Journal of Chemical and Petroleum Engineering (JChPE)
Print ISSN: 2423-673X Online ISSN: 2423-6721

Experimental Investigation of the Influence and Comparison of Microwave and Ultrasonic Waves on Carbonate Rock Wettability

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ARTICLE INFO

Article History:
Received: 17 October 2022
Revised: 04 November 2022
Accepted: 06 November 2022

Article type: Research

Keywords:
Carbonate Rock,
Microwave,
Surface Charge,
Ultrasonic Waves,
Wettability Alteration

ABSTRACT

In this research, the influence and comparison of ultrasonic and microwaves on the wettability of carbonate rock have been investigated. Wettability is one of the most fundamental parameters of the oil reservoir, according to which the fluid movement in the porous medium can be examined. The aged thin sections were placed in a microwave oven and an ultrasonic bath and they were exposed to radiation for 2-10 minutes. Using the contact angle analysis, it was observed that the angle between the rock and oil drop of microwaved and ultrasonicated samples changed by 57 and 71 degrees, respectively. Contact angle and temperature changes started faster for the ultrasonicated samples. The surface charge of the rocks was determined by zeta potential analysis, and it was found that in both samples, in the first minutes of radiation, negatively charged colloids were liberated from the samples by absorbing the waves, which reduced the surface negative charges, and with the continued radiation, positively charged colloids were decreased due to the evaporation of light oil compounds. The reduction of zeta potential occurred faster for the ultrasonicated sample, but the rate of decrease was lower. By examining Fourier-transform infrared spectroscopy (FTIR) results, it was concluded that the heavy compounds on the surface of the samples were cracked and turned into lighter hydrocarbons, and the changes for both samples were almost equal. Also, the polar compounds, sulfur, and nitrogen in samples increased, decreased, and decreased respectively for both samples, and these changes were more for the ultrasonicated samples.

Introduction

Today, more than 85% of the global energy needs supply by fossil fuels, and fossil fuels have been the main source of energy for decades. Therefore, oil production with higher efficiency from oil reservoirs is important to supply the required energy. The natural energy of oil reservoirs is the main source of primary recovery energy, and it only extracts 5-15% of the original oil in place. Therefore, it is very important to use methods that increase the production rate from oil reservoirs [1- 5]. Wettability is one of the influencing parameters on the oil recovery factor and has a great impact on the fluid flow during production in reservoirs. It can also affect parameters such as capillary pressure, relative permeability, and irreducible water

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Journal of Chemical and Petroleum Engineering, 2022, 56(2): 341- 353.
Publisher: University of Tehran, College of Engineering *DOI: 10.22059/JCHPE.2022.350031.1411*

saturation [6, 7]. Most of the world's oil reservoir rocks are oil-wet [8-10] and according to initial research, changing the wetting of the reservoirs towards water wet can improve the recovery factor by replacing water in a porous medium [11]. Carbonate rock has a positive surface charge, therefore heavy oils, due to having acidic compounds with high polarity such as carboxylic acid, absorb the positively charged surface of the rock and make the rock surface more oil-wet [4, 12]. So, extracting oil from carbonate reservoirs is difficult due to the high absorption of oil by the reservoir rock [2]. It is possible to check the surface charge and wettability of reservoir rock with zeta potential, contact angle, scanning electron microscopy, and atomic force microscopy analyses [4, 12, 13].

One of the methods that can affect wettability is the use of ultrasonic waves. Ultrasonic waves are high-power mechanical waves with more than 20 kHz frequency and these waves are used in the oil industry to improve wettability, reduce oil viscosity, remove skin, rheology improvement, reduce heavy precipitation of oil such as asphaltene and sulfur reduction [14-16]. To use ultrasonic waves, a resonator is placed near the pay zone and sent into the well by wireline [17]. Another way to change the wettability is to use microwaves. Microwave a part of electromagnetic waves with a frequency between 300 MHz- 300 GHz, which are created by the interaction of magnetic and electric fields [18]. Microwave is also used in the oil industry for oil viscosity reduction, oil upgrading, sulfur reduction, and asphaltene precipitation reduction [19, 20]. To radiate microwaves to the reservoir, an antenna is used that is sent into the well by wireline [17]. Microwave has the property of heating certain parts of oil. Parts of oil that have a higher dielectric constant are heated more by absorbing more microwaves. Therefore, increasing the temperature in certain parts of the oil cracks the heavy compounds in this area and turns them into lighter hydrocarbons [21-23]. Jaber Taheri-Shakib et al. 2018 investigated the effect of microwaves on the wettability of carbonate reservoir rock. By examining the changes in the surface charge of rocks, oil structure, and the contact angle between oil drop and rock, they concluded that with increasing microwave irradiation time, rock samples become more water wet [24]. Karami et al. 2021 concluded that the improvement of rock wettability with the use of microwaves is not only dependent on the changes caused by the temperature increase. By weakening van der Waals forces, microwave caused the cracking of organic compounds on the rock surface and changes in wettability [25]. Hui Shang et al. [26] examined the effect of microwaves on the viscosity of crude oil and concluded that changes in viscosity depend on the compounds in the oil. after microwave irradiation, a high amount of oxygen compounds would cause an increase in oil viscosity. Karami et al. [18] investigated the removal of condensate blockage using microwave and ultrasonic. They concluded that ultrasonic waves and microwaves had a greater effect on the removal of light oil compounds compared to heavy compounds. They also concluded that the effect of microwave and ultrasonic depends on the dielectric constant and acoustic properties of the compounds in the oil, respectively. Jaber Taheri-Shakib et al. [27], by comparing the effect of ultrasonic and microwave on asphaltene structure, concluded that these waves make asphaltene particles smaller. In the microwaved samples, the asphaltene particles are smaller than in the ultrasonicated samples, and the molecular changes of the asphaltene in the microwaved samples are more.

In this research, the effect of ultrasonic waves and microwaves on the wettability of carbonate rock has been investigated and compared. After rock aging, they were exposed to microwave and ultrasonic radiation for 2-10 minutes. Contact angle analysis, Fourier transform infrared (FTIR) and zeta potential has been used to investigate the wettability changes due to wave radiation.

Materials and method

In the experiments, oil from an oil reservoir in the southwest of Iran was used. To investigate the effect of microwave and ultrasonic waves on rock wettability, a carbonate rock sample was used. The FTIR and XRF test results of the rock powder used are given in Table 1. According to Table 1, the main components of the rock include Ca CO3. First, thin sections with dimensions of 2 x 3 x 1 cm, and a completely flat surface were prepared from the rock, and it was washed with toluene for 3 days and then with methanol for 1 day to be completely clean. In the next step, the thin sections were placed in a cell filled with oil and it was pressurized to 1000 psi so that the oil penetrates the rock porous medium. In the next step, the thin sections were kept at 80°C for 30 days to age. After drying, the thin sections were placed in a microwave oven and an ultrasonic bath, and waves were radiated to them for 10 minutes in 2-minute intervals with maximum power, and in each interval, the temperature of the samples was measured using a laser thermometer (ZOTEX). Then, FTIR, zeta potential, and contact angle analysis were used to investigate the wettability changes of thin sections and compare the effect of ultrasonic waves and microwaves on wettability.

Table 1. XRF results of carbonate rock powder

Compounds	**%**
L.O.I.	42.8
Na_2O	0.024
MgO	0.553
Al2O3	0.192
SiO_2	0.657
P_2O_5	0.078
SO3	0.285
Cl	0.006
K2O	0.033
CaO	54.736
Fe_2O_3	0.0135
Sr	0.511

FTIR analysis was taken from the powdered sample of thin sections aged with oil. Therefore, the peaks related to calcite are also visible in the FTIR diagram together with the hydrocarbon peaks. To separate these peaks from each other, an FTIR test was taken from the rock powder before aging, which can be seen in Fig. 1. Table 2 shows more detail about the peaks related to carbonate FTIR, which will compare with the hydrocarbon peaks in the results section.

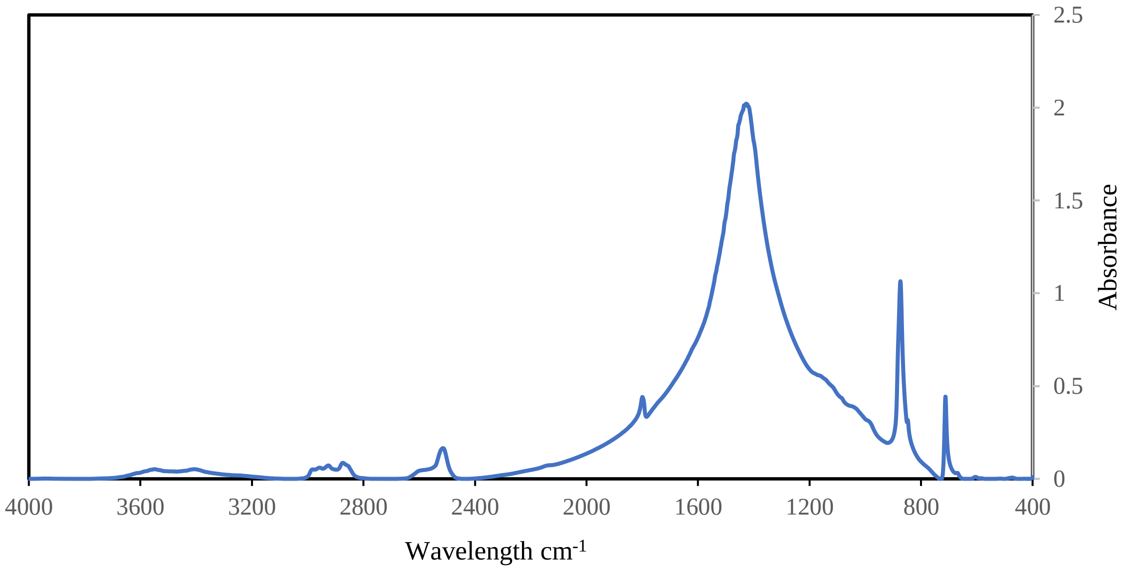

Fig. 1. the FTIR spectra results of carbonate rock powder

Table 2. FTIR spectra peak analysis of carbonate rock powder [28-31]

Peak	Assignments
3550	O-H, N-H, Mg-OH stretching
3408	O-H, N-H, AL2SiO5
2926	CaCO3
2873	CaCO3
2515	CaCO3
1799	CaCO3
1431	CaCO3
874	CaCO3, C-O, Si-O-Al, Al-O,
712	CaCO3, NH2, SiO4, Si=O=Si

Ultrasonic Bath and Microwave Oven

An ultrasonic bath with a frequency of 37KHz and maximum power (Pm) of 850 watts with the ability to adjust the power as Pm, 0.9Pm, 0.8Pm, 0.7Pm, 0.6Pm, and also a microwave oven with a maximum power of 1300 watts and operational frequency of 2.45 GHz with the ability to adjust the power as Pm, 0.8Pm 0.6Pm 0.4 and 0.2 Pm were used in the experiments.

Contact Angle Analysis

One of the most important effective parameters in fluid movement in the porous medium is wettability [32]. Therefore, in the oil industry, the use of methods that are effective on wettability is important to wash the oil inside the porous medium and replace it with water. The contact angle test was used to determine the changes in the wettability between carbonate thin sections and crude oil drop. The thin sections were placed in distilled water and an oil drop with a volume of 6µL was placed on the surface of the rocks from below after 10 minutes, when the contact angle of the oil drop is fixed, it is photographed by a camera. Then the contact angle of the oil drops has been measured by Digimizer software.

FTIR Analysis

FTIR tests were conducted to determine functional groups present in powdered thin-section samples [33]. During the FTIR spectroscopy process, rock powder that have been aged after wave radiation processing was mixed with potassium bromide (Merck, Germany) at a ratio of 1:40 to make a pallet. FTIR was examined at wavelength 400 to 4000 cm^{-1} and obtained in the absorbance mode. The effect of microwave and ultrasonic waves on the functional groups was investigated by examining the changes in the integrated area under the diagram peaks (Origin software has been used for calculating the area of the peak) (This method is based on the Karami et al. work [34]).

Zeta Potential

Zeta potential test was used to obtain variations in the electric charge of samples exposed to wave radiation [24]. the aged thin section samples were crushed into fine powders, and then to test zeta potential, 0.1 gr of the powdered thin sections, that have been aged by oil and radiated by microwave and ultrasonic waves, was added to 9.9 mL distilled water and stirred for one 24 hours with a magnetic stirrer. After 10 minutes of microwave and ultrasonic radiation, the PaticleMetrix device has been used to determine the zeta potential of powdered samples. Zeta potential technique can identify the heterogeneity of rock surface and wettability profiles of radiated samples [25, 35].

Results and Discussion

Temperature Changes

The changes in the temperature of rock samples under microwave and ultrasonic radiation are shown in Fig. 2. Microwave energy converts into thermal energy by changing the direction of the electric dipole of polar compounds. In this way, the kinetic energy of compounds with high dielectric Constance increases and heats up by being placed in the direction of the created magnetic field [34, 36, 37]. On the other hand, the ultrasonic waves also cause the material to heat up by emitting and losing part of its energy. The US cavitation and mechanical vibration increase the oil temperature. This process mostly depends on the ultrasonic cavitation that happens at the interphase boundary of the molecules [38]. Therefore, the temperature rise of tested samples depends on the acoustic and thermal properties of the oil. The increase in temperature may cause physical and chemical changes in the oil. These changes include viscosity, IFT, evaporation of light oil compounds, and heavy compounds cracking in the oil [39]. By comparing the temperature changes, it can be seen that the samples under ultrasonic radiation had a higher and faster temperature increase.

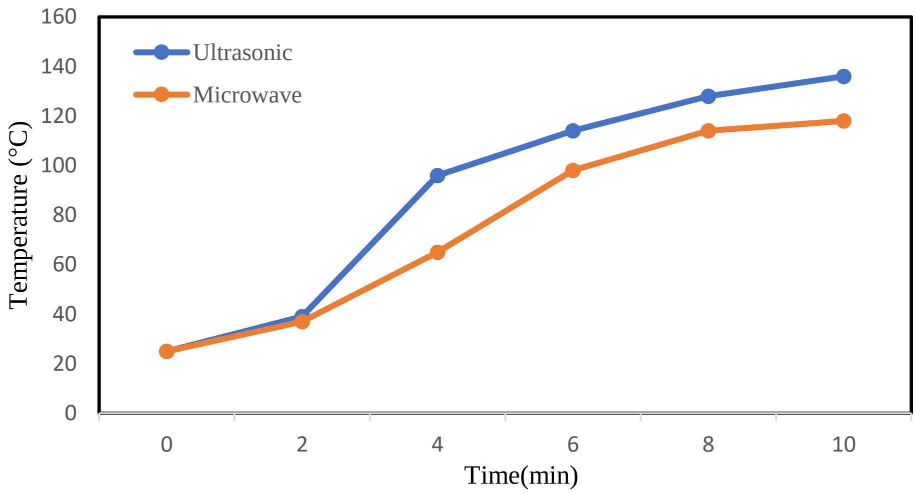

Fig. 2. temperature changes of aged thin sections according to microwave and ultrasonic waves irradiation time

Contact Angle

Fig. 3, Fig. 4, and Fig. 5 show the contact angle changes of an oil drop on thin sections radiated by microwave and ultrasonic waves, respectively. The thin sections which aged by the oil were exposed to microwave radiation for 2-10 minutes and then placed in water, after that a drop of oil was placed on their surface from below. In each experiment, 3 drops of oil with a volume of 6µl were placed on different parts of the rock and photographed.

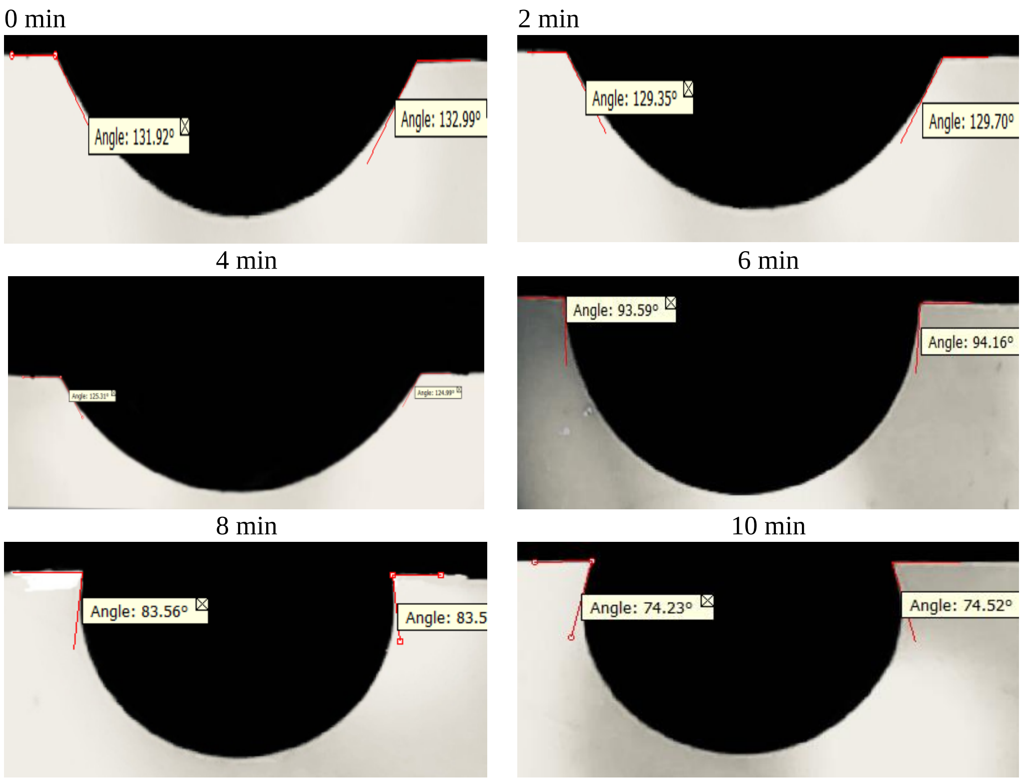

Fig. 3. The contact angle between aged thin sections and crude oil after exposure to microwave for 2, 4, 6, 8, and 10 min

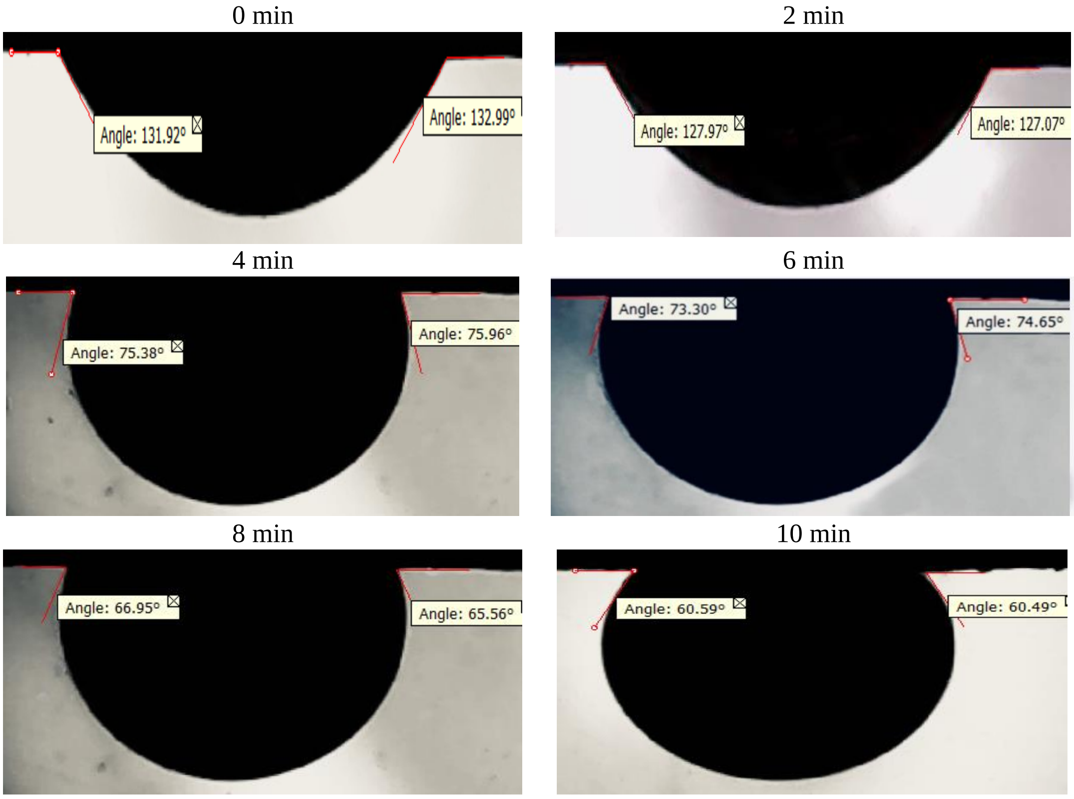

Fig. 4. The contact angle between aged thin sections and crude oil after exposure to Ultrasonic waves for 2, 4, 6, 8, and 10 min

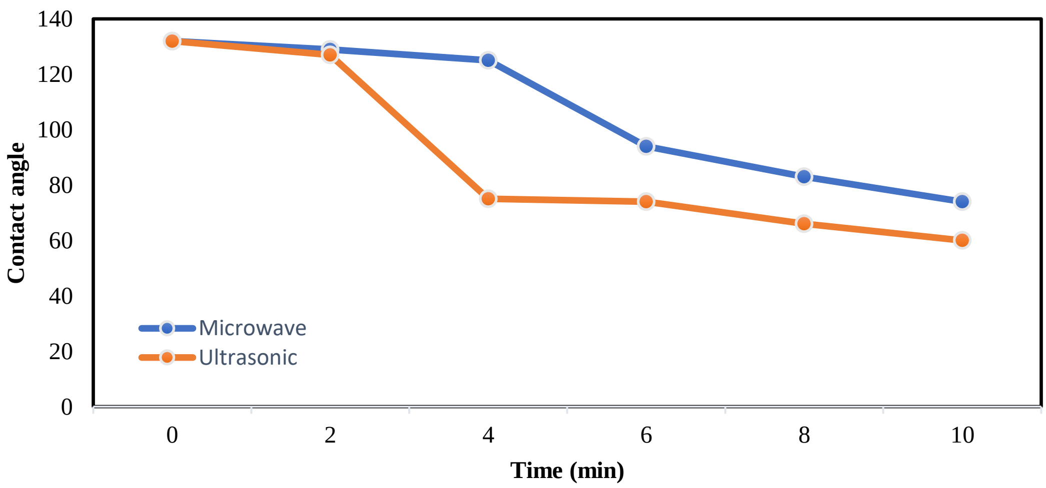

Fig. 5. The contact angle change of microwaved and ultrasonicated samples

The increase in temperature causes the evaporation of hydrocarbons on the surface of the rock, which makes the rock surface more water wet. Microwaves have different effects on different parts of oil, so the parts with a high dielectric coefficient, which have more microwave absorption properties, are more affected than other parts [22, 40-42]. Polar parts and SNO elements in oil have a high dielectric coefficient. Asphaltene precipitation is one of the most polar compounds in crude oil, and its complex molecular structure includes SNO elements, therefore, these particles have a high absorption capacity of microwaves. So, microwaves, by affecting these particles, cause them to crack and turn into lighter hydrocarbons [23, 34, 41]. Therefore, according to Fig. 5, at the beginning of microwave radiation, heavy oil particles are cracked into light hydrocarbons, and due to the increase of hydrocarbons on the rock surface, the contact angle does not change significantly. After 4 minutes of irradiation, with a gradual increase in temperature and reaching the bubble point, the evaporation of light hydrocarbons of the oil begins. As a result, the contact angle starts to decrease and the rock surface becomes more water wet. Also, by increasing the temperature and reaching the bubble point, ultrasonic radiation causes the evaporation of light hydrocarbons on the rock surface and the reduction of the contact angle. A sharp decrease in the contact angle (52 degrees) occurred in 2-4 minutes. By comparing the results in Fig. 2 with contact angle changes in ultrasonicated samples, it can be seen that sudden temperature changes also occur within 2-4 minutes. Therefore, the changes in the mentioned period are related to temperature increase. So, it was concluded that the radiation of waves and temperature increase in both samples caused the cracking of heavy oil particles and also the reduction of hydrocarbons due to their evaporation.

Zeta Potential

In a continuous dispersing medium, zeta potential is used to determine the degree of electrostatic repulsion between similarly charged colloidal particles [43]. Microwave and ultrasonic radiation affect these charged colloidal clusters by affecting the molecular structure of the oil. Fig. 6 shows the results of the zeta potential test for powdered samples.

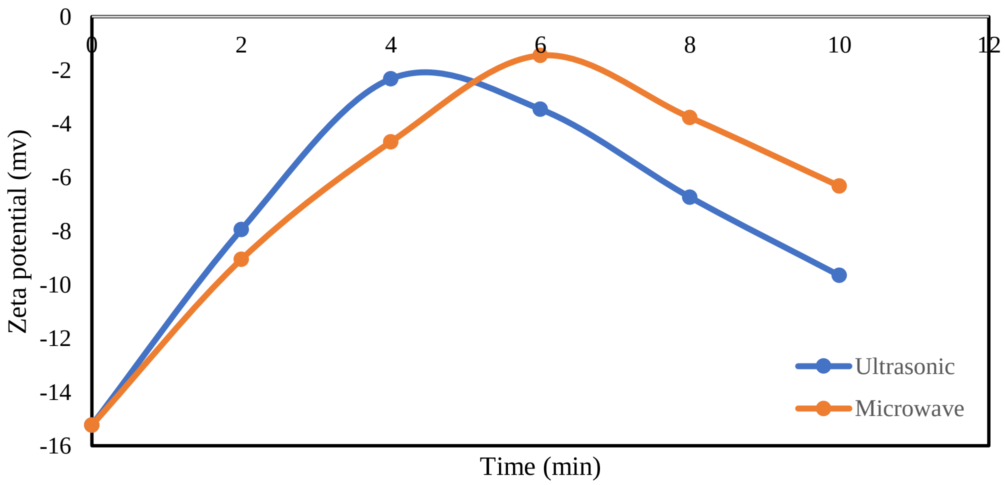

Fig. 6. The Zeta potential test results for samples under ultrasonic and microwave at different time ranges

At the beginning of microwave and ultrasonic radiation, compounds such as sulfur and oxygen with negative electrical charges, which have a high ability to absorb these waves, begin to be released from the oil, and this process reduces the negative charges for both samples (Fig. 6) [24, 25]. This process has been observed until 4 and 6 minutes for ultrasonicated and microwaved samples, respectively. Therefore, the reduction rate of negative charges is higher for ultrasonicated samples than for microwaved ones. As the radiation continues and the temperature increases, heavy oil particles such as asphaltene begin to crack with the radiation of waves and turn into lighter compounds [18, 21, 22, 26]. This process causes the increase in positively charged hydrogen and nitrogen in oil and the zeta potential begins to decrease [24, 25]. Due to the faster temperature increase of the ultrasonicated sample, the mentioned process happens faster. By examining the peak in Fig. 6, it can be seen that the negative charges have decreased more for microwaved samples than the ultrasonicated ones. Therefore, it can be concluded that microwave reduces the negative particles in oil at a lower speed but a greater amount than ultrasonic waves.

Fourier-Transform Infrared Spectroscopy (FTIR)

FTIR spectra were used to determine the functional groups and wettability changes of aged thin sections. The samples were tested before and after 10 minutes of ultrasonic and microwave radiation. First, in Fig. 7, the rock powder peaks must be identified and separated from peaks related to oil hydrocarbons. By examining Table 1 and Fig. 1, calcite in carbonate rock powder has a high-intensity peak at wavelengths of 712, 874, 1431, 1799, and 2515 cm^{-1}, so mentioned peaks cannot be analyzed. Therefore, it is possible to determine the peaks related to hydrocarbons, and by calculating the area under the diagram, the changes in hydrocarbon bound and functional groups can be determined [44].

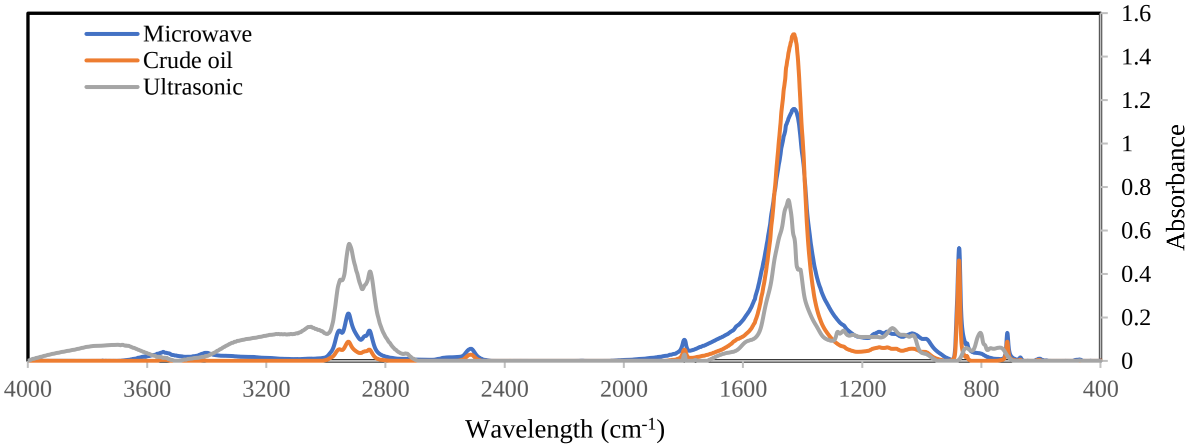

Fig. 7. The normalized FTIR spectra

Table 3. The calculation of integrated area ratios for FTIR spectra

	Crude oil	Microwave	Ultrasonic
polar/Total	0.004	0.148	0.319
S=O/Total	0.133	0.103	0.030
C-N / Total	0.222	0.166	0.075
CH3/Total	0.084	0.076	0.097
CH3/CH2	0.285	0.266	0.243

In FTIR spectroscopy, the wavelength of 2700-3500 cm^{-1} indicates O-H, N-H, and C-H bonds, hence it is called the hydrogen stretching zone. More precisely, polar bonds including O-H and N-H are defined in the range of 3000-3500 cm^{-1} [21, 24]. Changes in polar bonds on the rock surface can be related to its wettability. By examining the polar compounds in Table 3, it can be concluded that the polar bounds have been increased on the rock surfaces compared to other bounds with ultrasonic and microwave radiation. The increase is more evident for ultrasonicated samples and considering that the increase of polar compounds on the rock surface makes the rock more water wet, the FTIR results are compatible with the contact angle test results. Also, in the hydrogen stretching zone, the wavelengths of 2723-2879 cm^{-1}, 2879-2944 $cm^{-1,}$ and 2944-3000 cm^{-1} are referred to CH2 symmetric Stretch, CH2 asymmetric stretch and CH3 asymmetric stretch, respectively [18, 22, 25]. CH3 mostly indicates lighter hydrocarbons and CH3/CH2 ratio can be used to measure the lightness and heaviness of oil [18]. By checking Table 3, it can be seen that the ratio of CH3 increased and decreased with ultrasonic and microwave radiation, respectively. The decrease in CH3/CH2 ratio also indicates the lightening of oil compounds on the surface of the rock, and the cracking of heavy oil particles was also obtained in contact angle test results. In FTIR, the wavelengths of 990-1043 cm^{-1} and 1053-1133 cm^{-1} correspond to S=O (in sulfoxide) and C-N (in aliphatic amine) peaks, respectively [18, 25, 45-48]. The proportion of sulfoxide bonds in the aged rocks has decreased with the radiation of both waves, and the decrease in ultrasonicated samples is much more intense than the microwaved ones. For the C-N bond, a decrease was observed for both samples, but unlike S=O bounds this decrease was more intense in microwave radiation.

Conclusion

In this research, the effect of ultrasonic waves and microwaves on the wettability of an aged carbonated rock has been examined and compared. Ultrasonic waves lose part of their energy inside the irradiated sample and create the phenomenon of cavitation in the trapped oil. The temperature of aged rocks increases by ultrasonic radiation and the temperature increase depends on the acoustic and thermal properties of rock and oil. On the other hand, microwave also heats the samples by creating a magnetic field, and interaction between the polar compounds supplies the energy for temperature increase. The temperature increase was higher for ultrasonicated samples.

The effect of ultrasonic and microwave on rock wetting was investigated using the contact angle test. At the beginning of the radiation, the waves crack heavy compounds and increased the light hydrocarbons on the surface of the rock. This process caused the contact angle to remain almost constant for ultrasonic and microwave in the first 2 and 4 minutes respectively. Then by the increase in temperature and reaching the boiling point, lighter compounds began to evaporate from the rock surface, and rocks starts to become more water wet, due to the faster increase in temperature of ultrasonicated samples, the contact angle results are consistent with temperature changes results.

Zeta potential test was used to identify the changes in charged colloidal particles in the samples. It was concluded that at the beginning of the radiation, in both samples, the waves affected the negatively charged particles in the oil (oxygen and sulfur radicals) and caused a decrease in the concentration of negatively charged particles. By the continuation of the radiation, with the increase in temperature and cracking of heavy hydrocarbons, the positively charged particles (nitrogen, hydrogen, and carbon) are released from oil, and the concentration of negatively charged particles increased again. This process also happened faster for ultrasonicated samples, but the concentration of negative surface charge decreased more in microwaved samples.

FTIR spectroscopy was used to check the changes in functional groups and bonds in the powdered samples. After determining the peaks related to hydrocarbons, the changes in hydrocarbon bonds were determined by calculating the area under the diagram. In FTIR results, there was an increase in polar compounds in both samples, which was higher for the ultrasonicated ones. An increase in polar compounds directly affects rock wettability. Also, the waves at the beginning of radiation cause heavy oil particles such as asphaltene to crack and turn them into lighter hydrocarbons. As the radiation continues, the increase in temperature causes lighter hydrocarbons to evaporate from the rock surface, which makes the rock more water wet. The bonds related to sulfur and nitrogen decreased for both samples, and this decrease is more for the ultrasonicated sample.

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How to cite: Yazdani B, Saeedi Dehaghani AH. Experimental Investigation of the Influence and Comparison of Microwave and Ultrasonic Waves on Carbonate Rock Wettability. Journal of Chemical and Petroleum Engineering. 2022; 56(2): 341-353.