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OPTIMISATION PROCESS FOR DEMULSIFICATION OF WATER-OIL EMULSIONS USING MICROWAVE HEATING ASSISTED WITH CALCIUM CARBONATE

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Abstract

Emulsion formation is considered one of the major challenges in oilfields, production and transportation of crude oil. It affects the downstream processing units and causes serious corrosion problems, as well as increasing the volume ratio of liquid and viscosity. The present work focuses on an alternative way to break up the emulsions using microwave irradiation techniques assisted with inorganic salts. The experimental work was designed according to the central composite rotatable design method (CCRD) to investigate the effect of operating variables irradiation power from 200 to 1000 W and treatment time from 40 sec to 200 sec, using 2 g of inorganic salt (Calcium Carbonate) in accordance with its ability to neutralise acidic water and prevent corrosion, in addition to increasing microwave irradiation and improving separation efficiency. The emulsion was prepared according to a standard method of 30 vol.% and 40 vol.% water-oil emulsion. The results revealed that the optimum separation efficiency was obtained at 800 W, 160 seconds, 102 °C, and 2 g of calcium carbonate, with 87.5% separation efficiency at 40 vol.% of water emulsion. Also, in the other case of emulsion, the optimum results revealed as 800 W, 160 seconds, 99 °C, 2 g of calcium carbonate, with 79.5% separation efficiency at 30 vol.% of water emulsion, cost saving rate was 86.64% for 40 vol.% emulsion, and by 85.24% cost saving for 30 vol.% emulsion. The results proved that the electromagnetic wave is an applicable technology to break up water-oil emulsions effectively.

Keywords: Calcium carbonate, Crude oil, Microwave irradiation, Optimisation, Separation.

1. Introduction

Water-in-oil emulsions in the petroleum sector can cause issues with crude oil transportation, storage, and pipeline damage [1, 2]. Emulsions increase the viscosity of crude oil, causing problems in pipelines [3], downhole equipment damage [4], and downhole separator performance reduction [5, 6]. Demulsification is crucial to maintain high crude oil quality. Heavy crude oil emulsions are more stable than light emulsions due to their high-molecular-weight naphthene and frictional resistance [2, 7, 8]. High temperatures affect the stability of these emulsions, requiring frequent demulsification [9, 10].

Understanding emulsion production and stabilisation is crucial for better separation of water from crude oil. Alkali Surfactant Polymer (ASP) flooding stabilisation of oil/water emulsion was studied and reported by [10, 11]. Microwave irradiation reduces thermal gradients, saves energy, and damages polar molecules [12]. Emulsion formation occurs in three stages: flocculation, coalescence, and Ostwald repining, as shown in Fig. 1. During flocculation, dispersed droplets form a big group that remains uncoated. Coalescence combines all droplets into a single drop, while Ostwald repining causes big internal droplets to grow at the expense of smaller ones, resulting in the emulsion's breakdown [13, 14].

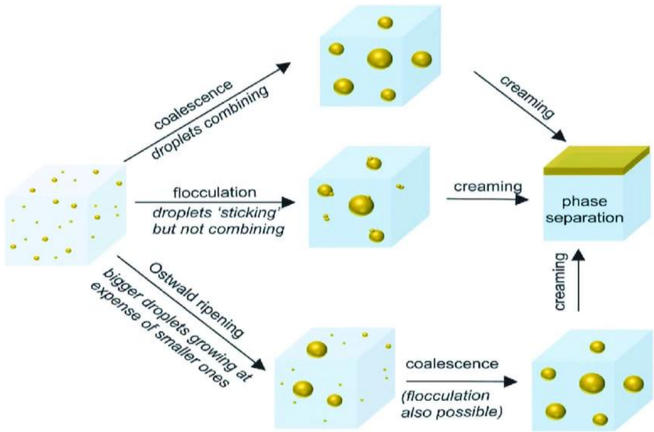


Fig. 1. Water-in-oil emulsion formation (Gupta et al. [15]).

Emulsions are classified into oil/in/water (O/W) and water/in/oil (W/O) based on the surfactant's solubleness. W/O emulsions are commonly used in industries like food, cosmetics, pharmaceuticals, and agrochemicals, providing functional molecules or texture [16]. Stabilisation of the inter-facial coating is crucial for crude oil emulsions, preventing droplet coalescence. Surfactants or emulsifiers are needed to minimise interfacial tension [17]. Asphaltenes stabilise oil emulsions due to their strong viscoelastic network at oil-water interfaces. Factors that impact stability include medium aromaticity, resin fraction concentration, and asphaltene polarity [18]. Researchers used a turbiscan Lab expert analyser to assess emulsion processes, identifying emulsification tendencies. Microwave demulsification is efficient, sustainable, and efficient, enhancing separation efficiency [19]. Martínez et al. [20] showed that electromagnetic demulsification is classified into a number of the following mechanisms:

i. Microwave irradiation caused polarised water molecules and charged liquid beads to rotate, causing charge displacement and potential zeta at the O/W interface, causing asphaltene and resins to dissolve.
ii. Water molecules consume microwaves more effectively than oil molecules, causing water droplets to expand, while thinner interface films have lower mechanical strength and are more likely to break.
iii. Microwaves create a magnetic field that attracts non-polar oil molecules, creating a vortex parallel to their axes, weakening intermolecular interaction, reducing oil viscosity, and accelerating demulsification and dehydration.
iv. Polar molecules' dipole relaxation causes instant heat in the solution, causing droplet viscosity to decrease and movement speed to accelerate, resulting in the formation of beads.

Microwave demulsification reduces interfacial tension by heating aquatic phases, reducing viscosity, and preserving energy. Experiments confirm this hypothesis, constructing a model for droplet formation [21]. Microwave-heated emulsions separate faster than traditional ones due to reduced Zeta potential and increased demulsification efficiency [22]. Hyde et al. [23]. Microwave irradiation affects water/alkane interactions and the breaking performance of crude oil emulsions [24]. High conductivity in both oil and water makes microwaves ideal for demulsifying oil emulsions with varying oil-to-water ratios. This method is particularly beneficial for heavy oil and polymeric well liquids with high mineralisation but low crude oil and water density. The microwave operation principle relies on the magnetron, which generates microwaves through LC Circuit Resonance [25]. Figure 2 shows a microwave mechanism.

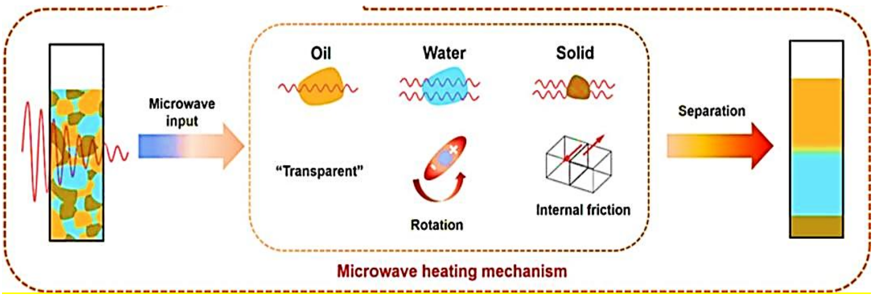


Fig. 2. Microwave mechanism for separation process (Murungi and Sulaimon [26]).

Previous research has utilised microwave heating for separating water/oil emulsions. This method uses the principle of dielectric heating, causing inner heating due to molecule rotation and ionic conductivity [27]. The optimal microwave radiation power and exposing period are estimated to be 420 W and 12 seconds, with a separation efficiency of about 95% [28]. Microwave demulsification does not require chemical additions and may be a substitute for traditional demulsification techniques. The thermal impact of microwave heating results in enhanced settling times [29]. The presence of polar composition and their interactions within the emulsion is critical to emulsion stabilisation [30].

The main goal of this work is to generate a cost-effective and environmentally friendly technique for water-oil emulsion separation using microwave irradiation.

This study investigates the generation and stability of W/O emulsions of Iraqi heavy fuel oil using eco-friendly, inorganic salts. It compares results with previous research [31, 32]. Multiple samples of heavy Iraqi crude oil under the effect of different treatment times and irradiation power have been performed. Also using inorganic additives to improve microwave irradiation and improve the emulsion separation process. This work utilised microwave irradiation to separate the water in Iraqi heavy crude oil emulsions using calcium carbonate, which is the first time testing this technique locally instead of traditional heating, which causes a lot of pollution and environmental hazards and also greater expenses.

2. Materials and Methods

2.1. Materials

In the present work, Heavy Iraqi fuel oil was utilised, which was collected from the AL-Dorah refinery (Basra heavy oil) with the following specifications as listed in Table 1.

Table 1. Basra heavy fuel oil Specifications.

Test	Result
Kinematic viscosity at 40 °C (cSt)	12.36
Density at Obs. Temp	0.8814
API Gravity@ 40 °C	28.8
Water Cont. (Vol.%)	0.1
Salt content (Wt.%)	0.0098
Density @15°C gm/ml	0.8850
R.V.P kg/cm^2	0.60

Calcium carbonate powder was used in experimental work as an inorganic combination. It could be found in the crust of the earth and the crust of eggs. Also, it possesses a very strong electrostatic force of attraction; therefore, all atoms are very closely linked together. The attraction is so powerful that it is hard to separate them apart. This is what occurs when calcium carbonate dissolves in water. Calcium carbonate is also used in some techniques for Purifying water from impurities and toxicity and preventing corrosion to the equipment, so it was a very good addition to the current work. Also, efficiency rose by using it as a result of inorganic salts destroying double charge layers.

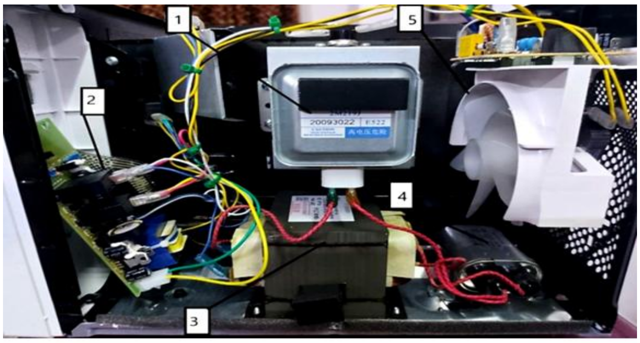
2.2. Experimental equipment

Electromagnetic heating was utilised by using a microwave reactor, which can affect oil emulsions by reducing the viscosity of oil and allowing the water drops to collide and gather in the current experiment. There are multiple advantages of the reactor, such as precise control over time. Also, it possesses high safety factors and a high degree of precision when providing electromagnetic radiation to the sample using fixed frequencies and a variable quantity of power. The working principle of the device relies on converting electromagnetic energy into thermal energy exposed to the fluid particles. When electromagnetic waves affect the fluid particles, it will cause the molecules to rotate and lead to friction between the molecules which leads to the generation of thermal energy from the inside to the outside of fluid. The frequency of the wave is about 2450 MHz. The main part which will be responsible generation of electromagnetic energy among other devices is denoted as the magnetron, in addition to other parts such as high voltage transformers, which have output ranges between 3000-4000 Volts, cooling fan, waveguide, control unit and a high voltage capacitance of 2100 V.AC and 1 μF. Figures 3 and 4 show the experimental apparatus.

1:	Stop and start button	6:	Water entering the condenser
2:	Control buttons	7:	Condenser of rising vapors
3:	LED display screen	8:	Water tank
4:	Glass beaker	9:	Water pump
5:	Water from the condenser		

Fig. 3. The microwave heating reactor.

A high-voltage capacitor is connected to the secondary winding of the transformer, and the other side of the capacitor is connected to a diode and a magnetron and then grounded. The other side of the magnetron is connected to the central thin coil of the high-voltage transformer.



1:	Magnetron
2:	Panel of controlling
3:	High-volt transformer ranges from 220-4000 volts
4:	High volt capacitance 2100 volt
5:	Fan for cooling.

Fig. 4. The electromagnetic heating circuit diagram which is used in voltage conversion.

A magnetic stirrer is also used, which is a laboratory device used for mixing samples to create emulsions. It consists of two magnets; one attached to an electric motor and the other immersed in liquid. When the lower magnet spins, the immersed magnetic rod rotates without mechanical contact, minimising friction, oxidation, or liquid material escape. The magnetic stirrer is shown in Figs. 5 and 6.

Fig. 5. Hot plate and magnetic stirrer.

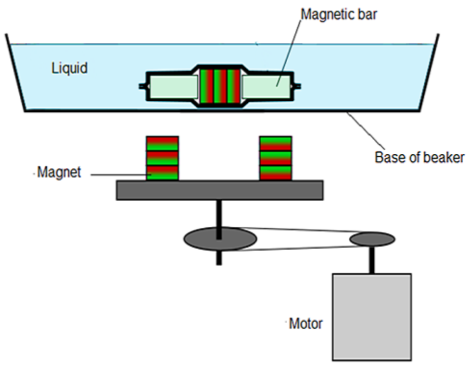


Fig. 6. Schematic of hot plate and magnetic stirrer device.

2.3. Measuring equipment

Multiple pieces of equipment have been used in work as listed in Table. 2 with a brief description.

Table. 2. Measuring equipment.

Unit	Description
Graduated Cylinder	laboratory equipment that is used for measuring the level of fluid or volume of a liquid.
Graduated Beaker	laboratory equipment which has been used for preparing emulsions. It has been used for measuring the amounts of distilled water added and the amount of heavy crude oil.
Electronic Weighing Scale	The sensitive weighing device has been used for measuring the amounts of materials added, such as the dosages of calcium carbonate that has been added to the heavy crude oil emulsions.
Temperature Measuring Device	microwave reactor presents a difficult challenge because most of the temperature sensors and measuring instruments are made of metal; therefore, after a deep search for the types of temperature sensors and the materials they are made of, we found that digital thermometer is good for measuring because the material of the probe is made of stainless steel and withstand high temperature.

2.4. Preparation of samples and testing procedure

According to CCRD (Central Composite Rotatable Design) in Tables 3 and 4, twenty experiments for each case were conducted in order to achieve the impact of irradiation time and power emitted, as research variables, on the separation efficiency of Iraqi crude oil, the procedure done as the following steps below.

Table. 3 Central composite design (CCRD).

Factor	Symbol	Used ranges of variable				
		-2	**-1**	**0**	**+1**	**+2**
Power Emitting (W)	X1	200	400	600	800	1000
Irradiation Time (sec)	X2	40	80	120	160	200

Table. 4. Sequence of experimental work due to (RSM).

Run	Coded values		Real values		% of water separation 40% vol.	% of water separation 30% vol.
	Power emitted (X1)	Irradiation Time (X2)	Power Emitted (Watt)	Irradiation Time (sec)		
1	+1	-1	800	80	42	34
2	-1	+1	400	160	17	10.5
3	-1	-1	400	80	15.5	7
4	+1	+1	800	160	54	46
5	+1	0	800	120	51	43
6	0	+1	600	160	34	26
7	-1	0	400	120	16	9
8	0	-1	600	80	28	20.5
9	0	0	600	120	31	23
10	0	+2	600	200	35	27
11	+2	0	1000	120	50	43
12	0	-2	600	40	24	16.5
13	-2	0	200	120	14	8
14	0	0	600	120	31	23
15	0	0	600	120	31	23
16	+2	+2	1000	200	48	39
17	+1	+2	800	200	52	44
18	0	-2	600	40	25	16.5
19	-1	+2	400	200	18.5	11.5
20	-2	+2	200	200	14.5	10

i. Preparing heavy crude oil samples of emulsions in 500 ml graduated beaker by adding specified amounts of distilled water and oil depending on the concentration of water that needed 40% or 30% type of emulsion. The samples prepared included heavy crude oil that was collected from the AL-Dorah refinery. The preparation procedure of emulsion, which is used here, depends on the standard way that [13] is utilised in his work. Two types of laboratory samples of 40 vol.% of water to oil and 30 vol.% water to oil emulsion were prepared by using heavy crude oil and water. After adding the specified amounts of water to heavy crude oil in a glass beaker, each sample was mixed with a glass rod until the mixture was all perfectly mixed. Then, the samples were mixed by using a magnetic stirrer (78-1 Hot plate stirrer) for 10 minutes. Next, the samples were left for twenty-four hours to ensure the stability of the emulsions produced. Multiple samples were tested according to the CCRD optimisation technique, which was utilised in the experimental procedure in order to achieve the optimum condition of microwave heating.

ii. Leave the emulsions prepared for 24 hours to prepare stable emulsions.

iii. Multiple samples of 100 ml were conducted in the microwave reactor with different irradiation powers. The powers were subdivided into 200, 400, 600, 800, and 1,000 W through the control unit of the microwave reactor.

iv. The treatment time that was conducted on the samples was subdivided into intervals of 40, 80, 120, 160, and 200 seconds.

v. Each sample was dropped in a graduated cylinder to recover the water phase.

vi. Calculate the separation efficiency using the Karl-Fisher method for each sample and list it in a table.

vii. Analyse the data using professional software (STATISTICA and Design Expert).

viii. After the software is analysed, the general equation will be extracted and the optimal values for separation efficiency are extracted with the best time and power.

ix. Different values of calcium carbonate are used, as shown in Table 5, with different dosages in 40 vol.% and 30 vol.% to enhance separation efficiency in this case. Figure 7 shows the experimental procedure.

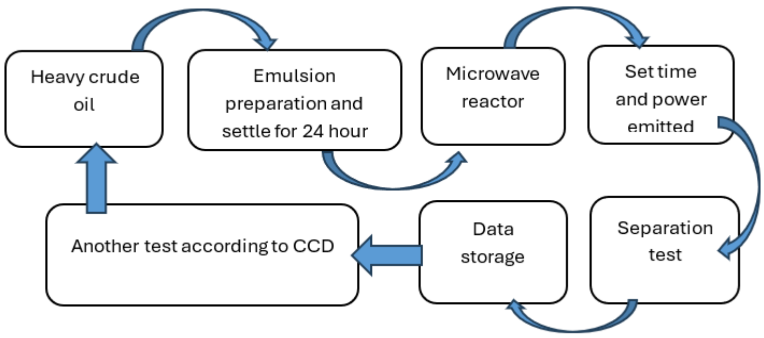


Fig. 7. Schematic diagram of microwave heating process for testing crude oil sample.

A series of studies have to be done in order to understand the influence of each factor. Experimental design is a methodological strategy that tends to cover the operation with the minimum number of experiments. The first part of the experimental design tends to perform a design to the experiments according to a known plan, and each variable in the plan is denoted with a coded factor, which is denoted as X1 for power emitting and X2 for irradiation time. The second Section needs regression analysis of a specified set of operations in the plan, including calculating the encoded model of factors and analysing the goal function for each experience in the group. With the use of CCRD central composite rotatable design, five points at -2, -1, 0, +1, and +2 were utilised in the process. Each level is identified in Table 3. It is the most effective design that has been used for representing the surface methodology to establish the second-order quadratic model for the variables avoiding the necessity for the usage of three-level factorial.

After achieving the optimum condition of the microwave, which was 800 W and 160 sec, the sample was dropped in a 200 ml glass container. An inorganic salt $CaCo_3$, which is calcium oxide and carbon dioxide, was added to the sample and mixed with a glass rod for different dosages of the salt of 0.5, 1.0, 1.5, 2, and 2.5 g at 800-Watt power emission and 160 sec irradiation time. After exposing it to microwave the samples were taken quickly and placed in a graduated cylinder in order to see the separation by sedimentation of the sample in the cylinder. Table 5 shows the experimental results of inorganic salts at different dosages of additions.

Table 5. Treatment by microwave heating and inorganic salt with Irradiation time (160 sec) and power (800 W).

Separation efficiency at 40% Vol. (%)	Separation Efficiency at 30% Vol. (%)	Concentration of addition (gram)
54	48	0.5
58	55	1
68	60	1.5
87.5	79.5	2
81	73	2.5

3. Results and Discussion

3.1. Effect of irradiation power on demulsification efficiency

Figures 8-11 present the results of separation efficiency. It can be seen that separation efficiency increases continuously with irradiation power increase until there is no further rise in the sample temperature. The interaction between water drops, which have dielectric properties with electromagnetic radiation of microwave, tends to absorb energy. The energy absorbed is proportional to the wavelength and penetration depth. Wavelength and penetration depth depends on the type of medium that it travels through (vacuum, air, water), so when the water amount increases, it increases the effect of wavelength and penetration depth. The wavelength is shorter to emulsions with lower water content, so the separation efficiency becomes greater at 40% vol. content of water.

In the case of increasing water amount, the separation will increase due to the effect of irradiation power and the effect of water amount increasing on the separation efficiency. At the same time, lower water content in an emulsified sample requires more energy to reach the set point temperature. Increasing the initial amount of water in the emulsified sample narrows the space between the droplets. This reduction in distance enhances the possibility of interaction between the droplets. This finding agrees with the funding of [24].

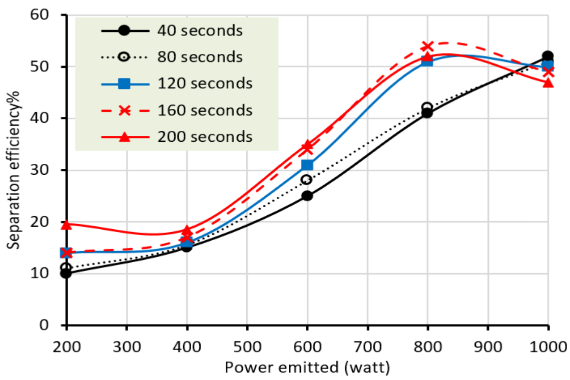


Fig. 8. Effect of irradiation power on separation efficiency at 40% vol. of water.

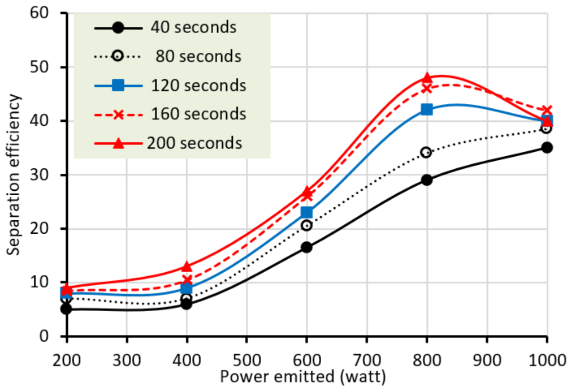


Fig. 9. Effect of irradiation power on separation efficiency with 30% vol. water.

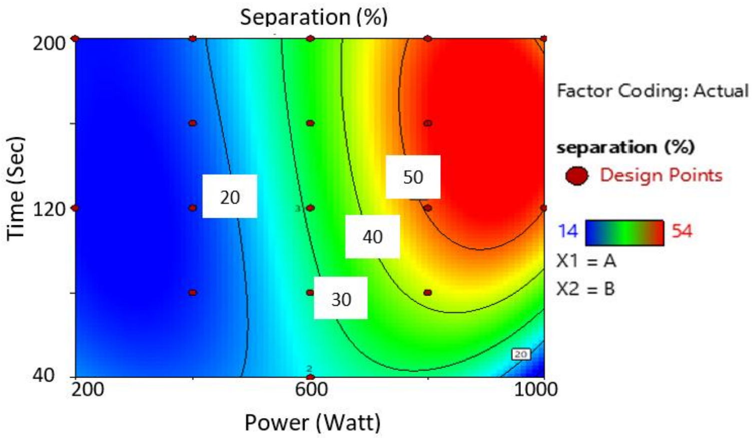


Fig. 10. 2D plane of power emitting (watt) and irradiation time (sec) for 40 vol.% water.

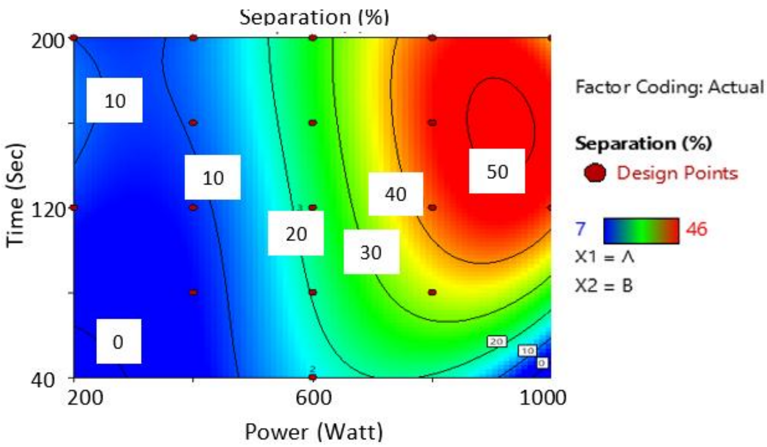


Fig. 11. 2D plane of power emitting (watt) and irradiation time (sec) for 30 vol.% water.

3.2. Effect of irradiation time on demulsification efficiency

Treatment time or irradiation time is an important factor that has an impact on the effectiveness of the electromagnetic heating wave application process on viscous fuel oil. The microwave exposure duration, shown in Figs. 12 and 13, is critical for emulsified separation. A shorter exposure period is inadequate for large numbers of small droplets to aggregate and settle. The greater the irradiation time, will cause water drops to aggregate and combine due to their electric properties, leading to the gathering of the water phase. The separation efficiency increases rapidly with time of irradiation for about 160 seconds before the boiling of the emulsion, which is 100 ^{o}C. Utilising 800 W of microwave irradiation for a 40% vol. W/O emulsion yields a separation effectiveness of 54% at this irradiation time. Moreover, under the same conditions, 30% vol. yields a separation of about 46%.

The difference in the separation efficiency between the two concentrations of water and oil is because the dielectric properties of water are greater than oil; water that is purified has a static dielectric relative permittivity equal to 80, which reduces long-range electrostatic forces between charges while the oil has dielectric constants ranging from 2.1 to 2.4, depending on the oil's viscosity, density, relative paraffinic, naphthenic, and aromatic content, and the oil's additive package. When the amount of water is higher, it causes water drops to aggregate and combine due to their electric properties, leading to the gathering of water phase [24].

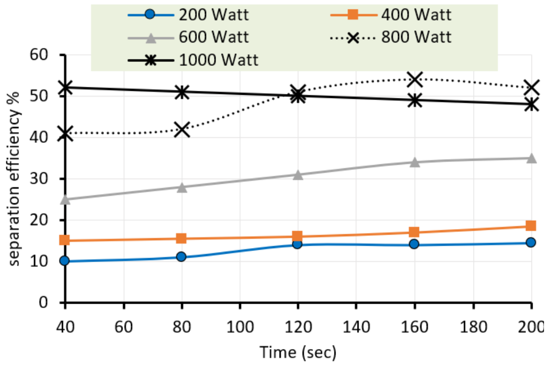


Fig. 12. Effect of irradiation time on separation efficiency with 40% vol. of water.

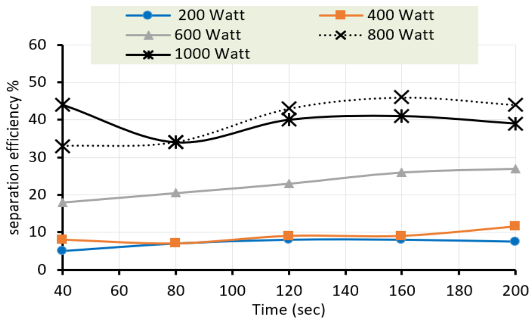


Fig. 13. Effect of irradiation time on separation efficiency at 30% vol. of water.

3.3. Inorganic salt and water concentrations effect on demulsification efficiency

The investigation of the effect of salt concentration on microwave demulsification might be useful for breakdown emulsions formed during the extraction of oil and desalting processes. The concentration of salts in the water phase influences dielectric heating. It is also widely known that the presence of charged particles in the medium increases the effectiveness of heating the mixture. Figure 14 demonstrates that water's separation efficiency rises with increasing salt concentration, perhaps due to the breakdown of double charge layers, which is an area lying at the border of two phases and thought to be composed of two different-charged layers (for example, a layer of negatively charged ions adsorbed on colloidal particles that attract a layer of positively charged ions in the surrounding electrolytic solution by $CaCo_3$. These findings agree with those of [8]. It is important to consider the medium's components to understand the relationship between dielectric properties and water content in an emulsion and the effectiveness of $CaCo_3$ in demulsifying oil samples with 30% and 40% water content. The water content of the emulsion can have an impact on coalescence efficiency during demulsification. Increasing the initial amount of water in the emulsified sample narrows the space between the droplets. This reduction in distance enhances the possibility of interaction between the droplets.

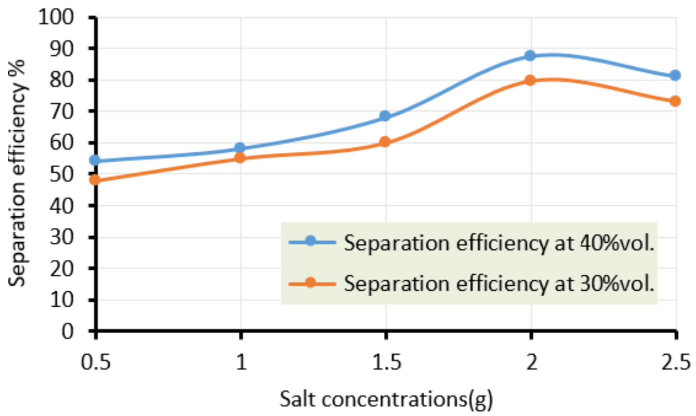


Fig. 14. Effect of inorganic salt additions on separation efficiency at 40% and 30vol.% of water.

Tests were conducted under similar conditions but with varying emulsion water contents. Results showed that higher starting water content improved emulsion resolution, with 40% of the water removed in test one (higher water content sample). The test with the highest starting amount of water had the greatest decrease in water. In test two, the proportion of water eliminated was around 87.5%. Only around 13% of the water remained after demulsification, while in lower water concentrations of 30%, the separation efficiency achieved was 79.5%, which is lower than the efficiency in the samples with greater water content. The structure of the emulsion affects the dielectric heat phenomenon, which is widely recognised in microwave radiation. $CaCo_3$ powder possesses a dielectric constant of 6.0-6.8, which decreases as the applied frequency increases. The crystal structure, crystallite size, lattice strain, and dielectric characteristics of $CaCo_3$ powder rely on the temperature. The higher amounts of temperature will decrease the viscosity of the sample and tend to the aggregate and gathering of water drops, which leads to settling them and reaching the separation between water and oil. Lower water content in an emulsified sample requires more energy to reach the set point temperature.

4. Conclusions

This research investigated the emulsion stabilisation and coalescence demulsification using microwave radiation as a novel green approach that is environmentally friendly and low economically cost compared to traditional methods used in breaking emulsions. The microwave heating method was effectively used to demulsify 40%-60% water to oil and 30%-70% water to oil emulsions.

The separation efficiency increases with irradiation time as the viscosity decreases, resulting in a faster film draining rate and drop coalescence. An emulsion sample is created with 40% and 30% vol. of water content. The samples are tested by a microwave reactor with different irradiation powers and different times to achieve the optimum condition, which is identified as 800 watts and 160 secs with separation efficiency of about 54% in 40% vol. and 46% vol.

After that, several tests are conducted using inorganic salts $CaCo_3$ and microwave heating. The results with inorganic salts achieved 87.5% in the sample of 40% vol. water and it 79.5% in the sample of 30% vol. water separation efficiency proving the effectiveness of microwave reactor heating and inorganic salts for the separation of very stable heavy crude oil emulsions in a remarkably brief amount of time which is about 30-40 seconds.

It is recommended to investigate different types of oil, like heavy and light crude with different water content. Also, investigating the use of different types of emulsifying agents, like natural substances, is important for the EOR topic.

Abbreviations

CCRD	Central composite rotatable design
O/W	Water in oil
RSM	Response surface methodology
SEC	Second
VOL	Volume
X1	Power Emitting
X2	Irradiation Time

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