



## Formation and characterisation of mint oil/S and CS/water microemulsions

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### ABSTRACT

Preparation and characterisation of microemulsions (ME) with mint oil (MO) as the oil phase were conducted to find a system which could provide a controlled, sustained, and prolonged delivery of MO. The influence of surfactant type and processing parameters on the formation of MO-ME's was studied by comparing the areas of the monophasic ME regions in pseudoternary phase diagrams. A 1:1 mixture of the surfactants, AOT and CrEL, was found to be the most effective, and it produced a ME monophasic area of about 70%. The monophasic regions were analysed by conductivity measurements; the results suggested that O/W regions within the ME phases were formed when the water content was higher than 60–65%. As tested with dynamic light scattering (DLS) and head space (HS) GC analysis, the particle size of O/W ME, with 60% water and AOT/CrEL = 1:1 as surfactants, was 20.0 nm and the encapsulation efficiency was 78.4%. The results indicated that a ME of MO/AOT and CrEL/ethanol/water, may be a promising dispersion for the protection of MO in food products.

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### 1. Introduction

Mint oil (MO) is a complex mixture of comparatively volatile and labile components. These components can degrade during processing and storage, which will lead to changes in the sensory properties of MO. It is thus difficult to manufacture and preserve products containing MO. In addition, the poor solubility of MO in water also makes the manufacturing of stable dispersions difficult. However, MO is a popular flavouring agent in food, pharmaceutical or cosmetic formulations.

In order to increase the stability of products formulated with MO, microencapsulation has been used as a method for entrapping this essential oil within a protective layer of coating materials (Jackson & Lee, 1991). A microemulsion (ME) is a homogeneous dispersion of oil, surfactants and co-surfactant, within an aqueous phase, normally as nano-sized droplets (Moulik & Paul, 1998). Due to their optical isotropic state and thermodynamic stability, MEs have several advantages over emulsions, such as enhanced hydrophobic solubility, better thermodynamic stability, ease of manufacturing (Lawrence & Rees, 2000), stability against oxidation and controlled release (Park & Kim, 1999).

There are two significant problems related to the formulation and preparation of MEs in food, pharmaceutical or cosmetic products. One is the large surfactant concentration needed for the formation of a ME. High surfactant contents can lead to clinical

liabilities as such levels can have potential toxic effects (Lawrence & Rees, 2000). The other is that many of the MEs investigated suffer from a drawback of low capacity of oil solubilisation (Malcolmson & Lawrence, 1995). Rather than a single surfactant, the use of mixed surfactants has been reported, which has helped to overcome this problem (Leung & Shah, 1987; Moreno, Ballesteros, & Frutos, 2003). Moreno et al. (2003) reported that the combined use of Tween 80 and soybean lecithin could increase the oil content in MEs by three-fold. Li et al. (2005) also reported a synergistic effect when using a mixture of surfactants for the formation of oil-in-water MEs. The addition of various polyols or short-chain alcohols as co-surfactants was also found to be helpful in further reducing the interfacial tension, which promotes the formulation of O/W MEs (Garti, Yagmur, Leser, Clement, & Watzke, 2001).

There have been numbers of investigations concerning the preparation of O/W MEs with hydrophobic flavour compounds. Microstructure and phase behaviour-studies of food-grade MEs, with R-limonene as the oil phase, have been reported (Garti, Clement, Leser, Aserin, & Fanun, 1999; Garti et al., 2001; Garti et al., 2004). Tokuoka, Uchiyama, Abe, and Chrishtian (1995) have formulated synthetic perfumes with anionic-nonionic mixed surfactant systems to improve their solubility. However, the effect of a ME formulation on the decrease of the volatilisation of essential oils has not received much attention. The objective of this work was to optimise the formulation of the surfactant system for the formulation of a ME with MO to increase the oil capacity of the ME and to reduce the degree of MO volatilisation.

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## 2. Materials and methods

### 2.1. Materials

Mint oil (70%, w/w) was obtained from Nantong Menthol Factory Co. Ltd., (Nantong, China). Surfactants used were AOT [sodium bis(2-ethylhexyl) sulfosuccinate], Cremophor EL (CrEL) [polyoxyethyl (35) castor oil], Tween 20 [polyoxyethylene (20) sorbitan monolaurate], Brij-35 [polyoxyethylene(23) lauryl ether], Span-20 [sorbitan (20) monolaurate] and lecithin. AOT and Cremophor EL were purchased from Sigma–Aldrich (USA), and the others were purchased from the Sinopharm Group Chemical Reagent Co. Ltd., (Shanghai, China). Sudan IV, Eosin blue, ethanol and NaCl were AR grade products and supplied by the Sinopharm Group Chemical Reagent Co. Ltd., (Shanghai, China). The water used in the formulations was double-distilled.

### 2.2. Influence of surfactant type on formation of mint oil MEs

The phase diagrams were constructed with data from surfactant/co-surfactant (S/CS) titration measurements (von Corswatt & Thoren, 1999). Various ratios of MO and water (10:0, 9:1, 8:2, 7:3, 6:4, 5:5, 4:6, 3:7, 2:8, 1:9, 0:10) and were dispersed in vials. To each vial, a 1:1 (w/w) mixture of a single surfactant and co-surfactant (ethanol) was added dropwise. Following each S/CS addition, the vial was vortexed for 2–3 min and the optical transparency change of samples was evaluated by visual observation. When a critical change in transparency was observed, the sample was kept in a thermostatic water bath at the desired temperature for 24 h to ensure equilibrium. For each vial, the concentration of water, MO, and S/CS at the critical point of transparency change was considered as a point on the boundary between a transparent ME phase and a translucent two phase region. With these points, from the titrations of the 11 vials, a pseudoternary phase diagram was constructed.

Phase diagrams were constructed where the single surfactant was replaced with a binary mixture (1:1, w/w). Three different binary mixtures were made from combinations amongst AOT, Brij35 and CrEL. Phase diagrams with AOT and CrEL at different ratios (2:1, 1:1 and 1:2, w/w) were also constructed. For all the binary surfactant mixtures, the total surfactant to co-surfactant ratio was 1:1.

### 2.3. Influence of processing parameters on formation of mint oil MEs

The effects of the component order and temperature of mixing on the phase diagrams were evaluated. For evaluating the effect of the order of mixing, the stepwise addition of either water, S/CS or MO into a mixture of MO and S/CS, MO and water or S/CS and water, respectively, were evaluated at 30 °C. In order to evaluate the effect of temperature, a binary mixture of AOT and CrEL (1:1, w/w) was used as the surfactant with 1:1 ratio of S:CS. The S/CS was used to titrate a MO and water mixture at 20, 40, or 60 °C.

### 2.4. Conductivity measurements

Three mixtures of MO, AOT and CrEL (1:1), and ethanol (S:CS 1:1) with initial mint oil contents of 10%, 30% and 40% were prepared. Water was added to each mixture, stepwise, with agitation and stirring. The conductivity of the each dispersion was recorded as a function of water content (DDS-307 conductivity metre, Rex Instruments Factory, Shanghai, China and a DDS-307 platinum conductance electrode). Temperature was held constant at  $25 \pm 0.1$  °C. At the endpoint, each sample was a single phase that was optically transparent.

### 2.5. Particle size distribution measurement of mint oil ME

MEs with MO/AOT and Brij35/ethanol/water (AOT:Brij35 = 1:1, S:SC = 1:1, mint oil:S/CS = 3:7, w/w) and MO/AOT and CrEL/ethanol/water (AOT:CrEL = 1:1, S:SC = 1:1, mint oil:S and CS = 3:7, w/w) were prepared by dropwise addition of S/CS at processing temperatures of 30 and 60 °C. The particle size distributions of the resulting MEs were measured with a Nano-Zs90 (Malvern Instruments, Worcestershire, UK), which is based on the technique of laser Doppler electrophoresis. The electrophoresis mobility,  $U_E$ , was measured and the zeta potential  $\zeta$  was calculated by the Henry equation (Deshiikan & Papadopoulos, 1998)  $U_E = (2\varepsilon\zeta/3\eta)f(k_a)$ , where  $\varepsilon$ ,  $\eta$ ,  $f(k_a)$  were the dielectric permittivity of the solvent, viscosity of the solution, and Henry's function. The value of  $f(k_a)$  here was determined to be 1.5 according to Smoluchowski approximation. The parameters for the measurement were as follows: Ar laser, wavelength 488 nm, detector angle 90°, dispersant viscosity 15.514 mPa s at 25 °C, refractive index 1.362. The temperature of the scattering cell was controlled at 25 °C. The droplet size (according to the particle number distribution) was taken as the mean value of three measurements.

### 2.6. Encapsulation efficiency measurement of mint oil ME by HS-GC assay

Encapsulation efficiency of three ME types was measured by a HS-GC method, using an HP-7694 Automatic Headspace Sampler and Model HP-6890 capillary gas chromatograph equipped with a thermal conductivity detector (Agilent Technologies, Palo Alto, CA, USA) and capillary column with an I.D. = 0.32 mm and a length of 30 m (model HP-1,19091z-413, J&W Scientific, Folsom, CA, USA).

Headspace Sampler operating conditions were as follows: oven temperature of 60 °C; vial pressurised with helium; pressurisation time of 0.2 min; sample-loop fill time of 0.05 min; loop equilibration time of 0.05 min; loop fill time of 0.1 min; MHE recycle time of 1 min.

GC operating conditions were as follows: the column temperature was maintained at 60 °C for 3 min after the injection, and then programmed at 4 °C/min up to 140 °C. The analysis was done with a split/splitless injector (split ratio 1:10) at 230 °C. The carrier gas was helium at a constant flow rate of 1.3 ml/min.

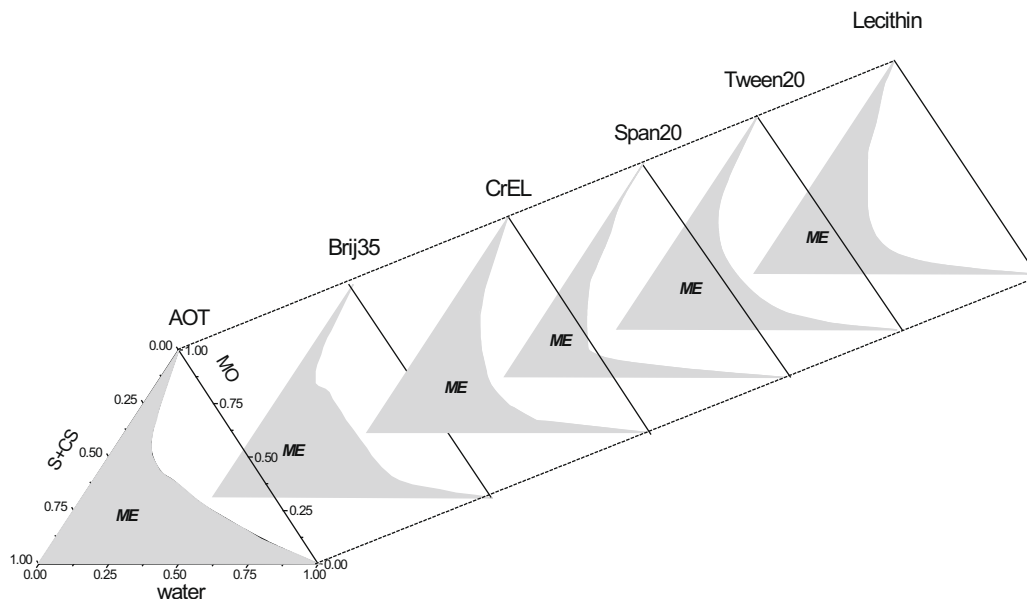
## 3. Results and discussion

### 3.1. Influence of surfactant type on the formation of MO-ME

Factors, which effect the formation of an O/W ME, include the type of surfactant (S), co-surfactant (CS), the ratio of S/CS, the order of dispersion or mixing, and the temperature (Liu, Ma, Cheng, & Zhao, 1998). These factors can affect the spontaneous curvature and the elasticity (or rigidity) of the interfacial film formed by the surfactant system (Leung & Shah, 1987).

The phase diagrams of MO/surfactant/ethanol/water with six single surfactants were measured at 30 °C, (Fig. 1). The transparent isotropic ME region corresponded to the area of mutual solubility of the MO/surfactant/ethanol/water components. The areas for the transparent regions were 59.3%, 48.5%, 45.4%, 26.8%, 40.4% and 41.7% of the total phase diagram for the surfactants AOT, Brij35, CrEL, Span20, Tween 20 and lecithin, respectively. The results indicated that the MO-ME forming abilities of AOT, Brij35 and CrEL were greater than that of the other three surfactants. AOT, Brij35 and CrEL were used for making three binary mixtures for additional experiments.

Three binary mixtures (1:1, w/w), from the possible combinations of AOT, Brij35 and CrEL, were used at the same concentration



**Fig. 1.** Pseudoternary phase diagrams of microemulsions with mint oil (MO)/single surfactant(S)/ethanol(co-surfactant-CS)/water at 30 °C with a constant weight ratio of S:CS = 1:1. Lecithin, Tween 20, Span20, Brij35, CrEL and AOT were employed as single surfactants. The grey areas represent the microemulsion phases.

as in the single surfactant MO-ME dispersions experiments. When the ionic surfactant AOT was mixed with either of the nonionic surfactant, Brij35 or CrEL, the ME phase areas increased (69.7% and 65.8%, respectively), (Fig. 2), as compared to ME phase areas formed with either of the single surfactants, AOT, Brij35, or CrEL, (Fig. 1). But the ME phase area decreased with the mixture of the two nonionic surfactants, CrEL and Brij35, compared with either of the single surfactants. The same trend for a nonionic and ionic surfactant mixture was found by Mitra and Paul (2005). However, our results with the mixture of two nonionic surfactants were different from those of another report (Li et al., 2005), in which a mixture of two nonionic surfactants showed the opposite trend. However, the overall trend was still consistent and the same as that in another report (Bidyut & Rajib, 2005) with comparisons amongst mixed ionic-nonionic or nonionic-nonionic surfactants that were used to disperse water in different oils. They found that ionic-nonionic mixed surfactants were more likely to synergize and increase interfacial rigidity.

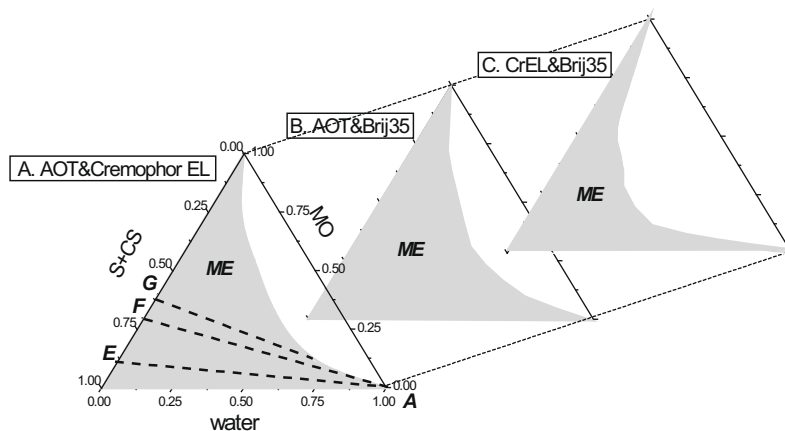
Since the ME phase area for the combination of AOT and CrEL was slightly higher than that of AOT and Brij35, it was used as

the surfactant for further studies. The effect of the AOT:CrEL ratio on the formation of MO-ME was also investigated. When the AOT:CrEL ratio was increased from 1:2 to 1:1 to 2:1, no significant difference was found amongst the ME phase areas (Table 1).

In contrast, the impact of the S:CS ratio on the formation of the ME phase area was more dramatic. The phase diagrams with AOT and CrEL(1:1)/ethanol at three different S:CS ratios (1:1, 1:2, and 1:4) were measured (Table 1). In these dispersions, the extent of the ME phase area decreased from 68.6% to 8.5% with the increasing co-surfactant content. The largest ME phase area was obtained at S:CS = 1:1. This result was in accordance with a system of eucalyptus oil/AOT and Brij35/butanol/water studied by Rajib and Bidyut (2005).

### 3.2. Influence of processing parameters on the formation of MO-ME

The influence of mixing order of components on the formation of AOT and CrEL/ethanol/MO/water ME was investigated, (Table 1). The ME phase areas were 68.2%, 68.1% or 67.9% when water, S/CS, or mint oil, respectively, was the last component added to



**Fig. 2.** Pseudoternary phase diagrams for the systems MO (mint oil)/AOT and Brij35(S) (1:1)/CS/water, MO/AOT and CrEL/CS/water and MO/Brij35 and CrEL/CS/water at 30 °C. Ethanol was used as the co-surfactant. The ratio of surfactants (S) in the mixtures was 1:1, and the S:CS was 1:1. The grey areas represent microemulsion phases. All ratios were by weight.

**Table 1**  
The effect of various factors on the microemulsion (ME) phase area of dispersions of mint oil and water with a surfactant mixture (S) of AOT and CrEL and ethanol as a co-surfactant (CS).

Surfactant and co-surfactant ratios (by weight)		Last component added	Temperature (°C)	ME phase area (%)
AOT:CrEL	S:CS			
1:2	1:1	Water	30	67.9
1:1	1:1	Water	30	68.1
2:1	1:1	Water	30	68.1
1:1	1:1	Water	30	68.6
1:1	1:2	Water	30	57.3
1:1	1:4	Water	30	8.5
1:1	1:1	Water	30	68.2
1:1	1:1	S/CS	30	68.1
1:1	1:1	MO	30	67.9
1:1	1:1	Water	20	67.4
1:1	1:1	Water	40	68.0
1:1	1:1	Water	60	67.9

the formulation (AOT:CrEL = 1, S:CS = 1). The results indicated the independence of MO-ME phase area formation with regard to the addition sequence of the components. The S/CS addition was used for the remaining experiments, as it was easier to detect the critical point with this addition order.

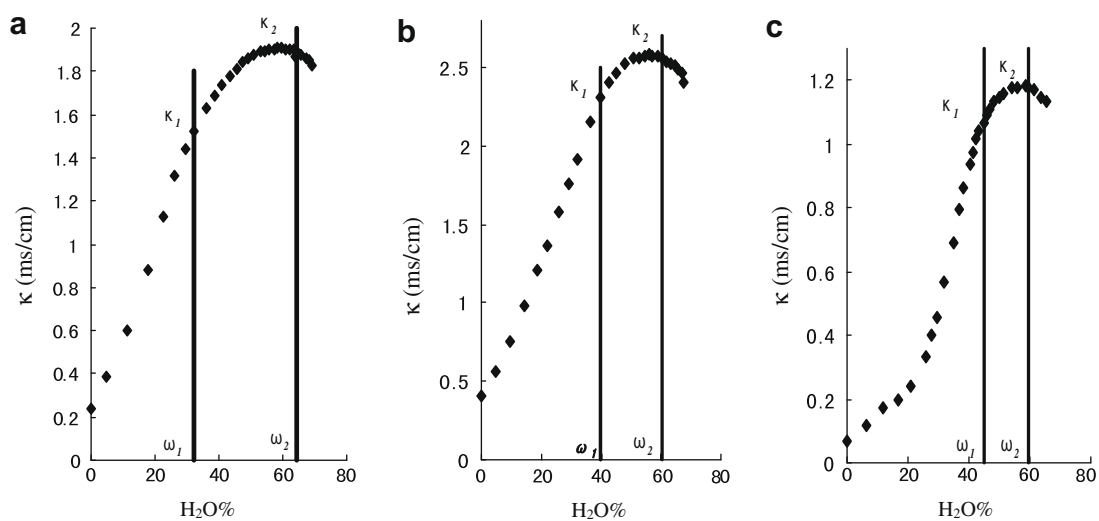
The phase behaviours of the system with AOT and CrEL (1:1), ethanol (S:CS = 1:1), MO and water, were studied at three temperatures, 20, 40, and 60 °C. The ME phase area did not vary much over this temperature range (Table 1). It has been reported that the temperature sensitivity of the magnitude of the ME phase area is related to the type of surfactant used. Karlström (1985) indicated that temperature played a significant role in formation of MEs when stabilized by nonionic surfactants. The same conclusion was reached by Kunieda, Nakano, and Pes (1995). The hydrophile-lipophile balance of nonionic surfactants was strongly dependent on temperature because of the dehydration of the oxyethylene group. This made the nonionic surfactants more lipophilic at higher temperatures. On the other hand, for ionic surfactants, Aveyard, Binks, and Fletcher (1989) reported that the effect of temperature was less pronounced, and the dissociation of the ionic group increased and ionic surfactants became more hydrophilic at elevated temperatures. The surfactant used in our experiment was a mixture of an ionic surfactant (AOT) and nonionic surfactants (CrEL). The insensitivity of the size of ME phase area to the temperature of formation suggested either a dominance of the io-

nic surfactant over the nonionic surfactant in the binary mixture or the compensation of opposite hydrophilic-hydrophobic changing trends between two surfactants with temperature. Similar results were reported for a binary surfactant mixture of ionic (AOT) and nonionic (dodecylpentaoxyethylene glycol ether, C<sub>12</sub>E<sub>5</sub>) surfactants which produced a ME phase area whose size was temperature-insensitive (Binks, Fletcher, & Taylor, 1997). Kunieda, Hanno, Yamaguchi, and Shinoda (1985) and Aramaki, Ozawa, and Kunieda (1997) also reported the same trend for the binary surfactant mixtures of AOT and SDS/C<sub>12</sub>EO<sub>n</sub> (where n = 2, 3, 6)/brine in hydrocarbon oils.

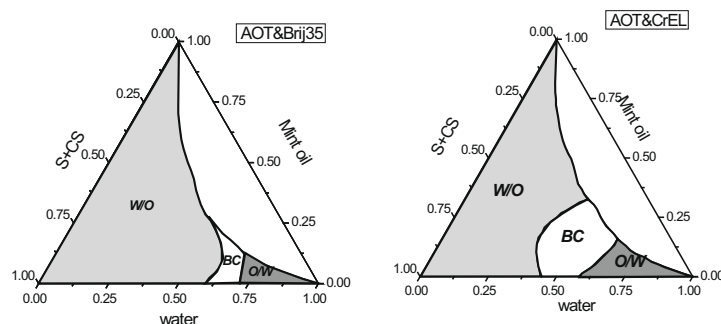
### 3.3. Electrical conductivity and phase transitions of the MEs

Within the ME phase area, there were three possible ME structures, O/W, W/O and bi-continuous (BC) structures, which had been thought to exist (Mehta & Bala, 2000; Zheng, Minamikawa, Harada, & Inoue, 2003). Even though there were no visible differences between these regions, the boundaries were detected by a change in electrical conductivity.

When MEs were prepared with MO (10%, 30% or 40%)/AOT and CrEL/ethanol and then diluted with water at concentrations along the lines EA, FA and GB (Fig. 2), changes in the conductivity were observed (Fig. 3). These patterns were similar to those in the typical conductivity curves described by Fang and Raymond (1978). As



**Fig. 3.** Variation of the electrical conductivity ( $\kappa$ ) in the ME phase as a function of water content; (a) along line EA in the microemulsion region of Fig. 2a; (b) along line FA in the microemulsion region of Fig. 2a; (c) along line GB in the microemulsion region of Fig. 2a. The two vertical lines in each graph indicate the boundaries between W/O and bi-continuous and bi-continuous and O/W regions within the microemulsion phase of Fig. 2a.



**Fig. 4.** Pseudoternary phase diagrams for the systems mint oil/AOT and Brij35(S) (1:1)/ethanol/water and mint oil/AOT and CrEL (1:1)/ethanol/water with a S:CS of 1:1 at 30 °C. All the ratios are by weight. The grey, white and light grey areas represent O/W, bi-continuous and W/O microemulsion regions, respectively. All ratios were by weight.

the water content increased, the conductance increased sharply until reaching a plateau. This pattern has been related to a transition from W/O to bi-continuous to O/W microstructure with the addition of water in a mixed system (Lagues, Dvolaitzky, Le Pesant, & Ober, 1980; Lv, Zheng, & Tung, 2005).

The water content at the two boundaries between three ME regions was indicated by specific changes in electrical conductivity curves, (Fig. 3). The results suggested that the W/O region of the MEs had water contents of up to 32–45% and that the O/W region of the MEs had water contents larger than 60–65%. With the data from the conductivity measurements, the boundaries between the three regions, for each of the two systems MO/AOT + Brij35/ethanol/water and MO/AOT + CrEL/ethanol/water, were determined (Fig. 4). The diffusion of dye (Sudan IV and Eosin blue) in various regions of the MEs confirmed the presence of three regions within the ME phase, as determined by conductivity measurements (pictures not shown). It was also observed (Fig. 4), that the O/W ME region of AOT and CrEL was larger than that of AOT and Brij35; this suggested that surfactant AOT and CrEL was more efficient in entrapping mint oil. That is this O/W ME was more stable under water dilution and thus more flexible for applications.

#### 3.4. Particle size distribution measurement of mint oil ME

Particle size distribution measurements of selected samples in the O/W region of the ME phase was determined by dynamic light scattering (DLS). Particle size distributions, which were typical for single domain and narrow distributed dispersions, were observed for the O/W regions of the MEs. Measurements were made on samples from the O/W regions of MEs prepared with three single surfactants (AOT, CrEL, and Brij35) and two binary mixtures (AOT and Brij35 and AOT and CrEL, 1:1). The measurements were made with samples with a MO/surfactant/ethanol/water system, at water content of 60% and MO:S/CS = 3:7. The effects of sample composition and temperature on the “lipodynamic” diameter DL and dispersibility of the reverse micelles in O/W ME on the same dilution line (MO:S/CS = 3:7) were measured, (Table 2). The results indicated that the droplets size of the O/W of MEs formulated with either the single surfactants or the binary mixtures were in the range of nano-dispersions. The particle size of the O/W particle within the

ME phase decreased from 45.0 to 20.0 nm when using a mixed surfactant of AOT and CrEL instead of AOT and Brij35 or any of the single surfactants. This result was in accordance with the results from phase diagrams, which indicated that AOT and CrEL was more effective than were AOT and Brij35 with regard to the formation of O/W MEs.

No remarkable difference in the particle sizes for MO/AOT and CrEL/ethanol/water ME was observed as the processing temperature changed from 30 to 60 °C. This result also supported the earlier observation that the MO/AOT and CrEL/ethanol/water ME was temperature-insensitive.

#### 3.5. Encapsulation efficiency measurement of mint oil ME with HS-GC

There have been few reports on how to estimate the encapsulation efficiency of flavouring oils within a ME. Methods of membrane dialysis and ultracentrifugation have been used to evaluate controlled release of some drugs encapsulated by MEs (Kang et al., 2004; Podlogar, Bester Rogă, & Găşperlin, 2005), but the method of membrane dialysis is limited to macromolecule encapsulates. It was difficult to find a solvent to remove the residual mint oil and not destroy the MO-ME. In this case, however, the volatility of mint oil could be used to estimate the effectiveness of the ME. The initial amount of the aroma compound, minus the amount of it in the air after an air–liquid equilibration in a closed system, could represent encapsulation efficiency of the ME. Thus, the feasibility of using HS-GC to evaluate the ability of ME to encapsulate MO was investigated.

The advantage of using O/W ME for delivery of essential oils is the possibility that it could retard the volatilisation, oxidation, and degradation of the essential oil during processing and storage. Therefore, the degree of volatilisation could be used as an index of the encapsulation efficiency of a MO-ME. MO is a complex mixture, but its major component is menthol. Menthol was selected as an index for quantitative analysis. The equilibration of the headspace specimens required a balance between several factors. Pre-tests showed that a headspace menthol equilibrium concentration in the headspace was attained after 30 min at 60 °C and the area of the menthol peak increased linearly with the volume of MO increasing from 0 to 2 µl. A further increase in

**Table 2**

Mean lipodynamic diameter of O/W ME droplets in a MO/surfactant (S)/co-surfactant (CS)/water dispersion on the same dilution line MO:S/CS (3:7) with a C:SC of 1:1 and at a different processing temperature ( $n = 3$ ). The co-surfactant was ethanol.

	Surfactant					
	AOT (30 °C)	CrEL (30 °C)	Brij35 (30 °C)	AOT and Brij35 (30 °C)	AOT and CrEL (30 °C)	AOT and CrEL (60 °C)
Mean diameter (nm)	47.2 ± 0.2	55.7 ± 0.3	67.0 ± 0.2	45.0 ± 0.3	20.0 ± 0.1	20.0 ± 0.1



**Table 3**

Encapsulation efficiency within various regions (O/W, bi-continuous (BC) and W/O) of a ME analysed by head space-GC. ( $n = 5$ ).

ME phase	Temperature (°C) <sup>a</sup>	Peak area <sup>b</sup>	Encapsulation efficiency (%) <sup>c</sup>
O/W	30	61.85 ± 1.45	79.36 ± 0.48
BC	30	146.65 ± 2.95	51.07 ± 0.50
W/O	30	252.3 ± 2.80	15.82 ± 0.85
O/W	60	65.15 ± 0.65	78.26 ± 0.21

<sup>a</sup> Temperature of processing or formation of the ME.

<sup>b</sup> Peak area on menthol measured by HS-GS analysis.

<sup>c</sup> Encapsulation efficiency (%) =  $(1 - A/A_0) \times 100\%$ , where  $A_0 = 299.70 \pm 5.60$  which was the peak area of menthol in a dispersion of water and 2  $\mu\text{l}$  of MO (30 °C). A was the peak area of menthol in the respective phase of a MO-ME containing 2  $\mu\text{l}$  of MO.

MO volume resulted in smaller increases of the menthol peak area (result not shown).

The encapsulation efficiency of three MO-ME types containing 2  $\mu\text{l}$  of MO is shown in Table 3, which indicated that encapsulation efficiency decreased with the transition from O/W to BC to W/O structures, at a processing temperature of 30 °C, from 79.4% to 51.1% to 15.8%, respectively. The results confirmed that the O/W region of the ME was more effective in reducing the degree of volatilisation of MO. It can also be seen that the temperature of the processing had little effect on encapsulation efficiency of MO-ME between 30 and 60 °C, (Table 3).

#### 4. Conclusion

The formation of a ME with MO as the oil phase was highly dependent on surfactant types and composition. It was found that the mixed surfactants of AOT and CrEL enhanced the ME formation and decreased the amount of surfactant required compared to the other mixed surfactants. This was thought to be due to differences in the structures and mutual solubility properties of the two individual surfactants.

It was noteworthy that processing parameters, including mixing order and temperature, had little effect on the formation of a MO/AOT and CrEL/ethanol/water ME. These results suggested that a MO-ME formed with AOT and CrEL was temperature-insensitive and could be prepared by random order of addition of ingredients.

Characterisation of MO-ME, using HS-GC assay, indicated that a ME of MO/AOT and CrEL/ethanol/water (AOT:CrEL = 1, S:CS = 1, H<sub>2</sub>O%  $\geq$  64.5%) retarded the volatilisation of MO, with only 22% of the menthol evaporating after exposure to air at 60 °C for 30 min as compared to the same volume of un-encapsulated mint oil. The results from the HS-GC studies suggested that this technique could be used to evaluate the encapsulation efficiency of ME systems with volatile components.

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