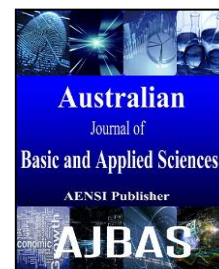




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Analysis of Microwave Heating Process for Demulsification of Water-in-Crude Oil Emulsions

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ABSTRACT

In this investigation, the process of microwave heating technology was evaluated to measure the effect of some important parameters such as dielectric properties (ϵ' and ϵ''), rate of temperature increase (dT/dt), volume rate of heat generation (Q_{mw}), wavelength (λ) and penetration depth (Dp) during the microwave irradiation on crude oil emulsions. Two types of Malaysian crude oil mixed together at a volume ratio of 50-50% and applied for further investigations. In order to ensure the efficiency of the process, the improvement of existing techniques and the development of new technology different ratios of water and oil were utilized to prepare the emulsions of water-in-crude oil (W/O). The emulsion samples were heated under 360 watt and 540 watt for 3 to 5 minutes. The findings of the microwave heating demulsification showed that higher microwave power (540 watt) along with the radiation time (5 min) were not much effective for water separation. This is because of the over boiling of the samples. Consequently, for microwave heating demulsification the best water separation efficiency was achieved at 3 (minutes), 360 (watt). Based on the result of microwave parameter's calculations, it was found that parameters such as; dT/dt , Q_{mw} , ϵ' and ϵ'' , were inversely proportional to the radiation time. However, parameters such as λ and Dp were directly proportional to the time of the radiation.

INTRODUCTION

In petroleum industries the most common demulsification methods are chemical and conventional heating. The chemical method is the addition of a demulsifier in emulsion and a conventional method is the application of hotplate and water bath to heat the emulsion. However, these methods are costly, time consuming and in some cases they are not able to break the emulsion properly (Issaka *et al.*, 2014).

Microwave irradiation technology has been used for long time due to its high efficiency of heat transfer. Historically, the concept of microwave heating was first introduced by Klaila and Wolf in their patent applications (Klaila, 1978; Wolf, 1986). Klaila conducted several field tests after his patent was authorized, and the results were encouraging. Later, it was followed by Wolf, as it was claimed that compared to conventional thermal heating; microwave irradiation provides a much higher efficiency in the breaking of water-oil emulsions, where the emulsions can be separated more rapidly when exposed to the electromagnetic radiation (Wolf, 1986).

The process of heating in microwave is generally different from the heating process used in conventional ovens. In conventional heating, the energy is transferred to the material by the mechanism of convection,

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conduction, and radiation through the external surface of material in the presence of thermal gradients and it takes time to heat all points of the sample (Abdurahman *et al.*, 2010; Saifuddin and Refal, 2014). However, microwave heating offers a volumetric heating affects which results in a rapid processing time and efficient separation (Nour *et al.*, 2012). Generally, the behaviors of materials are different when they come to the exposure of microwave irradiation and these materials are categorized into insulators, conductors, and absorbers according to their interaction with the electric field of microwave, as illustrated in Fig. 1.

- Insulators are the materials where microwave can pass through them without any losses
- Conductors are the materials where they reflect the microwaves and cannot penetrate
- Absorbers are materials that absorb the microwave radiation and are called dielectrics

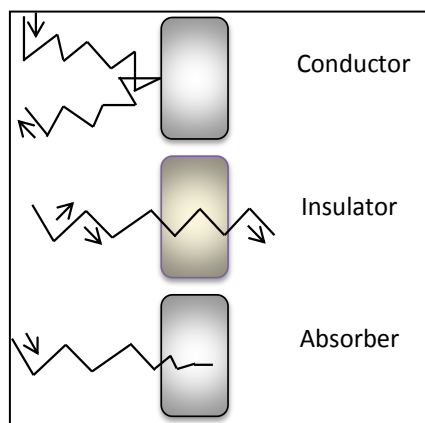


Fig. 1: Category of materials in microwave absorption.

Therefore, microwave heating is also referred to as dielectric heating. In microwave range, the interaction of dielectric materials with electromagnetic radiation results in energy absorbance in the material. Compared to the conventional heating, microwave heating process may lead to significant savings in energy consumption, increases the process yield; reduces the process time and environmental issues (Jones *et al.*, 2002). Therefore, this study is aimed to investigate the heating process for demulsification of water-in-crude oil emulsions via the calculation of microwave parameters such as; rate of temperature increase (dT/dt), volume rate of heat generation (Q_{mw}), dielectric constant (ϵ'), dielectric loss (ϵ''), wavelength (λ) and penetration depth (Dp) in samples.

MATERIALS AND METHODS

In this study, two types of Malaysian crude oils namely; heavy and light were mixed together at a volume ratio of 50-50 %. The physical and chemical properties of crude oil is shown in Table 1. For the purpose of emulsification, Span 80 with HLB of 4.3 and at concentrations of (2.5 vol. %) was selected to prepare the emulsions of W/O at a volume ratios of (20-80) and (40-60) vol.%. After the emulsification process, the demulsification of emulsions was performed using microwave-assisted chemical demulsification using a lipophilic demulsifier (Octylamine) from amine groups with an HLB of 6.88.

Table 1: Physical and Chemical properties of crude oil

Physical	Measurement	Chemical	Measurement
Density (g/cm^3)	0.8886	Saturates	65.2
Pour point ($^{\circ}C$)	-12	Aromatics	25.1
Viscosity (N/m^2) at 30 $^{\circ}C$	35	Resins	4.2
Water content (%)	0.4	Asphaltenes	5.5
API Gravity	28	Resin/Asphaltene ratio (R/A)	0.76
Surface Tension (mN/m) at 25 $^{\circ}C$	26.566	-	-
Interfacial Tension (mN/m) at 25 $^{\circ}C$	15.831	-	-

2.1. Emulsion Preparation:

The water-oil emulsions were prepared at volumes of (20-80) and (40-60) vol.%. The emulsions were prepared with the dissolving of Span 80 into the crude oil and vigorously sheared for 5 minutes. Following the dispersed phase (water was slowly added to the continuous phase (crude oil) while they are mixing in a standard three blade propeller and sheared for another 5 minutes at mixing speed of 2000 rpm at room temperature. Then, the emulsion type was identified by using the test tube method, whether is the W/O or O/W type. Afterwards, in

demulsification process the demulsifier (Octylamine) was added at the same concentration and agitated for 5 minutes more. Each emulsion was prepared at a volume of 300 mL.

2.2. Demulsification of the Emulsion:

A domestic microwave oven model EMO-2305 (900 output power, 23 L capacity, Elba, Malaysia) was used for the demulsification process. The freshly prepared W/O emulsion with a volume of 300 mL was placed in a 500 mL glass beaker. The top and bottom of the beaker were covered with aluminum foil and placed in the center of the microwave oven. To record the temperature during the microwave heating at three points, three thermocouples, model J-IEC-584-3, were inserted in the emulsion sample at three different locations; top, middle, and bottom. The thermocouples were connected to Pico- TC 08, data logger device. The data logger was connected to a PC with Pico Log software to record the temperature (The microwave heating was performed using different watts (360-540) W and different microwave processing time (3-5) minutes, with operation frequency at 2450 MHz.

2.3. Calculation of Microwave Parameters:

Microwaves are a form of electromagnetic energy, the most significant characteristic of microwave heating is its volumetric heating, which makes it quite different from conventional heating. Volumetric heating means that the microwave energy can be absorbed directly and internally by materials and then converts into heat. In conventional heating, the energy is transferred to the material via convection, conduction, and radiation phenomena through the external surface of the material. However, in microwave, energy is delivered to the material directly through the molecular interactions with the electromagnetic field via conversions of electromagnetic energy into thermal energy or with the electric field of the incident radiation (Venkatesh and Raghavan, 2004). Since, the microwave has the volumetric heating property; hence the volume rate of heat generation in a microwave can be calculated using Eq. (1).

$$Q_{mw} = \underbrace{\frac{hA}{V}(T_m - T_a)}_{\text{Convection}} + \underbrace{\frac{\varepsilon A \sigma}{V}[(T_m + 273.15)^4 - (T_a + 273.15)^4]}_{\text{Conduction}} + \underbrace{\rho C_p \left(\frac{dT}{dt}\right)}_{\text{Radiation}} \quad (1)$$

In microwave heating the contribution of heat transfer for convection and conduction is not significant, thus they can be ignored. In Eq. (2), the rate of heat transfer for convection and conduction is assumed zero, due to the rapid transformation of heat from emulsified water droplets to the continuous phase (oil). Accordingly, the temperature of water and oil is almost at the same rate (Fang and Lai, 1995; Anisa, 2011). Therefore, the volume rate of heat generation of emulsion can be simplified as below:

$$Q_{mix} = \rho_{mix} C_{p,mix} \left(\frac{dT}{dt}\right) \quad (2)$$

Where, Q_{mix} (cal/s.cm³) is the volumetric rate of heat generation of the mixture (emulsion), ρ_{mix} and $C_{p,mix}$ are the density and heat capacity of materials in (g/cm³) and (cal/g.⁰C), respectively, dT/dt is the rate of temperature increase (⁰C/sec). For calculation of volume rate of heat generation in Eq. (2), the density (ρ_{mix}) and heat capacity ($C_{p,mix}$) of the emulsions calculated from mixing rules as described in the following equations: Eqs (3) to (5).

$$\rho_{mix} = \rho_w \phi + \rho_o (1 - \phi) \quad (3)$$

$$C_{p,mix} = C_{p,w} \phi + C_{p,o} (1 - \phi) \quad (4)$$

$$\phi = \frac{\frac{\rho_w}{\phi_w}}{\frac{\rho_w}{\phi_w} + \frac{\rho_o}{\phi_o}} \quad (5)$$

In microwave system, energy transfers directly to the materials through molecular interactions with the electromagnetic field via conversions of electromagnetic energy into thermal energy as a result of dielectric loss that caused by rotation of polarized molecules (Venkatesh and Raghavan, 2004; Routray and Orsat, 2012).

Where ϵ' , is the dielectric constant that reflects the ability of the material to store electric energy when in an electromagnetic field; ϵ'' , is the dielectric loss factor that influences the conversion of the electromagnetic energy into thermal energy (heat). The ratio of dielectric constant and dielectric loss factor represent the tangent loss factor, $\tan \delta$, also called the dissipation factor or the dielectric loss tangent as expressed by Eq. (6).

$$\tan \delta = \frac{\epsilon''}{\epsilon'} \quad (6)$$

The dielectric constant is a measure of a material's ability to store the microwave energy as it passes through, while the dielectric loss factor measures the material's ability to dissipate that energy.

The dielectric constant and dielectric loss of water were given by Wolf (1986) as shown in Eqs (7) and (8).

$$\epsilon'_{rw} = 85.2 - 0.3358T \quad (7)$$

$$\epsilon''_{rw} = 320.66T^{-1.03} \quad (8)$$

Hippel (1954) proposed equations for dielectric properties of various petroleum oils. Accordingly, dielectric constant and loss tangent of crude oil represented in Eqs (9) and (10).

$$\epsilon'_{ro} = 2.24 - 0.000727T \quad (9)$$

$$\tan \delta_o = (0.527T + 4.82) \times 10^{-4} \quad (10)$$

Since the frequency for most types of the microwave apparatus is set at 2.24 GHz, the wavelength of the electromagnetic fields in the liquids as a function of temperature expressed by Eq. (11).

$$\lambda = \frac{\lambda_o}{\sqrt{\epsilon'}} \quad (11)$$

Where, λ is the wavelength, $\lambda_o = 12.2$ cm is the wavelength in free space (Patil, 2012).

The penetration depth (D_p) is also one of the important parameters in microwave heating. This parameter is defined as the depth into a sample where the microwave power has dropped to 36.8% of its transmitted value. The D_p within a sample for a radiation is related to dielectric constant and dielectric loss as expressed in Eq. (12).

$$D_p = \frac{C}{2\pi f \sqrt{2\epsilon'} \left[\sqrt{1 + \tan^2 \delta} - 1 \right]^{1/2}} \quad (12)$$

Where, C is the speed of light (cm/s), and f is the applied frequency (Hz). However, when $\tan \delta < 1$, the D_p is as Eq. (13), which is usually the case (Veggi *et al.*, 2013).

$$D_p = \frac{\lambda_o \sqrt{\epsilon'}}{2\pi\epsilon''} \quad (13)$$

RESULTS AND DISCUSSION

3.1. Demulsification of the Emulsions:

The result of demulsification for 20-80 and 40-60 vol.% water oil is shown in Fig. 2. As it can be observed that microwave radiation can enhance the demulsification rate by means of its dielectric heating, therefore, the separation efficiency reached to 100 % in second day of the observation in emulsion with 40 % water. It is also clear that water content were also effective in the demulsification rate. Meaning that emulsions with higher water content achieved a better separation efficiency. Where, this can be attributed to the nature of water, because water can absorb more energy to heat compared to the oil phase (Xia *et al.*, 2006; Tan *et al.*, 2007; Anisa and Nour, 2009).

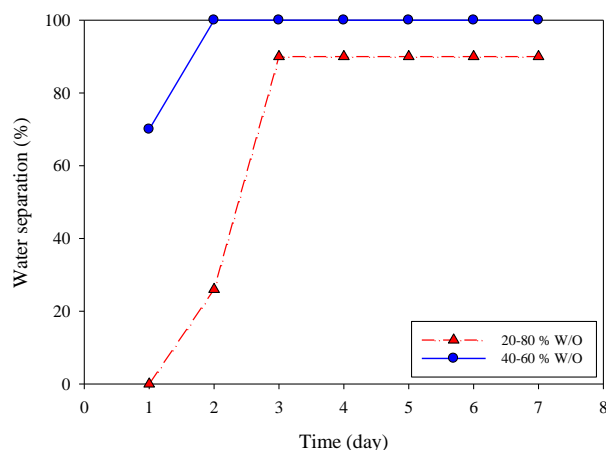


Fig. 2: Microwave demulsification in different water oil ratio.

Additionally, if the concentration of water is high, the mechanisms of coalescence and sedimentation can easily occur, due to the gravity force. The specific gravity of water is higher than oil so the water droplets on the top of the emulsion are inclined to migrate toward the bottom and finally aggregate to a separate layer (Xiaogang *et al.*, 2009). Through an optimization process, it was found that the best water separation efficiency was achieved at 3 (minutes), 360 (watt), and 2.50 vol. % surfactant. Whereas, for emulsion with 40 vol. % water content the separation efficiency reached to 100 %. It was also found that higher microwave power (540 watt) along with the radiation time (5 min) were not much effective for water separation. This is because of the over boiling of the samples during the heating of the sample in microwave.

3.2. Calculation of Microwave Heating Parameters:

Effect of Radiation Time on Temperature:

The study found that there are many factors that can influence on the temperature of the microwave radiated emulsions. Fig. 3 shows the influence of radiation time (300 s) on the emulsions at different water contents. It can be seen that the highest temperature rate was obtained in emulsions with a higher water phase ratio (40-60) %. However, 20-80 % W/O emulsions performed the lowest temperature, whilst, both emulsions were radiated for 300 s. It can be attributed to the content of the dispersed phase. Because, the dielectric properties of water are higher than oil, therefore, water can absorb more energy than oil, hence higher temperature can be achieved (Xia *et al.*, 2006; Tan *et al.*, 2007; Anisa and Nour, 2009). Thus, it can be claimed that the temperature distribution was significantly increased with the water content and radiation time.

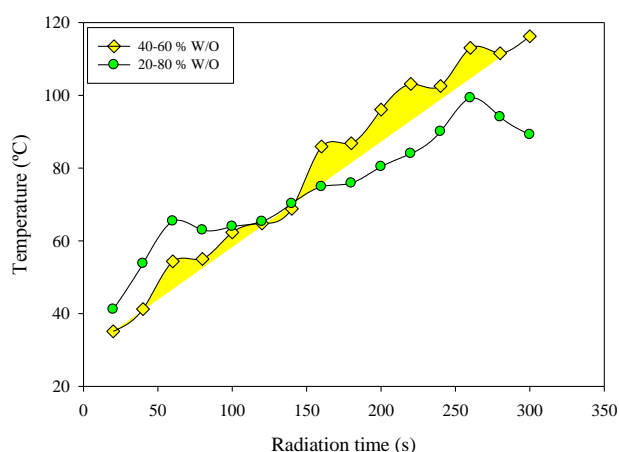


Fig. 3: Effect of radiation time on temperature in microwave heating at 540 watt.

The maximum temperatures achieved for emulsions at water contents of (40 and 20) vol. %, were (116 and 89) °C, respectively, when the microwave power was settled at 540 watt. Moreover, as the microwave power decreased the values of the temperatures declined as well. Because, wavelength and penetration depth declines with the power reduction. The calculation of microwave parameters for 20-80 % and 40-60 % W/O emulsions irradiated under 540 watt for 5 minutes are shown in Table 2.

Effect of Radiation Time on the Rate of Temperature Increase:

The rate of temperature increase was also found to be as a function of the radiation time and microwave power (Fig. 4). It can be observed that reduction of microwave power was resulted to reduce the rate of temperature increase. Clearly, at 540 watt and 360 watt, the rate of temperature increase were at 0.66 °C/s and 0.32 °C/s, respectively when radiated for 20 seconds. However, by increasing the radiation time to 300 s, these values declined to 0.2 °C/s and 0.14 °C/s, respectively. The increasing of heating rates with the microwave power is caused by increasing the absorption of microwave energy.

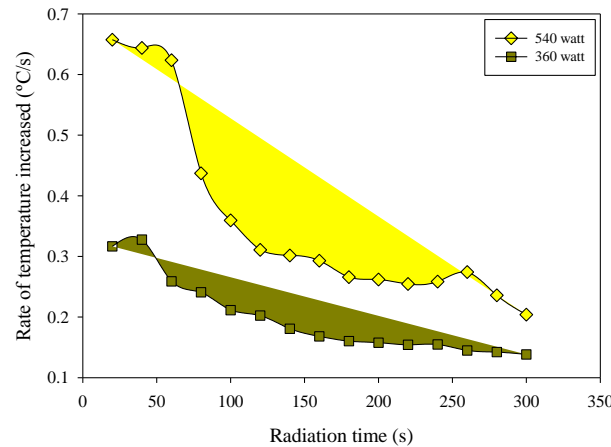


Fig. 4: Effect of radiation time on rate of temperature increase at 20-80 % W/O emulsion.

Effect of Radiation time on the Volume Rate of Heat Generation:

The volume rate of heat generation is also as a function of radiation time. Fig. 5 shows the effect of radiation time on volume rate of heat generation of emulsions at various water contents. It was found that the volume rate of heat generation of the samples decreased with increasing the radiation time from (20 to 300 min) this is also due to the temperature increase during the radiation time. Relatively, dilute emulsions achieved higher rate of heat generation, since water has high polarity and dielectric properties (Issaka *et al.*, 2014).

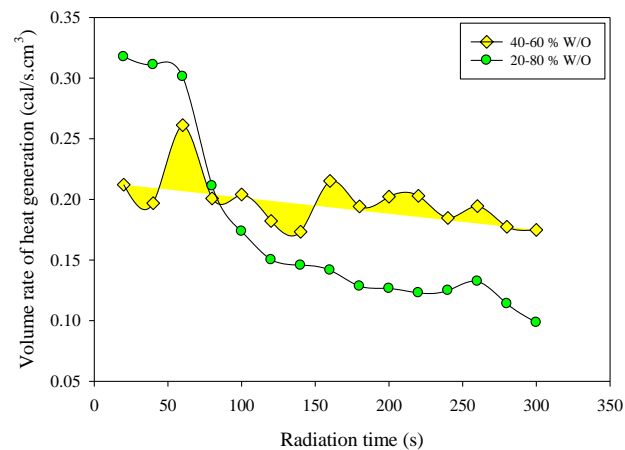


Fig. 5: Effect of radiation time on volume rate of heat generation of two different emulsions.

Effect of Radiation Time on the Dielectric Properties of Emulsion:

The dielectric properties of the emulsions are generally dependent on the factors such as; volume fraction of the dispersed phase, irradiation time, frequency, and temperature. The dielectric properties are also responsible for converting the microwave energy to heat (Eskilsson and Bjorklund, 2000; Erdogan, 2011).

Figs. 6 and 7 show the dielectric constant and dielectric loss of the emulsion at different water contents, respectively. As it can be seen that water content has strongly influenced both dielectric constant and dielectric loss of the emulsions. Meaning that higher water volume fraction (40 vol. %), obtained higher dielectric constant and dielectric loss values within the radiation time. This can be supported by the nature of water, since water has a high polarity and dielectric properties (Mohammed and Mohammed, 2013; Xia *et al.*, 2010).

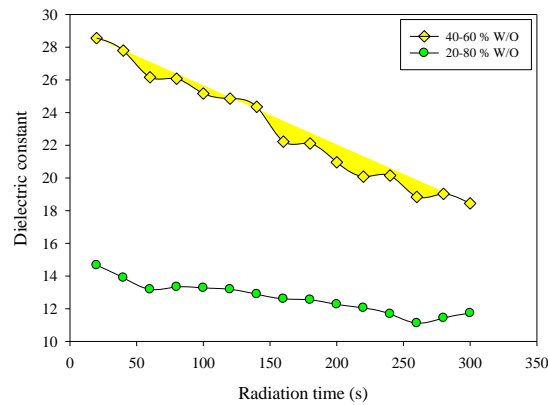


Fig. 6: Effect of radiation time on dielectric constant of the emulsions.

The interaction of dielectric materials with electromagnetic radiation in the range of the microwave leads to the absorption of energy. As well as, higher water content means more droplets inside the emulsion, where this leads to an increase in ionic mobility and hence dielectric properties. This result is in a good agreement with that found by (Anisa and Nour, 2009). As the frequency of the microwave was constant at 2.45 GHz, so only the temperature could affect the dielectric properties, while the temperature of the microwave radiation is also a function of the irradiation time.

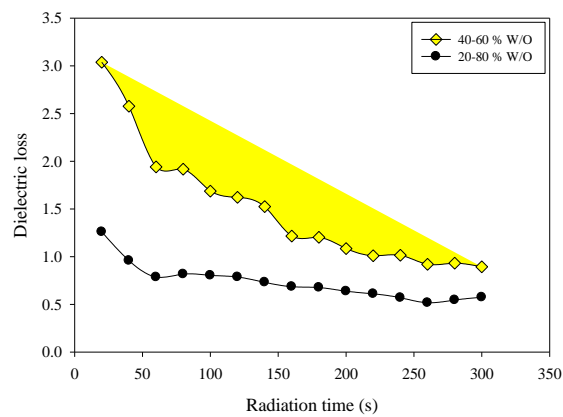


Fig. 7: Effect of radiation time on dielectric loss of the emulsions.

As stated by Issaka (2014), that the energy generation in a microwave is proportional to the wavelength. Fig.8 displays the influence of radiation time on the wavelength of emulsions with different water contents. It can be clearly observed that the wavelengths of emulsions with lower water content 20-80 %, were greater than that of with 40-60 % W/O.

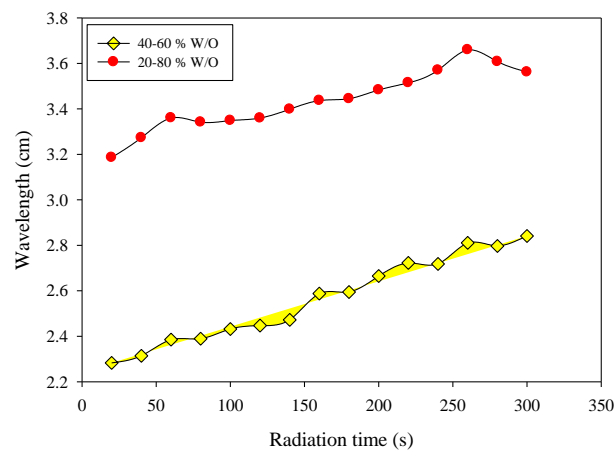


Fig. 8: Effect of radiation time on wavelength.

In fact, if the wavelength is short, the efficiency of heating would be better and faster. Therefore, the dilute emulsion (40-60 % W/O), achieved the best separation efficiency of 100 %. It is also found that the wavelength would increase when the radiation time and temperature increases. In fact, the temperature in microwave heating is proportional to the radiation time (Tan *et al.*, 2007).

Fig. 9 shows the effect of radiation time on the penetration depth of emulsions with two different water to oil ratios. The penetration depth is defined as the distance where the electromagnetic waves travel and decreases its power to 1/e or 36.8 % (Wu *et al.*, 2002).

As it can be seen that the behavior of penetration depth within the radiation time was similar with the wavelength. Meaning that increasing the water content resulted in a significant reduction of the penetration depth. Since the penetration depth and wavelength are strongly related to the dielectric properties. Therefore, by increasing the dielectric properties the penetration depth decreases.

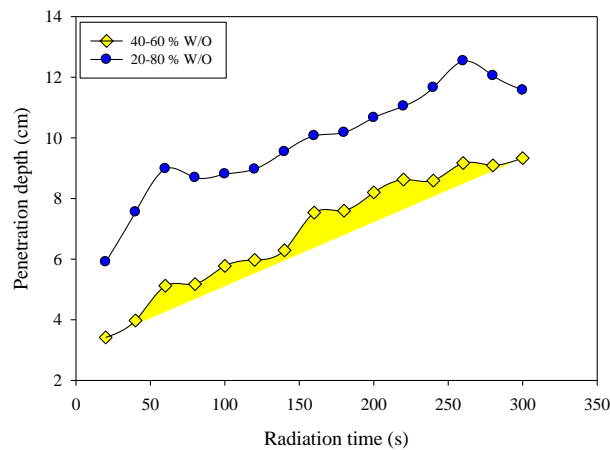


Fig. 9: Effect of radiation time on penetration depth.

Table 2: Calculation of microwave parameters

Time (s)	To = 28 (°C)	dT/dt, °C/s	Qmw (cal/s.cm ³)	ε (water)	ε'' (water)	ε' (mixture)	ε' (oil)	ε'' (oil)	ε'' (mixture)	tan δ (oil)	Wavelength (cm)	Penetration depth (cm)
20-80% W/O												
20	41.15	0.657	0.318	71.382	6.970	14.661	2.210	0.0057	1.2594	0.0026	3.1862	5.9066
40	53.76	0.644	0.311	67.149	5.293	13.892	2.201	0.0072	0.9586	0.0033	3.2733	7.5530
60	65.42	0.624	0.301	63.231	4.324	13.179	2.192	0.0085	0.7852	0.0039	3.3606	8.9817
80	62.96	0.437	0.211	64.057	4.498	13.329	2.194	0.0082	0.8163	0.0037	3.3416	8.6886
100	63.95	0.359	0.174	63.726	4.426	13.269	2.194	0.0083	0.8036	0.0038	3.3492	8.8067
120	65.29	0.311	0.150	63.274	4.332	13.187	2.193	0.0085	0.7868	0.0039	3.3596	8.9664
140	70.21	0.301	0.146	61.624	4.020	12.887	2.189	0.0090	0.7311	0.0041	3.3984	9.5394
160	74.88	0.293	0.142	60.054	3.762	12.602	2.186	0.0096	0.6850	0.0044	3.4367	10.0676
180	75.84	0.266	0.128	59.732	3.713	12.543	2.185	0.0097	0.6763	0.0044	3.4447	10.1739
200	80.39	0.262	0.127	58.207	3.497	12.266	2.182	0.0102	0.6378	0.0047	3.4834	10.6671
220	83.99	0.255	0.123	56.996	3.343	12.046	2.179	0.0106	0.6103	0.0049	3.5151	11.0470
240	90.03	0.258	0.125	54.968	3.112	11.677	2.175	0.0112	0.5694	0.0052	3.5702	11.6595
260	99.29	0.274	0.132	51.860	2.814	11.112	2.168	0.0123	0.5165	0.0057	3.6598	12.5382
280	94.06	0.236	0.114	53.616	2.975	11.432	2.172	0.0117	0.5451	0.0054	3.6083	12.0507
300	89.18	0.204	0.099	55.254	3.142	11.729	2.175	0.0112	0.5748	0.0051	3.5622	11.5751
40-60% W/O												
20	35.14	0.357	0.212	73.401	8.202	28.554	2.214	0.0050	3.0379	0.0023	2.2831	3.4171
40	41.25	0.331	0.197	71.349	6.953	27.791	2.210	0.0057	2.5763	0.0026	2.3142	3.9752
60	54.36	0.439	0.261	66.944	5.232	26.156	2.200	0.0072	1.9404	0.0033	2.3855	5.1202
80	55.03	0.338	0.201	66.722	5.167	26.073	2.200	0.0073	1.9164	0.0033	2.3893	5.1761
100	62.33	0.343	0.204	64.270	4.545	25.163	2.195	0.0081	1.6867	0.0037	2.4321	5.7774
120	64.78	0.307	0.182	63.446	4.368	24.857	2.193	0.0084	1.6213	0.0038	2.4470	5.9738
140	68.81	0.292	0.173	62.093	4.104	24.354	2.190	0.0089	1.5243	0.0041	2.4721	6.2897
160	85.92	0.362	0.215	56.349	3.265	22.221	2.178	0.0108	1.2150	0.0050	2.5881	7.5370
180	86.82	0.327	0.194	56.046	3.231	22.109	2.177	0.0109	1.2021	0.0050	2.5947	7.5984
200	96.06	0.340	0.202	52.942	2.911	20.956	2.170	0.0119	1.0845	0.0055	2.6651	8.2001
220	103.12	0.341	0.203	50.574	2.706	20.076	2.165	0.0127	1.0092	0.0059	2.7228	8.6252
240	102.56	0.311	0.185	50.759	2.721	20.145	2.165	0.0126	1.0147	0.0058	2.7182	8.5930
260	113.05	0.327	0.194	47.238	2.461	18.837	2.158	0.0138	0.9194	0.0064	2.8109	9.1709
280	111.56	0.298	0.177	47.738	2.495	19.023	2.159	0.0136	0.9318	0.0063	2.7972	9.0931
300	116.23	0.294	0.175	46.171	2.392	18.441	2.156	0.0141	0.8940	0.0066	2.8410	9.3319

Conclusions:

The result shows that microwave radiation can increase the demulsification rate due to its dielectric heating properties, and can be used to separate the emulsions of water-in-crude oil. This technique can heat the samples in a short time which can result to energy saving. This study also conducted some calculation of microwave parameters the result of calculation exhibited that rate of temperature increase, volume rate of heat generation, and dielectric properties (dielectric constant and dielectric loss) were decreased by increasing the radiation time. Inversely, penetration depth and wavelength increased by enhancing the radiation time due to the high efficiency of microwave absorption.

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