Study on Demulsification of Water-in-Oil Emulsions
Via Microwave Heating Technology

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Abstract: A batch microwave heating process of 2450 MHz was examined on crude oil emulsions. The
mechanism of microwave heating is essentially that of dielectric heating. After exposing the emulsion to the
microwave electromagnetic EM field, molecular rotation and ionic conduction due to the penetration of EM into
the emulsion are responsible for the internal heating. In this study, microwave demulsification method was
applied on a 50-50 and 20-80% water-in-oil emulsions with microwave exposure time varied from 20 to 180 sec.
Transient temperature profiles of water-in-oil emulsions inside a cylindrical container were measured. The
temperature rise at a given location was almost horizontal (linear). The rate of temperature increase of emulsions
decreased at higher temperature due to decreasing dielectric loss of water. Results of this work show that
microwave radiation is a dielectric heating technique with the unique characteristic of fast, volumetric and
selective heating is appropriate and has the potential to be used as an alternative way in the demulsification
process. Microwave demulsification of water-in-oil emulsions does not require chemical additions.

Key words: Demulsification, batch process, microwave heating, w/o emulsion

INTRODUCTION

Electromagnetic radiation in the frequency range
300 MHz to 300 GHz are known as microwaves
(Thamay and Ayappa, 1997), microwave energy is a
non-ionizing radiation that causes molecular motion by
migration of ions and dipole rotations, but does not cause
changes in molecular structure (Kingston and Tassie,
1988) and wavelengths ranging from a few cm to a
few nm (Chatterjee et al., 1998). In the past 20 years,
microwave (MW) energy has been widely applied in food
and chemical processing for heating, thawing (melting),
sintering of ceramics and many others (Ayappa et al.,
1992; Basak and Ayappa, 1997; 2001). Faster cooking
times and energy savings over conventional cooking
methods are the primary benefits. Various oil-in-
water (o/w) and water-in-oil (w/o) emulsions occur in
industrial operations, such as petroleum refining, oil
and gas production (Fang et al., 1995) and food
processing industries, in this regards, efficient heating of
emulsions is required for a faster processing based on
industrial demand. Microwave heating, because of its
volumetric heating effects, offers a faster processing rate.
In conventional thermal processing, energy is transferred
to the material through convection, conduction and
radiation of heat from the surfaces of the material. In
contrast, microwave energy is delivered directly to
materials through molecular interaction with the
electromagnetic field. In heat transfer, energy is
transferred due to thermal gradients, but microwave
heating is the transfer of electromagnetic energy to
thermal energy and is energy conversion, rather than heat
transfer (Thosterson and Chu, 1999). This difference in
the way energy is delivered can result in many potential
advantages to using microwaves for processing of
materials. The transfer of energy does not rely on
diffusion of heat from the surfaces and it is possible to
achieve rapid and uniform heating of thick materials. In
recent literature, many researchers reported non-thermal
phenomena that have been broadly termed 'microwave
effects'. Examples for microwave effect include enhanced
reaction rates of thermosetting resins during microwave
curing (Marand et al., 1992) and faster densification rates
in ceramics sintering (Janney and Kimrey, 1991). As
materials are processed, they often undergo physical and
structural transformations that affect the dielectric
properties. Thus, the ability of microwaves to generate
heat varies during the process. Sharp transformations in
the ability of microwaves to generate heat can cause
cdifficulties with process modeling and control.
Understanding the generation, propagation and
interaction of microwaves with materials is critical.
properties of the electromagnetic field, chemical composition of the material being processed, structural changes that occur during processing, size and shape of the object being heated and the physics of the microwave/materials interactions all complicate microwave processing. Therefore, in view of the above mentioned, the microwave heating technology was applied on water-in-oil emulsions with the following specific objectives: volume rate of heat generation, microwave power absorption at any location in the sample and temperature distribution in the sample.

MATERIALS AND METHODS

In this study, Elba domestic microwave oven model EMO 808SS, its rated power output is 900 watts and its operation frequency is 2450 MHz was used in heating water-in-oil emulsion samples. A 900 mL graduated cylindrical glass was used as sample container. The diameter and height of emulsion sample in the container were 11.5 and 11 cm, respectively.

Three thermocouples type (K-IEC-584-3) were connected to Fico-TC-08 data logging and then connected to microwave oven as shown in Fig. 1. The data logger was connected to PC; with Fico Log R5.08.3 software. The thermocouples were inserted to different locations top, middle and bottom of the emulsion sample to measure local temperatures.

Sample preparation and procedures: The crude oil samples were obtained from Petronas refinery at Malaka city, two types of crude oil were collected namely, heavy oil and light crude oil. 50-50 and 20-80% water-in-oil emulsions were prepared using the same volumes of oil and water. Emulsions were prepared in 900 mL graduated beakers, with ranges by volume of the water and oil phase. The microwave radiation was set to its highest power setting. The water phase is tap water. The emulsions were agitated vigorously using a standard three blade propeller at speed of 1600 rpm and temperature 28°C for 7 min. The concentrations of water in samples were 20-50% by volume. The container of emulsion sample was placed in the center of Elba domestic microwave oven model: EMO808SS. Three thermocouples were inserted in the emulsion sample at different locations, top, middle and bottom. The emulsion samples were heated with microwave radiation for 20, 40, 60, 80, 100, 120, 140, 160, 180 and 200 sec. Temperature profiles of emulsions inside a cylindrical container during batch microwave heating at 2450 MHz were recorded by Fico-TC-08 data logging. The surfactant used in this study was the commercially available Triton X-100; this Triton X-100 is a non-ionic water soluble molecule. The emulsifying agent was used as manufactured without further dilution. In order to prepare water-in-oil emulsions, the agent-in-oil method was followed; that is, in this study, the emulsifying agent (Triton X-100) was dissolved in the continuous phase (oil), then water was added gradually to the mixture. The volume of water settled to the bottom was read from the scale on the beaker with different times. The amount of water separation in percent was calculated as separation efficiency (e) from volume of water observed in the beaker as follows:

\[
\text{Volume of water layer (mL) } \times 100\% \\
\text{Original amount of water, mL}
\]

The prepared emulsion was used to check for w/o or o/w emulsions. All emulsions investigated were water-in-oil (w/o) emulsion (oil-continuous).

Microwave radiation: A number of studies were carried out on Microwave Heating (MW) of oil and water systems. Microwave heating because of its volumetric heating effects, offers a faster processing rate. The separation of emulsified water from crude oil has several stages, due to gravity settling, water droplet/droplet flocculation takes place as water droplets approach each other (Young et al., 1996). The purpose of heating water-in-oil emulsions with microwave radiation is to separate water from oil. When water-in-oil emulsion is heated with microwave radiation, two phenomena will occur; the first one is the increase of temperature, which causes
reduction of viscosity and coalescence. The result is separation of water without addition of chemicals (Fang et al., 1988, 1989). According to Stoke’s law, if oil is the continuous phase, the settling velocity of water droplets is given by:

\[ v_s = \frac{(\rho_w - \rho_o) g D^2}{18 \mu_o} \]  

(2)

where, \(D\) is the diameter of the droplets. The viscosity of oil very sensitive to temperature, as temperature increases, viscosity decreases much faster than the density difference, \((\rho_w - \rho_o)\) does, the result when viscosity decreases, the droplets size increases. Therefore, microwave heating increases the velocity of water \(v_s\) and accelerates the separation of emulsion. The second phenomenon is coagulation. The higher temperature and lower viscosity make the coagulation process easier. The results are larger particle diameter \(D\) and rapid separation.

**Microwave power generation**: Using microwaves as a source of heat in the processing (heating, melting, drying and thawing) of materials is one of the advantageous because it results in faster, more uniform heating than conventional heating does. This study, focus on generation of microwaves in the oven, temperature distribution, microwave power absorption as well as separation of emulsified water from crude oil. The variables affecting microwave power absorption by an element are dielectric constant and dielectric loss, location and microwave power incident at the load. For a sample in cylinder container, the local microwave power flux calculated as:

\[ P_o = \frac{453.2 + 59.8 ln(m)}{A} \]

(3)

Where, \(m\) is mass (g) of the sample, \(A\) is sample’s container area. The microwave power absorption density at any location within the sample is one of the interesting terms, in this regards, the electric field attenuates (decay) exponentially in \(x\) and \(y\) directions within the sample due to dissipation as heat and can be expressed as Swami (1982):

\[ P_z = P_o e^{-z \alpha} \]

(4)

Where, \(P_z\) is microwave power transmitted. The attenuation factor can be calculated from the electromagnetic field theory given by Von Hippel (1954) as:

\[ \alpha_z = \frac{2\pi f}{c} \left[ \frac{\varepsilon_z}{2} \left( \sqrt{1 + \tan^2 \delta} - 1 \right) \right]^{1/2} \]

(5)

The above equation will be used for calculation of the volume rate of heat generation by microwave radiation as:

\[ q_{mwf} = \frac{2\pi f}{4.184} P_o \]

(6)

If the dielectric properties are assumed to be independent of temperature at Frequency 2450 MHz, the wavelength \(\lambda_o\) and penetration depth \(D_p\) within a sample for a radiation of the above frequency (2450 MHz) are related to dielectric constant \(\varepsilon'\) and dielectric loss \(\varepsilon''\) as follows:

\[ \lambda_o = \frac{c}{f} \left[ \frac{\varepsilon_z \left( \sqrt{1 + \left( \frac{\varepsilon_z}{\varepsilon_c} \right)^2} + 1 \right)}{2} \right]^{1/2} \]

(7)

and

\[ D_p = \frac{c}{2nf} \left[ \frac{\varepsilon_z \left( \sqrt{1 + \left( \frac{\varepsilon_z}{\varepsilon_c} \right)^2} - 1 \right)}{2} \right]^{1/2} \]

(8)

Since microwave heats materials volumetrically, it is possible to calculate the volume rate of microwave heat generation from energy balance equation as:

\[ q_{mwf} = \frac{hA}{V} \left( T_w - T_a \right) + \frac{\varepsilon \sigma}{V} \left[ \left( T_w + 273.15 \right)^4 - \left( T_a + 273.15 \right)^4 \right] + \rho C_p \left( \frac{dT}{dt} \right) \]

(9)

The above equation assumes that the rate of heat transfer from emulsified water droplets to the continuous phase (oil) is very rapid; therefore, water and oil practically have the same temperature (Fang and Lai, 1995). The right hand side of Eq. 9 comprises of three terms, convective heat transfer, radiative heat due to microwave and conductive heat in the sample,
RESULTS AND DISCUSSION

In this study, a batch microwave heating technology was successfully applied and examined on water, oil and emulsion samples. For temperature distributions, a three temperature readings were placed at the top, middle and bottom of the cylindrical sample container. Figure 2 and 3 depict temperature distribution of 50-50 and 20-80% water-in-oil emulsions respectively, for microwave irradiation time varies from 20, 40, 60, 80, 100, 120, 140, 160 and 180 sec. Figure 2 and 3 show the motion and distribution of temperature of water-in-oil emulsions in horizontal direction were slow and emulsions were heated uniformly through the microwaves. This was expected result since the sample container was covered with aluminum foil at the top and bottom and microwaves enter only on the side surface of samples. Also these results shown the temperature rise at a given location was linear, this may attribute due to the small dielectric loss of oil. The same findings were reported by Fang et al. (1995).

![Fig. 2: Temperature distributions of 50-50% w/o emulsions](image)

![Fig. 3: Temperature distributions of 20-80% w/o emulsions](image)
The temperature increase rates of irradiated samples and their volume rates of heat generation were shown in Table 1 and 2 respectively. These samples comprise water, crude oil, 50-50 and 20-80% water-in-oil emulsions. The temperatures of emulsions were obtained from the average values of three location temperature readings. The rate of temperature increase was calculated from temperature increase divided by radiation time. The average rates of temperature increase of 50-50 and 20-80% water-in-oil emulsions are 0.351 and 0.437 °C/sec, respectively. It observed that, the rates of temperature were decreases at temperature increases; this was the expected results since the dielectric loss of water is small.

The energy balance Eq. 9 was used to calculate the volume rates of heat generation; this Equation included three terms, convective heat transfer, radiative heat
Since the purpose of heating water-in-oil emulsions with microwave is to separate water from oil, therefore, the separation efficiency of 50-50 and 20-80% water-in-oil emulsions calculated by using Eq. 1 were shown in Fig. 4 and 5, respectively. The same trend was reported by Fang et al. (1988), Fang et al. (1989) and Chan et al. (2002).

All experimental results showed that microwave radiation is very effective in separation of water-in-oil emulsions. Figure 4 and 5 shows that, microwave radiation can raise the temperature of emulsion, reduce the viscosity and accelerate separation process as suggested by Eq. 2. The rates of temperature increase were decreased at higher temperatures. Fig. 6 shows the phenomenon for water and oil, while Fig. 7 shown the same phenomenon for 50-50 and 20-80% water-in-oil emulsions, respectively.

The wavelength ($\lambda_w$) and penetration depth ($D_p$) were found 1.39 and 3.42 cm respectively. Figure 8 shows the heating rate of temperature increase for water, oil and emulsions versus the radiation time.

CONCLUSIONS

The batch microwave heating process was examined for water, oil and emulsion samples. Results of this study showed that, microwave radiation is a dielectric heating technique with the unique characteristics of fast, volumetric and effective heating is feasible and has the potential to be used an alternative way in the demulsification of water-in-oil emulsions. From temperature distribution profiles of irradiated emulsion, it appears water-in-oil emulsion has been heated quickly and uniformly by microwaves rather than by conventional heating. This new separation technology does not require chemical addition. Furthermore, microwave radiation appears to provide faster separation than the conventional heating methods.

Notation:

- $V_w$: Settling velocity of water droplet (cm/sec)
- $\rho_d$: Density of dispersed phase (g/cm³)
- $\rho_c$: Density of continuous phase (g/cm³)
- $g$: Gravity
- $D$: Droplet diameter (cm)
- $p_e$: Microwave surface power (W)
- $A$: Sample container area (cm²)
- $m$: Mass of sample (g)
- $p_t$: Microwave power transmitted (W)
- $\alpha_e$: Electromagnetic attenuation factor (cm⁻¹)
- $f$: Frequency of incident microwaves
- $\delta$: Loss tangent
- $q_{heat}$: Volume rate of heat generation (W/cm³)
REFERENCES

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