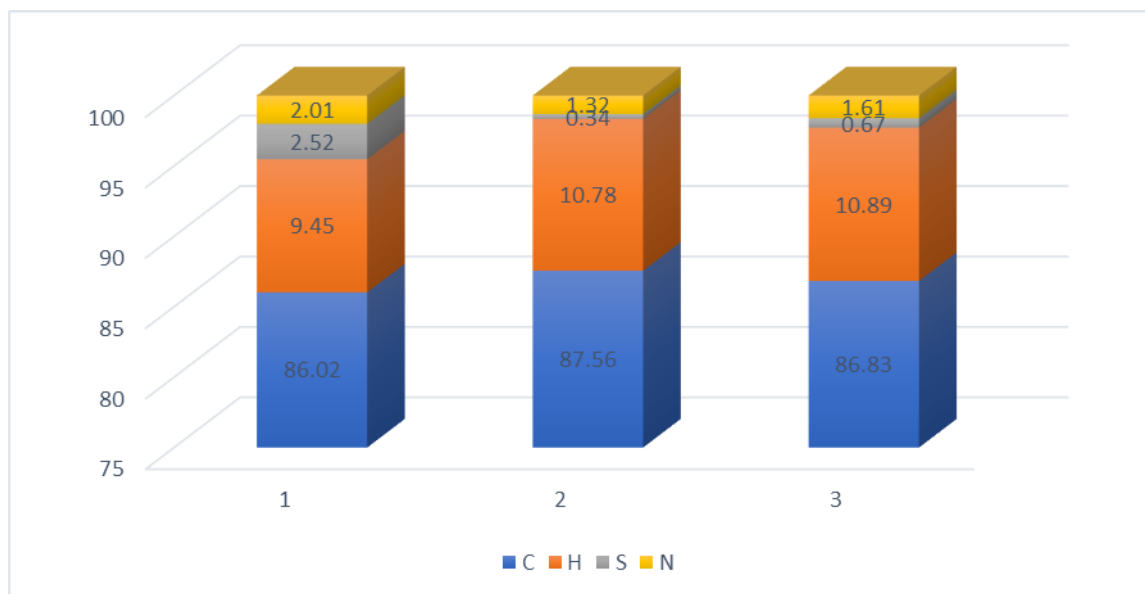
**Figure 3**

The variation in the IFT of the oil samples containing carbonate and sand rock powder under microwave radiation.

It can be seen that the IFT for the samples containing carbonate powder and sand is 105.452 and 92.3672 m N/m, respectively. According to the IFT of crude oil, which is 36.5534 m N/m, the IFT of both samples increases after 30 min of radiation. The increase in IFT can be attributed to the increase in temperature and evaporation of light oil compounds. It can also be seen that the variation in the IFT of both samples is almost the same, and with a slight difference, the IFT of the sample containing carbonate rock powder is higher. This slight difference can be attributed to the maximum temperature of the sample containing carbonate rock powder, which is about 10 °C higher than the other sample.

### 3.4. Asphaltene elemental analysis

CHNS analysis was used to determine the difference in microwave radiation in the presence of carbonate rock powder and sandstone. According to the IP143 test results, the amount of asphaltene after microwave irradiation decreases by 8.7% and 8.4% for carbonate rock powder and sandstone, respectively. **Error! Reference source not found.** shows the results of CHNS analysis for asphaltenes extracted from crude oil samples, asphaltene in crude oil combined with carbonated rock powder under microwave radiation, and asphaltene in crude oil combined with sand rock powder under microwave radiation.



**Figure 4**

CHSN analysis test results: 1) asphaltene extracted from crude oil; 2) asphaltene extracted from crude oil combined with carbonate rock powder; 3) asphaltene extracted from crude oil with sand rock powder.

As mentioned, elements with a high dielectric coefficient are more effective in absorbing microwaves, so microwaves separate elements such as sulfur, nitrogen, and oxygen from asphaltene particles (Taheri-Shakib Shekarifard et al., 2017). Figure 4 demonstrates that sulfur in asphaltene particles decreases for both samples with microwave radiation. For nitrogen, a slighter decrease is observed. Further, microwaves have a better desulfurization effect in the presence of carbonate rock powder because the amount of sulfur reduction for the sample containing carbonate rock powder and sandstone is 2.18% and 1.85%, respectively. According to the last parts of the research, it is also concluded that microwaves have a more significant effect on carbonate rock.

#### 4. Conclusions

This research investigated the changes in oil viscosity, temperature, interfacial tension, and asphaltene structure under microwave radiation in the presence of two different rock powders.

It is concluded that microwave radiation in both samples increases viscosity with the continuation of radiation. In this process, at the beginning of the radiation, certain parts of the oil begin to heat up. These parts generally contain particles with a high dielectric constant, like asphaltenes. Microwaves cause asphaltene particles to crack at the beginning of radiation and turn into lighter ones, so a decrease in viscosity is observed. This decrease in viscosity is more evident in the sample containing sand rock powder. As the process of microwave radiation continues, the heat is transferred to other parts of the oil, and the temperature of the whole sample increases, which causes a part of the light hydrocarbons in the oil to reach the boiling point and the viscosity to increase with the evaporation of them. It can be concluded that with the faster temperature increase of the sample containing carbonate rock powder, the evaporation process of light compounds starts earlier than the sample containing sand powder.

For this reason, in the first 5 min of radiation to the sample containing carbonate rock powder, despite the cracking of heavy oil particles, viscosity decreases are not evident. Therefore, carbonate rock causes better absorption of microwaves and faster oil temperature increase than the sample containing sand



powder. Crude oil samples also have similar changes in viscosity. Samples containing rock powder have a faster temperature increase, so the viscosity changes in samples containing rock powder occur faster.

By examining the calculated IFT of the tested samples and comparing them with the IFT result before microwave radiation, it is concluded that by increasing the temperature of the sample, the microwave causes the evaporation of light oil compounds and increases its density and IFT. Thus, the higher the temperature increase, the higher the changes in the IFT. Due to more changes in the temperature of the sample containing carbonate rock powder, the IFT of this sample increases slightly more.

The elemental analysis section used the CHNS test to determine the sulfur and nitrogen changes in asphaltenes extracted from two oil samples containing carbonate and sand rock powder. Microwave radiation causes a decrease in sulfur in both samples, which is slightly higher in the case of oil combined with carbonate rock powder. The changes in nitrogen also decrease, but the decrease in sulfur is more evident for both samples.

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