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# EMULSION SEPARATION RATE ENHANCEMENT VIA MICROWAVE HEATING TECHNOLOGY

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## ABSTRACT

The role of microwave concept in demulsifying water-in-crude oil emulsion was assessed experimentally for heating rate, volume rate of heat generation and dielectric properties. In this study, a microwave demulsification method was utilised for emulsions; 20-80 % and 50-50 % w/o emulsion, water and crude oil with varied microwave exposure time. The prepared emulsion was vigorously shaken with three blade propeller at 1500 rpm in 5 minutes. Results have shown for both phase ratios, the rate of temperature increased as decreasing the dielectric properties and volume heat of generation. Higher water content in the emulsion absorbed more microwave radiation thus, friction of molecules are more rapid and converted into heat and performed the higher dielectric properties.

***Keywords***: microwave, w/o emulsion, volume of heat generation, dielectric properties, heating rate

## INTRODUCTION

Historically and economically, the most important problems in the refinery and oil industry have been in the area of breaking the emulsion particularly the stable emulsion formed within the reservoirs. Microwave is one of an alternative method for breaking the water droplets in emulsions and attracts attention from researchers since 1970s (Klaika, 1976; Wolf, 1986). As pointed out from Cha-um et al. (2008), microwave method disrupts the emulsion allows for more rapid separation of the oil and water under electromagnetic wave and that penetrates the surface is converted into thermal energy within the material in a manner different from the traditional methods of thermal heating.

Microwave method offers a clean, non-pollution, high product quality and convenient heating process that in most of times results into better yields and excellent opportunities for high energy utilisation (Hui, 2006; Coutinho et al., 2008, Cha-um et al., 2008). Indeed, conventional methods such as chemical demulsifier and thermal heating present an economic burden and additional contaminated water disposal problems. Under microwave radiation influence, microwaves do not heat the crude oil directly as the oil is essentially transparent at microwave frequencies as mention before range from 300 MHz to 300 GHz. Instead, within the molecules of emulsion, which also contain the water, the emulsion is heated and converted into steam depends on the temperature. The efficiency of the microwave radiation on the emulsion separation has been attributed to the following effects:

(i) reduction of the viscosity of the continuous phase (oil) because of the increase of the temperature, which favors the water droplets contact, (Schramm, 2005; Hannisdal, 2005)
(ii) reduction of the stability as a result of microwave-induced rotation of water molecules, which neutralizes the zeta potential of the dispersed droplets, besides breaking hydrogen bonds between water and surfactant molecules,
(iii) reduction of the thickness of the interfacial surfactant film because of the expansion of the dispersed phase caused by the increase of the internal pressure of the water droplets during microwave irradiation, and
(iv) reduction of the stability as a result of breaking hydrogen bonds between the surfactant molecules and the water molecules ( Hui, 2006; Lidstrőm et al., 2001).

Generation of heating within materials by microwave energy is primarily caused by molecular friction associated with water molecules and ionic migration of free salts in an electric field of rapidly change polarity. As studied by Lidstrőm et al., (2001), the microwave energy is lost to the sample by two mechanisms which, dipole rotation and ionic conduction**.**

**Dipole Rotation**

Dipole rotation is the interactions of the electric field component with the medium and turned into heat resulting from the friction with the neighboring molecules. Heat generated from the microwave energy posses in dipole moment only. The dipole is sensitive to electric field and under this field; it will attempt to align itself (Lidstrőm et al., 2001; Countinho et al., 2008). In liquids state, the electric dipoles cannot rotate instantaneously and the ability of molecules to align under influence of electric field will vary with different frequencies and viscosities of liquid. For low radiation frequencies, the time where the electric field changes direction is higher than the dipole response time. Molecules gain some energies but overall heating effect with full alignment is small. However, at very high frequency electric field, dipoles do not have sufficient time to follow the oscillating field and molecules do not rotate and move. When there is no motion, so there is no energy takes place and there also no heating occurs**.**

**Ionic Conduction**

A solution containing ions will move through the solution under the influence of an electric field, resulting in expenditure of energy due to an increased collision rate, therefore converting the kinetic energy to heat (Lidstrőm et al., 2001). The resulting electrical currents heat the sample as a consequence of the electrical resistance. From physical point of view, microwave heating is a combination of at least four different processes which, distribution of power, absorption of power, heat transfer and mass transfer. Thus, through this study, microwave is being used as tool to demulsified water in crude oil. This study was examined an experimental study of demulsification method using a batch microwave process to investigated the dielectric properties and volume heat of generation by at varied phase volume ratio by radiation time.

## EXPERIMENTAL SET UP

The demulsification test applying microwave radiation was conducted using a domestic, Elba microwave oven model; EMO 808SS, maximum rate power consumption is 800 W with operation frequency at 2450 MHz. The microwave was modified to inserted thermocouples inside the microwave before connected to Pico-TC-08 data loggers. The data logger was connected to PC with Pico Log software.

### Materials

Heavy crude oil (API= 17.54) was used in this study and supplied by Petronas Refinery Melaka. The characteristics of crude oil have shown in Table 1.

Table 1: Crude Oil Characteristics

| Characteristic | Crude oil |
|---|---|
| Viscosity (cP) | 125.16 |
| Density (g/mL) | 0.9494 |
| API density | 17.54 |
| Pour point ($^oC$) | -20.4 |

Sorbitan sesquioleate (Span 83) reagent purchased from Sigma Aldrich was used as an emulsifying agent for emulsion preparation. The emulsifying agent was used as received without further dilution. In the present study, the surfactant is dissolved in the aqueous phase and used a fixed concentration of 0.1% (w/w).

### Methods

*Emulsion Preparation*

In this study, two types of phase ratios were prepared which 20-80 w/o % and 50-50 w/o % from heavy and light crude oil. The emulsion samples that have been prepared through dissolved Span 83 as an emulsifying agent in the continuous phase (crude oil). Span 83 was added at 0.1 wt % of continuous phase without further dilution. Then, water added gradually to the mixture (oil + emulsifying agents) at prescribed mixing speed and processing time. Emulsions were agitated vigorously using a standard three blade propeller at constant speed of 1500 rpm for 5 minutes to facilitate the contact among the water droplets in the emulsion. The prepared emulsions were checked for o/w or w/o emulsions. Only w/o types was chosen for further study.

*Microwave radiation*

Microwave radiation is a means of rapidly heating in a manner different from traditional methods of thermal heating. Generally, most demulsification methods in industry have been heated and treated using conventional methods. These heating techniques are, however rather slow with non-uniform heating within the emulsion. In contrast, the microwave radiation heating is commenced into breaking the emulsion in direct heating is obtained through penetration. Microwaves interact with polar water molecules and charged ions. The friction resulting from molecules aligning in rapidly alternating electromagnetic field generates the heat within food. Since the heat is produced directly in the emulsion, the thermal processing time is sharply reduced. Ideally, when emulsion is heated under influence of electromagnetic, friction between molecules of emulsion will generated heat and increases temperature. The increasing temperature causes reduction of viscosity and coalescence is promoted. Therefore, the droplets (water) can be separated from crude oil.

One of the advantages using microwave is volumetric heating which, heat emulsions by passing through a body. For microwave heating, the governing energy equation includes volumetric heat generation which, results from temperature rise in material:-

$$\frac{dT}{dt} = \alpha V^2 T + \frac{Q_{abs}}{\rho C_P} \tag{1}$$

$Q_{abs}$ (cal/ sec.cm$^3$) corresponds to volumetric rate of internal energy generation due to dissipation of microwave energy. Essentially, the rate of increasing temperature (dT) is measured. $C_P$ (cal/g$^o$ C) is heat capacity of the material and ρ (g/cm$^3$) is the density of the material. As an assumption, there is no temperature gradients in a small mass of dielectric medium and the equation (2) can be simplifying as:-

$$Q_{abs} = \rho C_p \frac{dT}{dt} \tag{2}$$

In this study, material that refers from equation was emulsion. For calculation of density (ρ) and heat capacity ($C_P$) of emulsions calculated from mixing rule as:

$$\rho_m = \rho_w \phi + \rho_o (1 - \phi) \tag{3}$$

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

The dielectric properties largely determine of microwave mechanisms. As mention above, the mechanisms of microwave involved polar and dipole molecules which, the ability of emulsions to generate heat from microwave through absorption the microwave energy and converted into heat must be taken into account. The effectiveness to store electrical potential energy and converted into heat is called as dielectric properties. These factors may be considered as tangent loss, tan δ, which normally expressed as equation below:-

$$tan\,\delta = \varepsilon''/\varepsilon' \tag{5}$$

The dielectric constant $\varepsilon'$ represents the ability of dielectric material to store electrical potential energy under influence of an electric field. Quantities the efficiency with which the absorbed energy is converted into heat refers to dielectric loss,$\varepsilon''$ respectively. Dielectric constant and loss of water as cited from Abdurahman (2006);

$$\varepsilon' r = 85.215 - 0.33583T \tag{6}$$

$$\varepsilon'' r = 320.658T^{-1.0268} \tag{7}$$

While for various petroleum oils, as proposed by Abdurahman (2006) cited from Von Hippel (1954), dielectric constants and loss tangent of crude oil for this study calculated as:-

$$\varepsilon' r_o = 2.24 - 0.000727T \quad (8)$$

$$\tan \delta_o = (0.527T + 4.82) x\ 10^{-4} \quad (9)$$

Detail of the result has been shown in part 3, respectively.

## RESULTS AND DISCUSSION

During this study, there are several assumptions have been made included the absorption of microwave by air is negligible; the walls of containers are perfect conductors and the effect of sample container on the electromagnetic can be neglected. The samples are investigated and varied with water, crude oil and emulsion. In microwave radiation, the heating rate is calculated by divided temperature rise, (dT) with radiation time, (dt). As stated in methodology part, volume rate of heat generation also measured because the volumetric heating in microwave radiation is uniform. Lastly, the ability of each samples to absorb and consent to electromagnetic penetrate through the samples is examined.

All experimental results showed that microwave radiation is very effective way to demulsified water in crude oil. The heating rates of irradiated samples, rate of heat generation, dielectric properties were shown in Table 2.0 (50-50 w/o %) and Table 3.0 for 20-80 w/o%. The heating rate also is compared with crude oil and water. (Listed in Table 4.0 and 5.0)

Table 2: Experimental Results for Microwave Radiation at 20-80 w/o %

| Radiation time (sec) | $T_o$= 27°C, ΔT (°C) | Heating rate, dT/dt (°C/sec) | Rate of heat generation, $q_{mw}$ (cal/sec. cm$^3$) | Dielectric constant, $\varepsilon'_r$ | Dielectric loss, $\varepsilon''_r$ | Loss tangent[a], tan δ = $\varepsilon''_r/\varepsilon'_r$ |
|---|---|---|---|---|---|---|
| 10 | 12.95 | 1.295 | 0.221 | 17.958 | 4.624 | 0.257 |
| 20 | 22.45 | 1.123 | 0.192 | 17.314 | 2.628 | 0.152 |
| 30 | 29.76 | 0.992 | 0.169 | 16.819 | 1.968 | 0.117 |
| 40 | 38.68 | 0.967 | 0.165 | 16.215 | 1.503 | 0.093 |
| 50 | 46.33 | 0.927 | 0.158 | 15.696 | 1.249 | 0.080 |
| 60 | 54.73 | 0.912 | 0.156 | 15.127 | 1.053 | 0.070 |
| 70 | 61.86 | 0.884 | 0.151 | 14.644 | 0.928 | 0.063 |
| 80 | 70.21 | 0.878 | 0.150 | 14.078 | 0.815 | 0.058 |
| 90 | 74.70 | 0.830 | 0.142 | 13.774 | 0.765 | 0.056 |
| 100 | 81.39 | 0.814 | 0.139 | 13.321 | 0.700 | 0.053 |
| 110 | 85.54 | 0.778 | 0.133 | 13.040 | 0.665 | 0.051 |
| 120 | 90.20 | 0.752 | 0.128 | 12.724 | 0.630 | 0.050 |

[a] Values determined at 2.45 GHz

Table 3: Experimental Results for Microwave Radiation at 50-50 w/o %

| Radiation time (sec) | $T_o$= 27°C, ΔT (°C) | Heating rate, dT/dt (°C/sec) | Rate of heat generation, $q_{mw}$ (cal/sec. cm³) | Dielectric constant, $\varepsilon'_r$ | Dielectric loss, $\varepsilon''_r$ | Loss tangent[a], $\tan\delta = \varepsilon''_r/\varepsilon'_r$ |
|---|---|---|---|---|---|---|
| 10 | 12.05 | 1.205 | 0.544 | 41.700 | 12.447 | 0.298 |
| 20 | 20.92 | 1.046 | 0.472 | 40.207 | 7.064 | 0.176 |
| 30 | 27.96 | 0.932 | 0.421 | 39.022 | 5.245 | 0.134 |
| 40 | 36.37 | 0.909 | 0.411 | 37.607 | 4.004 | 0.106 |
| 50 | 43.83 | 0.877 | 0.396 | 36.352 | 3.306 | 0.091 |
| 60 | 51.79 | 0.863 | 0.390 | 35.012 | 2.785 | 0.080 |
| 70 | 58.71 | 0.839 | 0.379 | 33.848 | 2.448 | 0.072 |
| 80 | 66.67 | 0.833 | 0.376 | 32.508 | 2.149 | 0.066 |
| 90 | 73.59 | 0.818 | 0.369 | 31.344 | 1.942 | 0.062 |
| 100 | 78.53 | 0.785 | 0.355 | 30.513 | 1.816 | 0.060 |
| 110 | 82.64 | 0.751 | 0.339 | 29.821 | 1.724 | 0.058 |
| 120 | 88.78 | 0.740 | 0.334 | 28.788 | 1.601 | 0.056 |

[a] Values determined at 2.45 GHz

Table 4: Experimental Results for Water in Microwave Radiation

| Radiation time (sec) | $T_o$= 27°C, ΔT (°C) | Heating rate, dT/dt (°C/sec) | Rate of heat generation, $q_{mw}$ (cal/sec. cm³) | Dielectric constant, $\varepsilon'_r$ | Dielectric loss, $\varepsilon''_r$ | Loss tangent[a], $\tan\delta = \varepsilon''_r/\varepsilon'_r$ |
|---|---|---|---|---|---|---|
| 10 | 9.56 | 0.956 | 0.955 | 82.004 | 31.572 | 0.385 |
| 20 | 17.86 | 0.893 | 0.892 | 79.217 | 16.619 | 0.210 |
| 30 | 24.54 | 0.818 | 0.817 | 76.974 | 11.993 | 0.156 |
| 40 | 30.07 | 0.752 | 0.751 | 75.117 | 9.734 | 0.130 |
| 50 | 35.28 | 0.706 | 0.705 | 73.367 | 8.261 | 0.113 |
| 60 | 38.88 | 0.648 | 0.647 | 72.158 | 7.477 | 0.104 |
| 70 | 43.56 | 0.622 | 0.622 | 70.586 | 6.653 | 0.094 |
| 80 | 47.83 | 0.598 | 0.597 | 69.152 | 6.044 | 0.087 |
| 90 | 50.83 | 0.565 | 0.564 | 68.145 | 5.678 | 0.083 |
| 100 | 54.63 | 0.546 | 0.546 | 66.869 | 5.273 | 0.079 |
| 110 | 59.82 | 0.544 | 0.543 | 65.126 | 4.804 | 0.074 |
| 120 | 64.42 | 0.537 | 0.536 | 63.581 | 4.452 | 0.070 |

[a] Values determined at 2.45 GHz

Table 5: Experimental Results for Crude Oil in Microwave Radiation

| **Radiation time (sec)** | **$T_o$= 27°C, ΔT (°C)** | **Heating rate, dT/dt (°C/sec)** | **Rate of heat generation, $q_{mw}$ (cal/sec. cm3)** | **Dielectric constant, $\varepsilon'_r$** | **Dielectric loss, $\varepsilon''_r$** | **Loss tangent[a], tan δ = $\varepsilon''_r/\varepsilon'_r$** |
|---|---|---|---|---|---|---|
| 10 | 1.98 | 0.198 | 0.000692 | 2.219 | 0.001 | 0.00059 |
| 20 | 2.97 | 0.149 | 0.000519 | 2.218 | 0.001 | 0.00064 |
| 30 | 4.31 | 0.144 | 0.000502 | 2.217 | 0.002 | 0.00072 |
| 40 | 5.67 | 0.142 | 0.000496 | 2.216 | 0.002 | 0.00079 |
| 50 | 7.02 | 0.140 | 0.000491 | 2.215 | 0.002 | 0.00086 |
| 60 | 8.32 | 0.139 | 0.000485 | 2.214 | 0.002 | 0.00093 |
| 70 | 9.95 | 0.142 | 0.000497 | 2.213 | 0.002 | 0.00102 |
| 80 | 11.49 | 0.144 | 0.000502 | 2.212 | 0.002 | 0.00110 |
| 90 | 12.69 | 0.141 | 0.000493 | 2.211 | 0.003 | 0.00116 |
| 100 | 13.63 | 0.136 | 0.000477 | 2.210 | 0.003 | 0.00121 |
| 110 | 14.84 | 0.135 | 0.000472 | 2.210 | 0.003 | 0.00128 |
| 120 | 15.75 | 0.131 | 0.000459 | 2.209 | 0.003 | 0.00132 |

[a] Values determined at 2.45 GHz

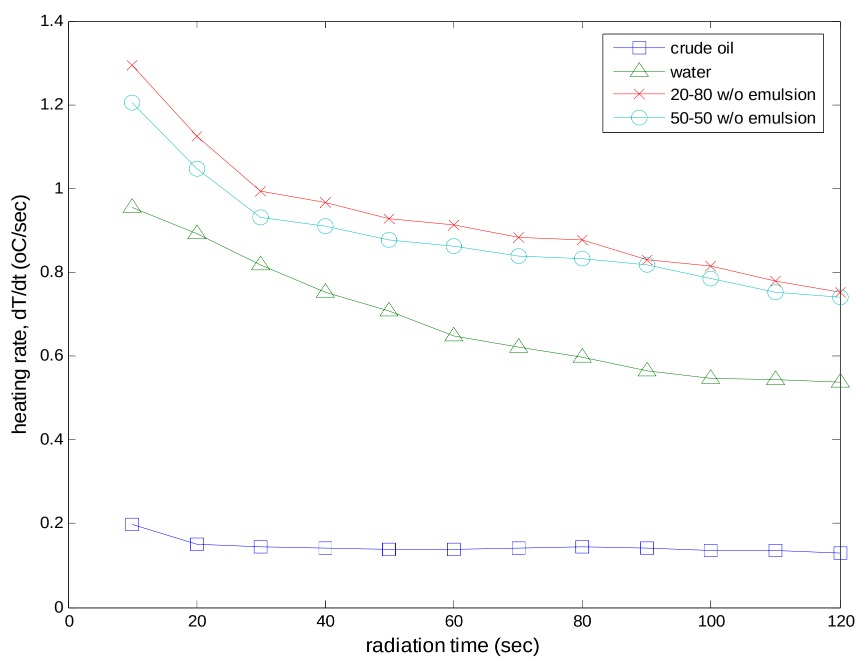


Figure 1: Rate of Temperature Increase for Varied Phase Volume Ratio and Water

The first question is to be discussed about the effects of heating rate for water, crude oil and emulsion under influenced of microwave radiation. It is crucial to confer the mechanisms of heating which at the early stage; conduction flow plays an important role than convective mode. As heating is proceed, the local heating on the surface samples layers causes different surface tension and called as Marangoni effects. During heating process, the convection flow plays an important role rather than conduction flow. This causes the flow of sample from hot region at the centre of the wall container to colder region at the side of wall container. In heating methods, temperature of materials can be increased either directly or indirectly (Hui, 2006). Heating rate was calculated from temperature increase divided by radiation time. Fig 1 illustrated the effects of water, crude oil and emulsion into heating rate. The average of heating rate of crude oil is 0.145 $^oC$/sec which is the lowest value compared with water and emulsion. Water obtained lower than emulsion (0.682 $^oC$/sec) and 20-80 w/o% emulsion shows the highest (0.911 $^oC$/sec) followed by 50-50 w/o %.( 0.894 $^oC$/sec). Results clearly observed the heating rate is inversely proportional with the temperature increasing. 20-80 w/o % shows the heating rate for radiation time until 120 seconds was higher than 50-50 w/o %.

Tables 2-4 indicated the results of dielectric properties. For both Tables 2 and 3 were observed volume phase ratio has great effect on dielectric properties and volume heat generation. As mention before, polar solvents are needed for microwave heating. The materials should be contained of water (polar solvents) to be heated under influenced of electromagnetic. Ideally, previous researchers (Hui, 2006; Ahmed et al. 2007), believed that the more polar solvent, more readily the microwave irradiation is absorbed and the higher temperature obtained as examined through this study.

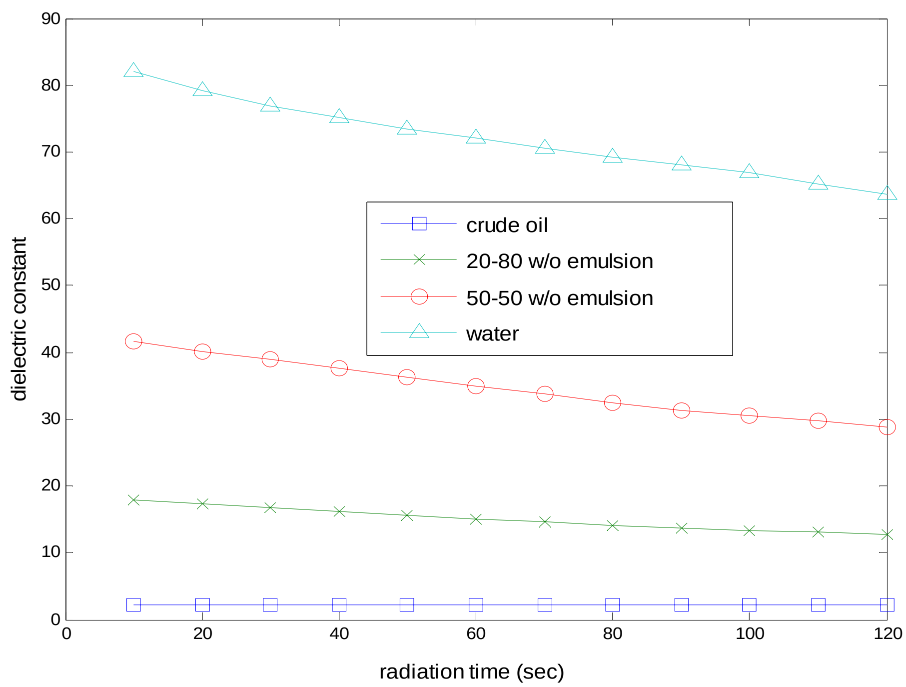


Figure 2: Effects of Dielectric Constant, ε'r onto Emulsions and Water

Secondly is examined the effects of dielectric properties for crude oil, water and emulsion. The study of dielectric properties is the crucial part when engross with microwave radiation. The ability of sample to absorbed and converted thermal heating within the samples is computed by measured dielectric properties. As mention earlier, non polar solvents are not heated under microwave radiation heating. As the increasing polar molecules into the samples, the ability of samples to absorb electromagnetic and converted into thermal heating also increases. In order to compare the abilities of emulsions (20-80 and 50-50 w/o %) and water to generate heat from microwave irradiation, their capabilities to absorb and convert energy to heat can be accounted by measuring the dielectric properties of each sample until 120 sec. The dielectric properties were described how the emulsions interact with electromagnetic radiation. Change of dielectric properties was correlated with the free and bound water content of the emulsion. Fig 2 shows the effect of phase volume ratio to the dielectric properties. Since the frequency of microwave is set as 2.45 GHz, the dielectric constant can only change with temperature. From Fig 2, water has dielectric constant which decreases from 82 at 35°C to 63 at 95°C. As dipole molecules (water) are increasing, the temperature gradually increased and lead reduction of viscosity. Thus, this cause will increase in ionic mobility and consequently dielectric properties. The higher loss tangent, the facilities of samples with microwave irradiation is better. As viewed in Fig 2, water possessed the highest loss tangent compared with the emulsion. This result is correlated with studied by Lidstrőm et al., (2001) which mentioned that loss tangent higher in polar solvent.

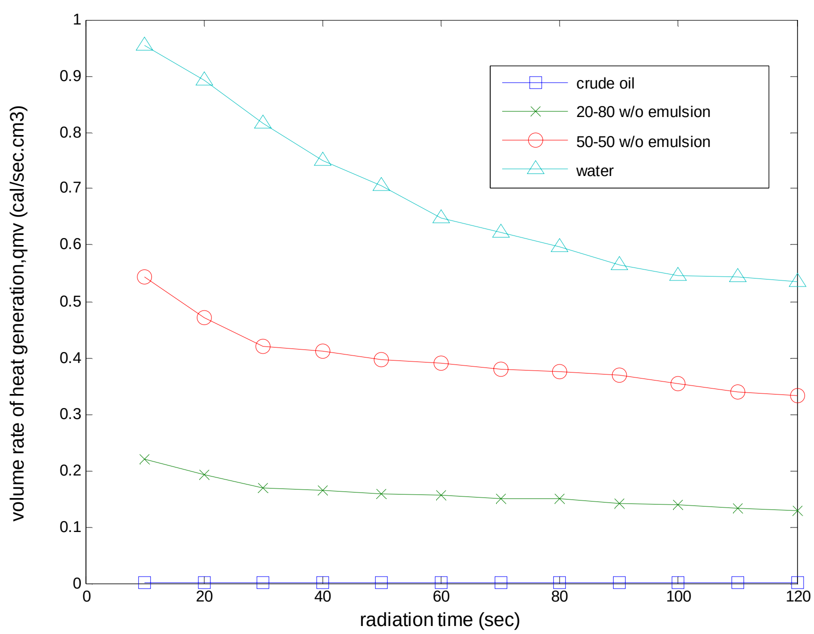


Figure 3: Effects of Phase Volume Ratio, Water and Crude Oil onto Volume Heat Generation

Figure 3 observed the volume heat generation decreased by radiation time. The average heat generation for 20-80 and 50-50 w/o % are 0.1555 and 0.4038. While water obtained the highest volume heat of generation, 0.6812 and crude oil is the lowest which almost zero, 0.000155 cal/sec.cm$^3$. It is obviously observed that heat generation increased as increasing phase volume ratio and polar solvents. As discussed before, temperature increased gradually by radiation time. The increasing of temperature reducing the heating rate thus, decreased volume of heat generation (Eq. 2). Water that has smaller volume in crude oil has higher rate of increasing temperature due to larger heat generation per unit volume. This is because the smaller water content has greater penetration depth than its thickness causes the interference of waves reflected from the interface of water and air at lower side due to the difference of dielectric properties of water and air (Cha-um et al., 2008).

## CONCLUSION

Microwave heating is very convenient to demulsified water in crude oil. The heating is instantaneous, very rapidly particularly at higher volume phase ratio which, microwave radiation can be absorbed by polar solvents. The effects of phase volume ratio also can be indicated through dielectric properties and volume heat of generation. Higher loss tangent obtained for the most effectiveness of materials absorbed the microwave radiation and converted into heat which, at high phase volume ratio.

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**Nomenclature**

| | |
|---|---|
| API | American Petroleum Institute gravity |
| cP | centipoise |
| Cp | specific heat capacity (cal/g.$^{o}$C) |
| $Q_{abs}$ | volume rate of heat generation (cal/sec.cm$^3$) |
| rpm | rotation per minute |
| T | temperature ($^{o}$C) |
| w/o | water-in-crude oil |
| w/w | weight per weight |

*Greek symbols*

| | |
|---|---|
| $\rho$ | density (g/cm$^3$) |
| $\varepsilon''$ | dielectric loss |
| $\varepsilon'$ | dielectric constant |
| $\tan\delta$ | loss tangen |
| $\Phi$ | volume fraction |
| $\alpha$ | constant |