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Abstract: This work investigates the effect of a set of operating variables, including irradiation time, irradiation power, dosage of NaCl, settling time, pH, and the initial oil concentration, on the separation efficiency in the treatment of an oil in water (O/W) type cutting oil emulsion by microwave assisted demulsification. As a result of a series of batch demulsification tests a set of optimum operating conditions was found, consisting of 2 min of microwave irradiation at 280 W, the addition of 14 g/L of NaCl, 1 h settling time, and at a pH of 9.5. A separation efficiency of 93.8% was obtained with these conditions for 50 mL of cutting oil emulsion with an initial oil content of 10 g/L. In addition, data from these tests were treated by a stepwise-regression method which results in a multi-variable equation. This empirical equation was able to describe the separation efficiency fairly well, after excluding those with a separation efficiency less than 40% and temperatures higher than the boiling point. Our principal component analysis (PCA) extracted four principal components with 78% of the total variance explained.

Keywords: Cutting oil emulsion, demulsification, microwave irradiation, optimum operating conditions, salt

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INTRODUCTION

Cutting oil in water (O/W) emulsions is widely used in metal fabrication industries to provide lubrication and cooling for various metal working processes. Used emulsion is routinely replaced by fresh emulsion to maintain proper performance. These processes therefore produce amounts of waste oil emulsion that have to be treated before being released into the environment. Separation of oil and water from this emulsion, a process described as demulsification, implies breaking the emulsified film around droplets of water or oil so that coalescing and gravitational settling may occur (1).

Typical demulsification techniques include chemical destabilization with dissolved air flotation (2,3), membrane processes (4,5), freezing and thawing (6–8), electrical (9),thermal (10–19), and mechanical methods. The dissolved air flotation process is commonly used to treat O/W emulsions, but the separated oil cannot be reused due to the added chemicals and disposal of the resultant scum is necessary.

Although membrane processes have high efficiency in terms of oil recovery, they are not feasible for high viscosity emulsions. In a W/O emulsion, the freeze/thaw method causes the molecules of surfactants to move away from the oil-water interface during the freezing process. Thus, the free water droplets coalesce and can be separated (6,8). Economic considerations for the freeze/thaw method make it critical to operate at the optimum freezing temperature, around −40°C (6). Electrical demulsification reduces the thickness of the film at the oil-water interface by applying a high electrical field, so that the droplets are allowed to approach each other (9). Although electrical demulsification is an effective process, it requires a special design of the high electrical field; the additional use of chemicals or heat is often found necessary.

Microwave irradiation has been successfully tested in laboratory and field operation (11,12). It does not require the addition of chemicals so the oil recovered from the emulsion can be reused. With microwave irradiation, there are two main mechanisms occurring simultaneously. One is the rapid increase of temperature which reduces the viscosity of the emulsion. The other is molecular rotation, which neutralizes the Zeta potential because of the rearrangement of electrical charges surrounding the water droplets (11). Hence, water droplets coalesce and result in the separation of the emulsion.

The concept of microwave demulsification was first introduced by Klaili (15) and Wolf (16). In Wolf's patent application (16), the demulsification is effective in treating emulsions with 50% (or greater) oil content by weight. More power was required for emulsions with higher water content. Later, Fang et al. (11,12) carried out a field test on 188 barrels of crude-oil/water emulsion in tanks by microwave irradiation. The emulsion was separated into 146 barrels of oil and 42 barrels of clear water under certain conditions. They also tested 1:1 and 3:7 water-in-oil systems by microwave demulsification, and found that more than 80% of the water could be separated from the emulsions.

The salt-assisted microwave irradiation process for demulsification has been found to be effective due to the increase in the conductivity of the solvent, which accelerates the rate of heating (17,19). Chang and Chen (14) studied the effect of salts on the demulsification of W/O emulsions by microwave irradiation. The demulsification rate was found to increase with the electrolyte concentration (NaCl, KCl, $NaNO_3$, and Na_2SO_4) in the range of dilute solutions ($<0.5\,M$). Electrolyte was applied to lower the Zeta potential, which results in destabilization of the emulsions. Rios et al. (20) destabilized a cutting oil emulsion by adding an inorganic salt and over 90% separation efficiency was obtained. Fortuny et al. (19) investigated the effect of pH, salt (NaCl) and water content on a crude oil emulsion (W/O) by a microwave demulsification process. According to this study, the addition of dissolved salts significantly increases the heating efficiency and destabilizes the emulsions. The increase of the initial water content of the emulsion increases the coalescence efficiency due to a decrease in the distance between the water droplets which enhances the probability of their collision. The pH value was also found to influence the stability of the emulsion. In general, the increase of pH favors the stability of O/W emulsions (21,22). However, in the W/O type emulsion, the increase of pH decreases the stability because of the increase in hydrophilicity (19).

The present study is aimed to examine the demulsification of an O/W cutting oil emulsion under microwave irradiation. The following operating variables are considered: the dosage of inorganic salt, the microwave irradiation power and irradiation time, the initial oil concentration, the settling time, and the pH value. A series of batch demulsification tests was carried out with variation of the operating variables in order to obtain an optimal operating condition for the demulsification of a cutting oil emulsion.

EXPERIMENT AND METHOD

Preparation of Emulsions

Emulsions were prepared by homogenizing known amounts ($2 \sim 30$ g) of a cutting oil (Casting, Chiew Tay Lubicant Company, Taiwan) with the addition of double-distilled water (DDW) to 400 mL in a juice mixer

Table 1. Properties of the cutting oil (casting) used in this study

Density at 15.6°C (kg m^{-3})	947
Viscosity at 40°C (cSt)	31.2
Pour Point (°C)	−9.0
Flash Point (°C)	180
Water Content (vol %)	0.2

(HR1700, Philips, 18000 ~ 21000 rpm, 250 W) for 1 min. The properties of this cutting oil are given in Table 1. Each emulsion was allowed to settle for at least 5 days to confirm its stability. The emulsifier in the cutting oil is composed of two anionic surfactants, sulfonate and fatty carboxylic acid salts. Results of the Zeta potential and the mean of particle size, measured by a Zetasizer apparatus (Nano ZS, Malvern), of the raw emulsion in different concentration are shown in Table 2. It indicates that the oil droplets of the emulsions are uniformly distributed and negatively charged.

Demulsification Tests

The demulsification tests were conducted using a domestic microwave oven (JMO3017, 2450 MHZ, Jyeproud, Taiwan) and an oil-bath heating plate. This microwave oven is capable of providing a maximum of 700 W of continuous microwave irradiation power.

In a series of batch demulsification tests, 40 mL of emulsion with different oil content was combined with 10 mL of a salt solution with varying NaCl content (0 ~ 0.8 g), placed in a glass vessel (100 mL) and

Table 2. The Zeta potential and mean of particle size of the raw emulsion in different oil concentration (at 25°C)

Oil concentration (g/L)	Zeta potential (mV)	Mean of particle size (nm)
8	−77.6	162
10	−76.3	164
12	−73.4	166
14	−71.5	163
16	−70.7	164
18	−67.8	165
20	−65.2	165

mechanically mixed with a magnetic stirrer. The initial oil content and the NaCl concentration in the resulting solution were $4 \sim 60\,g/L$ and $0 \sim 16\,g/L$, respectively. The pH value of the sample was adjusted using H_2SO_4(2.5 N) and NaOH (1.67 N) solution. Solutions of various inorganic salts, KCl, $MgCl_2 \cdot 6H_2O$, $CaCl_2 \cdot 2H_2O$ and $ZnCl_2$, were used instead of NaCl but with the same ionic strength and their demulsification efficiencies were compared.

Both microwave irradiation and oil-bath heating plate were used for demulsification tests in this study. The oil-bath temperature was maintained at 100°C by a heating plate ($350 \sim 700$ W). The microwave irradiation demulsification tests were carried out with specific values of irradiation time, irradiation power, salt content, settling time, pH, and the initial oil concentration. After microwave irradiation or oil-bath treatment, a thermometer (UF-1A, pen-type thermometer, $-40°C \sim 200°C$, Fecca Company, Taiwan) was immediately inserted into each sample to measure the instantaneous temperature in the microwave oven and the oil-bath. Then the sample was allowed to settle in a stainless tube (length 45 cm, inner diameter 1/2") at room temperature for cooling and coalescence. At 7 cm above the bottom of the stainless tube, 2 mL solution was sampled and its oil content was determined by a spectrophotometer (U-2100, Hitachi Cooperation, Japan) with a double-beam light path at 380 nm, using DDW as a reference. The data reported are an average of at least three runs. The performance of these tests is evaluated by the separation efficiency (S) which is defined as:

$$S(\%) = 100(R_0 - R)/R_0 \quad (1)$$

where R_0: oil content before demulsification (mg/L)

R (residual oil content): oil content after demulsification (mg/L).

RESULT AND DISCUSSION

Effect of Microwave Irradiation Time

Figure 1 shows the effect of the microwave irradiation time on the separation of the cutting oil emulsion (50 mL with initial oil content of 10 g/L) using 280 W of microwave irradiation, 14 g/L of NaCl, 1 h settling time, and a pH of 9.5. The separation efficiency continuously increases with irradiation time until 120 s have passed. The separation efficiency reaches 93.8% with a temperature of 97°C, for an irradiation time of 120 s. This operating condition, 280 W and 120 s of microwave irradiation, the addition of 14 g/L of NaCl, 1 h settling time, and a pH of 9.5, serves as the

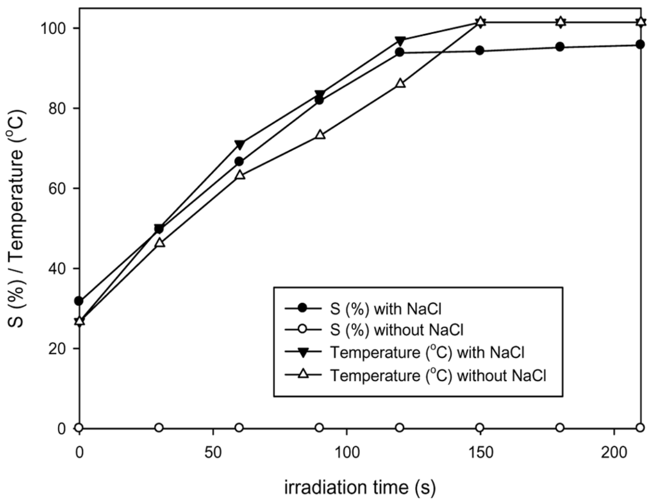

Figure 1. Effect of irradiation time on the treatment of 50 mL emulsion with an initial oil content of 10 g/L at pH 9.5, 14 g/L NaCl, irradiation power 280 W, and settling time 1 h.

baseline condition hereafter. In contrast, with the same operating conditions, there is basically no separation without the NaCl addition. Obviously, the addition of NaCl made a big difference. It is believed that NaCl reduces the electrical double layer thickness at the interface between the oil and water (20) and causes superheating of the emulsion during the microwave irradiation (17,19). In addition, it is observed that the temperature of emulsions with added NaCl is greater for irradiation times below 150 s. Similar observations were also reported by Xia et al. (17) and Fortuny et al. (19). On the other hand, when the irradiation time is above 150 s, the temperature of the emulsions reaches 101.5°C (boiling) whether NaCl is present or not. There is no further increase in temperature and separation efficiency (93.8%) with irradiation time. In this study, the temperature of the emulsions was controlled below the boiling point of water for safety concerns. Therefore, we conclude that 2 min of irradiation time is the optimum condition and used as the baseline from now on.

Effect of Microwave Irradiation Power

The separation efficiency and sample temperature continuously increased with microwave irradiation power until the power level reached 280 W

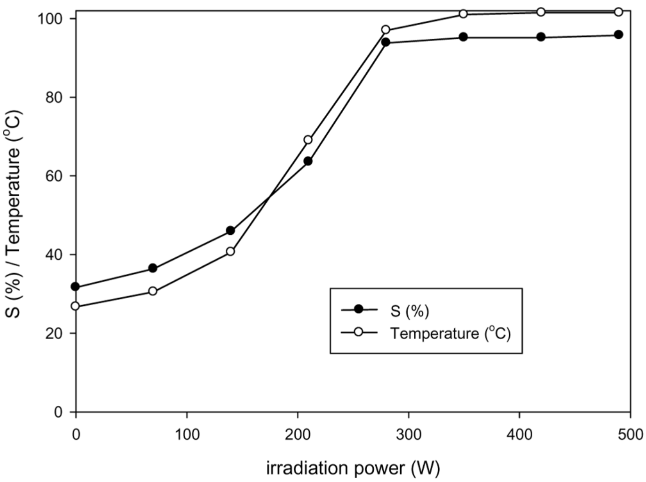

Figure 2. Effect of irradiation power on the treatment of 50 mL emulsion with an initial oil content of 10 g/L at pH 9.5, 14 g/L NaCl, irradiation time 2 min, and settling time 1 h.

(Fig. 2). There is no further improvement in efficiency or increase of sample temperature with greater irradiation power. This indicates that this microwave power is sufficient to raise the emulsion to the required temperature. Therefore, the optimum operating conditions for the microwave are 280 W power and 2 min irradiation time.

Effect of NaCl Dosage

As stated above, the addition of NaCl effectively enhances the separation efficiency of cutting oil emulsions (Fig. 1). The possible mechanism behind this enhancement has also been discussed above. Therefore the effect of NaCl was studied using the baseline microwave operating conditions with variable NaCl dosage. As shown in Fig. 3, the sample temperature is only 86°C without the addition of NaCl, but it increases to 96°C with only 2 g/L of NaCl added. This provides evidence of the proposed mechanism that the added NaCl causes superheating of the emulsions during microwave irradiation. The emulsion temperature then increases only slightly as the NaCl dose increases from 2 g/L to 16 g/L. However, the separation efficiency increases abruptly for a NaCl dose above 10 g/L for the standard solution (50 mL emulsion with oil 10 g/L) using the baseline conditions (pH 9.5, microwave power 280 W and 2 min irradiation followed by 1 h settling time). The separation

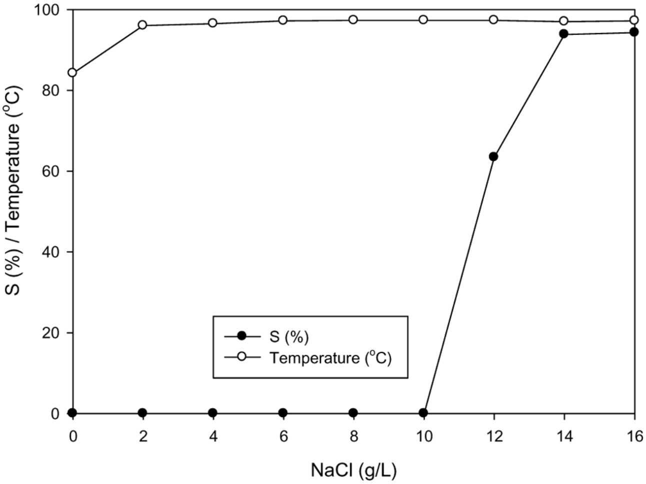

Figure 3. Effect of NaCl dosage on the treatment of 50 mL emulsion with an initial oil content of 10 g/L at pH 9.5, irradiation time 2 min, irradiation power 280 W, and settling time 1 h.

efficiency reaches 93.8% for a NaCl dosage of 14 g/L and there is no significant improvement in the separation as the dosage is increased further. Therefore, the optimum concentration of NaCl is 14 g/L.

Effect of Settling Time

After microwave irradiation, the droplets from the emulsion collide and coalesce during the settling time. Figure 4 shows the effect of settling time on separation efficiency (S) with the baseline microwave conditions as a function of NaCl dose. The results show that the separation efficiency after 1 h settling is 93.8% and 93.9%, with 14 g/L, and 16 g/L of NaCl added, respectively. As the separation does not improve significantly if the settling time is increased further, we conclude that the optimum conditions are a settling time of 1 h and with 14 g/L of NaCl added. Figure 5 shows that the residual oil content decreases exponentially as the settling time increases. The residual oil content can therefore be expressed as follows:

$$R(mg/L) = k_1 + k_2 \exp(-k_3 t) \tag{2}$$

where k_1, k_2, and k_3 are the adjustable parameters and t is the settling time (h).

When the sample settles, the free oil floats to the surface of the emulsion solution due to its lower density. The appearance of the solution

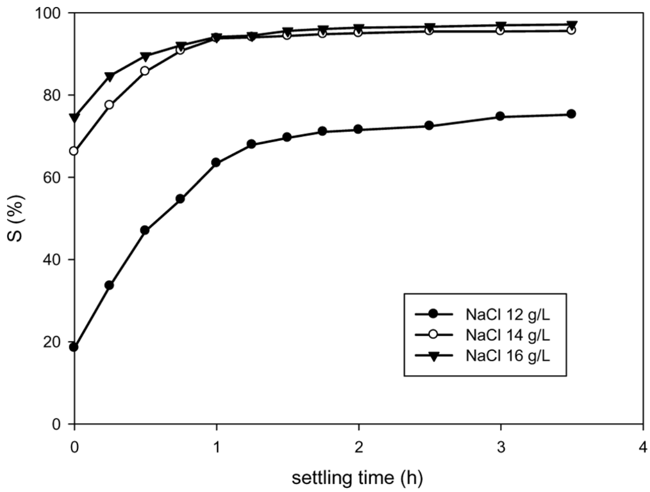

Figure 4. Effect of settling time on the treatment of 50 mL emulsion with an initial oil content of 10 g/L at pH 9.5, irradiation time 2 min, irradiation power 280 W and NaCl 12, 14, and 16 g/L.

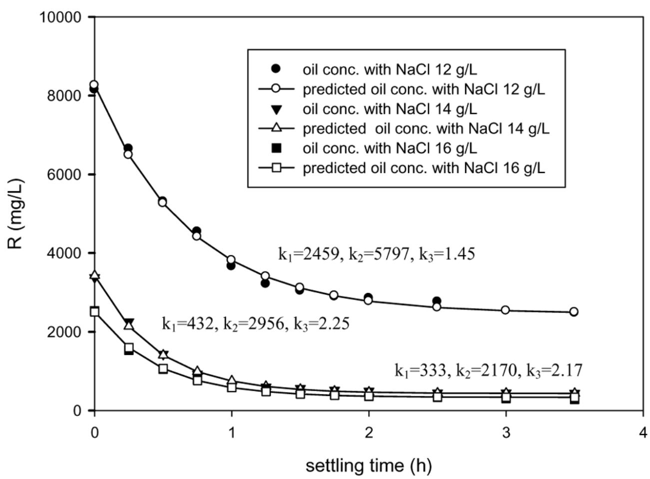

Figure 5. The model equation ($R = k_1 + k_2 \exp(-k_3 t)$) for the residual oil content as a function of settling time after treatment of 50 mL emulsion with an initial oil content of 10 g/L at pH 9.5, irradiation time 2 min, irradiation power 280 W and NaCl 12, 14, and 16 g/L.

Figure 6. Comparison in appearance before (L) and after (R) demulsification of 50 mL emulsion with an initial oil content of 10 g/L at pH 9.5, NaCl 14 g/L, irradiation time 2 min, irradiation power 280 W, and settling time 1 h.

after treatment is much clearer than the untreated emulsion as demonstrated in Fig. 6.

Effect of pH

In Fig. 7, it can be seen that the separation efficiency decreases with increasing pH. It is reasonable to expect this phenomenon for cutting oil emulsions of the O/W type (21,22). As shown in Table 2, the oil droplets in this study (O/W type) are negatively charged as generally expected. Increasing the pH of the solution means that more OH^- ions are adsorbed on to the oil droplets which results in an increase of their surface charge and Zeta potential. This increase favors the stability of emulsions and causes a decrease of separation efficiency (Fig. 7).

Effect of Initial Oil Concentration

The effect of initial oil concentration was investigated using the baseline conditions but with the NaCl dosage also varied. According to the results in Fig. 8, the separation efficiencies with NaCl 14 g/L added are 88.1%

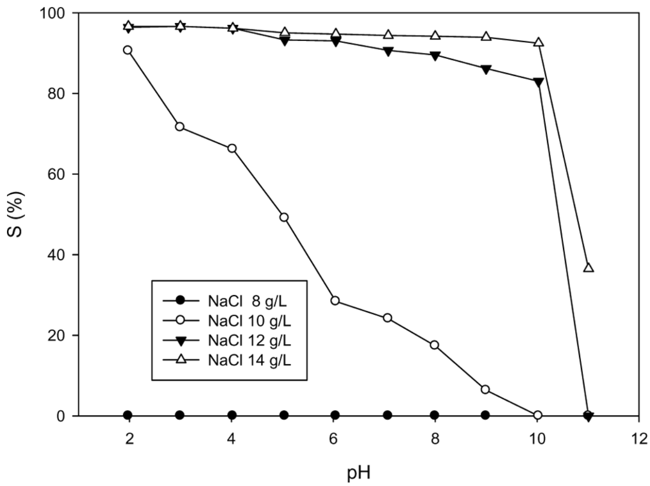

Figure 7. Effect of pH on the treatment of 50 mL emulsion with an initial oil content of 10 g/L at pH 9.5, irradiation time 2 min, irradiation power 280 W, settling time 1 h, and NaCl 8, 10, 12, and 14 g/L.

and 96.4 % for oil content of 4 g/L, 20 g/L, respectively. A similar result is found with the addition of 12 g/L NaCl. Increasing the initial oil concentration tends to decrease the distance between oil droplets and

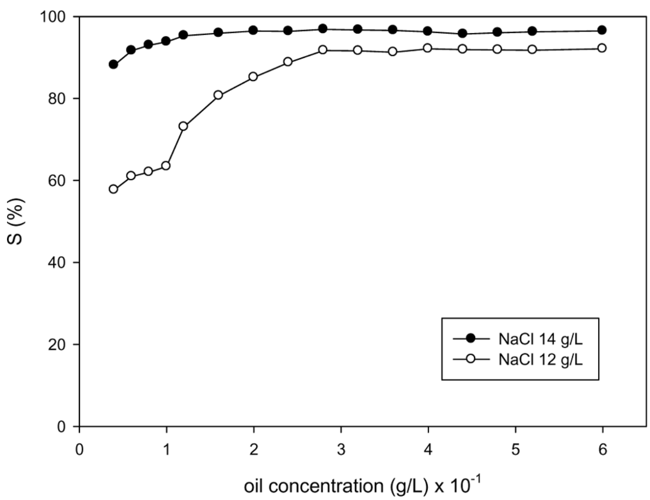

Figure 8. Effect of the initial oil concentration on the treatment of 50 mL emulsion at pH 9.5, irradiation time 2 min, irradiation power 280 W, settling time 1 h, and NaCl 12 and 14 g/L.

increase the collision frequency of the emulsion droplets. In addition, both dipole molecular rotation and ionic conduction caused by microwave irradiation are expected to further increase the collision frequency. This increase in collision frequency favors the coalescence of droplets and so increases the efficiency of treatment (19). The separation efficiency continuously increases to a maximum value of 96.4% and this efficiency remains approximately unchanged with oil content up to 60 g/L. However, it should be noted that though the treatment of emulsions with high initial oil content has high separation efficiency, the residual oil content of the resultant emulsion is expected to be high. Further treatment might be necessary.

The Comparison of Demulsification by Microwave Irradiation and Traditional Heating

Comparison of separation efficiencies by microwave irradiation and the traditional heating method were made using the baseline conditions but with NaCl dosage and heating time as variables. The results shown in Fig. 9 demonstrate that the demulsification efficiency by microwave irradiation is much higher. For example, with 14 g/L NaCl, the separation efficiency is 93.8%, 68.3%, 31.7% by microwave irradiation

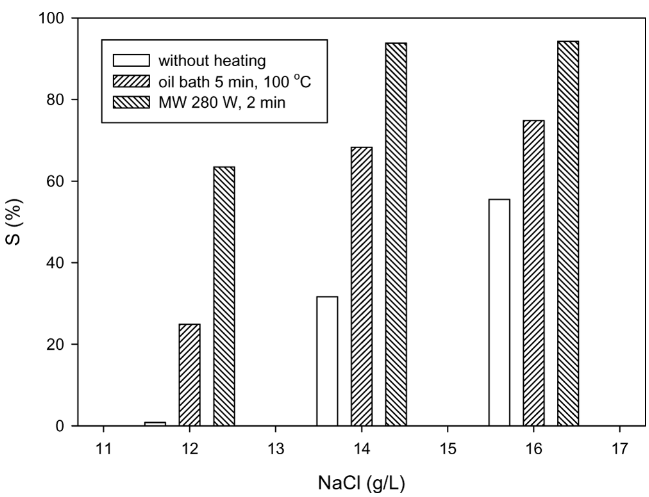

Figure 9. The comparison of microwave irradiation and traditional heating for the treatment of 50 mL emulsion with an initial oil content of 10 g/L at pH 9.5, settling time 1 h and NaCl 12, 14, and 16 g/L.

(280 W, 2 min), oil bath (5 min, 100°C), and without heating, respectively. Similar results are observed with 12 g/L, and 16 g/L of NaCl added.

Effect of Different Inorganic Salts

The effect of various inorganic salts (NaCl, KCl, $CaCl_2$, $MgCl_2$, and $ZnCl_2$) with the standard baseline operating conditions is shown in Fig. 10. The separation efficiency reaches 95% with the addition of $CaCl_2$, $MgCl_2$ and $ZnCl_2$ at ionic strengths of 2.7×10^{-2} M. In contrast, the separation efficiency reaches 93.8% with KCl and 95.3% with NaCl at an ionic strength of 0.24 M. These results show that salts with a divalent cation have greater capability of enhancing demulsification efficiency than those with a monovalent cation.

Statistic Analysis

The data shown in Figs. 1–4 and Figs. 7–8, were used to obtain an empirical equation to describe the demulsification separation efficiency for O/W cutting oil emulsion by the variables used in this study. Those data with efficiency below 40% or with a temperature higher than the boiling point were excluded from this analysis. The following multivariable equation was obtained after statistical analysis by the

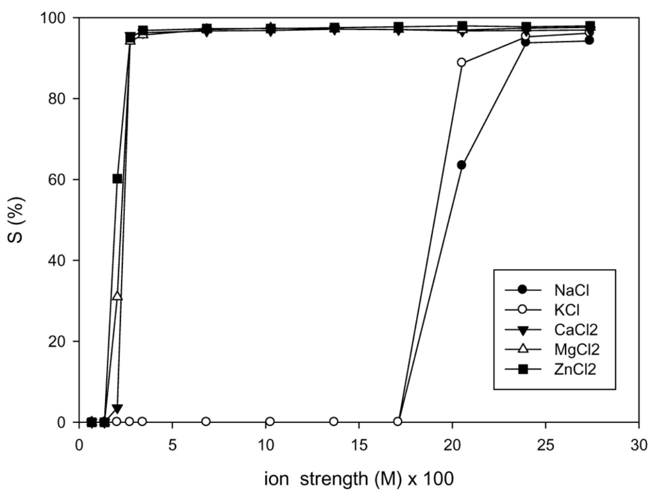

Figure 10. Effect of different inorganic salts on the treatment of 50 mL emulsion with an initial oil content of 10 g/L at pH 9.5, irradiation time 2 min, irradiation power 280 W and settling time 1 h.

stepwise-regression method.

$$S(\%) = 7.748X_1 + 0.311X_2 + 0.380X_3 + 3.559X_4 - 2.691X_5 + 0.412X_6 - 135.07 \quad (3)$$

where S(%) is related to the dosage of NaCl(g/L) added (X_1), the microwave power (W)(X_2), the irradiation time (s)(X_3), the settling time (h)(X_4), the pH value (X_5), and the initial oil concentration (g/L)(X_6). By using this empirical equation, the separation efficiency could be estimated with the operating variable modified. In Fig. 11, the data obtained from this empirical equation were plotted against those from experiments for comparison. The mean relative difference of separation efficiencies between the empirical equation and the experimental results is 8.5%. It demonstrated the empirical equation is able to describe the separation efficiency fairly well. As shown in Table 3, our principal component analysis (PCA) extracted four principal components that explain 78% of the total variance. The four principal components can explain the variance 23.37%, 18.42%, 18.16%, and 18.05%, respectively. A comparison of component loadings for the first component (PC1), the variable of pH is more dominant than others. In contrast, the settling time is the most dominant variable for PC2.

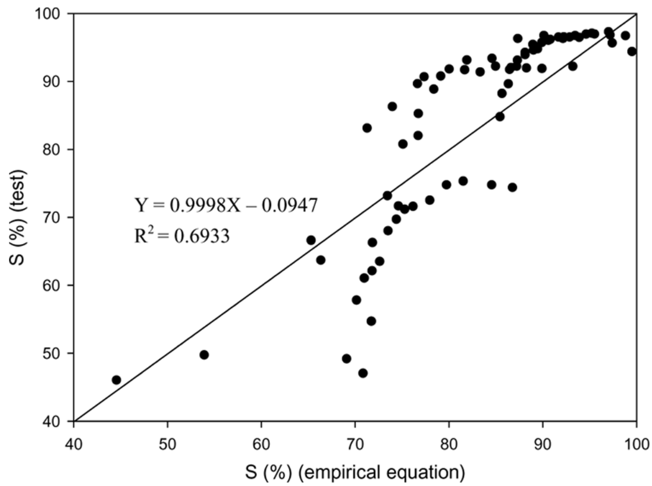

Figure 11. The comparison of separation efficiencies between those calculated from the empirical equation and results from test. (Data with separation efficiency less than 40% or temperatures higher than the boiling point were excluded.)

Table 3. The results of principal component analysis (PCA)

Component/Variable	*Component loading	Initial eigenvalues	% of variance explained
PC1			
pH	0.859		
The dosage of NaCl	0.627	1.43	23.37
Initial oil concentration	0.482		
PC2			
Settling time	0.915	1.15	18.42
Initial oil concentration	0.486		
PC3			
Microwave irradiation power	0.881	1.08	18.16
PC4			
Microwave irradiation time	0.889	1.02	18.05

*Component Loading >0.45.

CONCLUSION

In this study, the application of microwave irradiation has effectively demulsified an O/W type cutting oil emulsion in a short time (2 min) with high efficiency. Six operating variables, the irradiation time and irradiation power, the dosage of NaCl, the settling time, pH, and the initial oil concentration, were studied in a set of batch microwave demulsification tests. The separation efficiency was found to increase with increasing microwave power, irradiation time, NaCl dosage, settling time, and initial oil content until a plateau value was reached, while decreasing with an increase of pH. An optimum set of operating conditions for the treatment of 50 mL cutting oil emulsion with an initial oil content of 10 g/L was found after a set of batch demulsification experiments. The addition of salts (NaCl, KCl, $CaCl_2$, $MgCl_2$, and $ZnCl_2$) was effective in enhancing the separation efficiency due to destabilizing the oil-water interface and raising the rate of heating. Divalent cations were found to have greater capability for enhancing the demulsification efficiency than monovalent cations.

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