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ORIGINAL RESEARCH PAPER

Investigation of the effect of different parameters on the phase inversion temperature O/W nanoemulsions

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ABSTRACT

Objective(s): Nanoemulsions are a kind of emulsions that can be transparent, translucent (size range 50-200 nm) or "milky" (up to 500 nm). Nanoemulsions are adequatly effective for transfer of active component through skin which facilitate the entrance of the active component . The transparent nature of the system and lack of the thickener and fluidity are among advantages of nanoemulsion.

Materials and Methods: In this study, a nanoemulsion of lemon oil in water was prepared by the phase inversion temperature (PIT) emulsification method in which the tween 40 was used as surfactant. The effect of concentration of NaCl in aqueous phase, pH and weight percent of surfactant and aqueous on the PIT and droplet size were investigated.

Results: The results showed that with increasing of concentration of NaCl from 0.05 M to 1 M, PIT decrease from 72 to 50. The average droplet sizes, for 0.1, 0.5 and 1 M of NaCl in 25 ºC are 497.3, 308.1 and 189.9 nm, respectively and the polydispersity indexes are 0.348, 0.334 and 0.307, respectively.

Conclusion: Considering the characteristics of nanoemulsions such as being transparent, endurance of solution and droplet size can provide suitable reaction environment for polymerization process used in making hygienic and medical materials.

Keywords: *Nanoemulsion, Phase inversion temperature, Surfactant*

INTRODUCTION

Nanoemulsions are those emulsions having nano-scale droplets (often in the range of 50-500 nm) [1-3] which fall into the 5-200 nm transparent or semi-transparent range and they are milky opalescent up to 500 nm [4-5]. Unlike the micro-emulsions which are transparent and thermodynamically stable, nanoemulsions are synthetically stable. However, longstanding physical stability of nanoemulsions (without coagulation or accumulation of droplets) has made them as seamless such that some researchers considers them similar to thermodynamic stability [6-8]. Nanoemulsions have various industrial applications; for example they have been used to make the reaction medium for polymerization and to make cosmetic ingredients and health materials and chemicals used in the agriculture [9]. Due to presence on non-equilibrium systems, external energy requires to form the nanoemulsions [10-11]. Two main methods to prepare nanoemulsions are the dispersion or high-energy methods and condensation or low-energy methods. In dispersion or high-energy methods, the input energy was supplied by high power agitators along with ultrasonic homogenizers. However, in order to prepare the small droplets size nanoemulsions, much energy is needed. This preparation method is not possible for industrial application. Condensation or low-energy methods utilizes the resulted phase transitions during emulsification process due to changing the spontaneous curvature of the surfactant. This change of curvature could be done through two ways: The composition has been held constant while the temperature changes (the Phase Inversion Temperature method (PIT)) or the temperature has been held constant and the composition has been changed (Emulsion Inversion Point method (EIP)). Preparations of the non-ionic surfactants-stabilized nanoemulsions have been extensively reported through dispersion and condensation methods. In low-energy methods for oil-in-water nanoemulsions (O/W), the required change of curvature for preparing the nanoemulsions is possible

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through three methods. Firstly through changing the hydration grade of polyoxyethylene chains in non-ionic polyoxyethylene surfactants, secondly through changing the temperature (PIT method) and thirdly through changing the water content (EIP) [12-14]. PIT is determined through several methods. First method is a visual method in which the emulsion is watery and diluted above inversion temperature whereas the emulsion is white, concentrated and turbid below that temperature.

The second method is carried out by conductometry. The third method is done by pH. The pH is unstable above inversion temperature but it is completely fixed below that temperature. In fact, we measure the oil pH above inversion temperature and in order to measure the pH, we must have ionic force and conductivity while the oil does not have any of them [10]. In this paper, lime oil-in-water nanoemulsions were prepared by the PIT procedure. Tween 40 was utilized as surfactant and the effective parameters such as NaCl concentration in aqueous phase, pH and weight percentage of the surfactant and aqueous phase were studied on the particles size and PIT [15-18].

MATERIALS AND METHODS

Tween 40 as surfactant and the required NaCl salt were purchased from Merck company. Lime oil as oil phase and double-distilled water were used for preparation of NaCl aqueous solutions. In order to study the effect of changing concentration on the PIT, the hydrophilic-lipophilic balance temperature was determined using electric conduction procedure by conductometer. Emulsions with of 20% (W/V) oil, 7.5% (W/V) surfactant and 72.5% (W/V) NaCl solution were prepared in various concentrations and through hand shaking at room temperature. Emulsions were gradually heated and the conduction were measured as function of temperature. Hydrophilic-lipophilic balance temperature (HLB) was considered as average maximum temperature and minimum conduction was determined as PIT. In order to study the effect of changing weight percentage of surfactant and electrolyte solution on the PIT, NaCl (0.8 M) solution was prepared while weight percentage of oil was held fixed at 20% (W/V with varying weight percentage of surfactant and electrolyte solution (Table 1). Having prepared all of emulsions, the conduction was measured as function of temperature.

Table 1. Surfactant and electrolyte percentages in samples (Weight percentage of oil was held fixed on 20%)

Sample	Surfactant (%)	NaCl (%)
1	5	75
2	7.5	72.5
3	10	70
4	12.5	67.5
5	15	65
6	17.5	62.5

Emulsification by the PIT method

In order to prepare the oil-in-water nanoemulsions, initially the oil phase comprising the surfactant and aqueous phase comprising the various concentrations of NaCl were separately heated up to 15 ! above the PIT. Then, the aqueous phase was poured into the oil phase in that temperature and the mixture was removed from heating source and cooled down to PIT temperature through hand shaking. Then, the sample was cooled through being placed in the ice bath below 20 ^{o}C while hand shaking.

Finally, droplet size of the selected samples and their dispersion index were measured by nano-ZS device. The transmittance percentage of prepared nanoemulsions were measured by the spectrophotometer UV-Vis (Model UV-1600) until use.

RESULTS AND DISCUSSION

Phase inversion temperature values for nanoemulsions comprising 20% (W/V) oil, 7.5% (WV) surfactant and 72.5% (W/V) NaCl solution with 0.5, 0.1, 0.25, 0.5, 0.8 and 1 M concentration were obtained by conductometry measurements. HLB temperature or PIT were considered as average maximum temperature and minimum condition.

The results of conductometry for concentration changes have been shown in Figure 1 and the results of PIT have been listed in Table 2.

According to Table 2, gradual decrease in PIT from 72 to 50 was observed when the concentration of NaCl solution increased from 0.05 to 1 M.

Increasing the concentration of electrolyte causes to electrically prevent the water and increase the internal pressure of the solution. So water and non-ionic surfactant interaction was weaken and the nonionic surfactant become more lipophilic due to rise of electrolyte concentration. According to Table 3, it is evident that in the pH range from 5 to 7, PIT temperature is maximum.

As shown in Table 4, it could be suggested that significant changes in PIT temperature was not generated while changing the weight percentage.

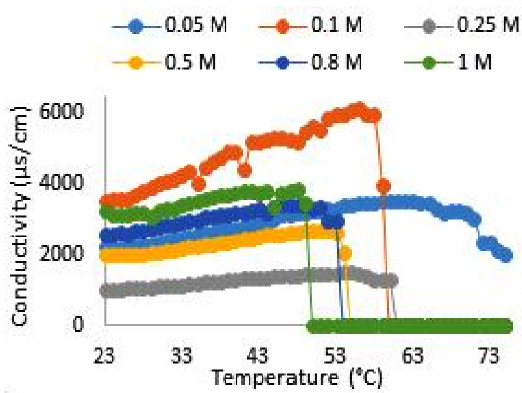

Fig. 1. The conduction chart by temperature related to nanoemulsions having various concentrations of NaCl

Table 2. PIT temperatures in the various concentration of NaCl

NaCl concentrations in aqueous phase (M)	0.05	0.1	0.25	0.5	0.8	1
PIT (°C)	72	59	58	54	52	50

Table 3. PIT temperatures in the various pHs

pH	3	4	5	7	8	9
PIT (°C)	65	67	72	71	70	60

Table 4. PIT temperatures in the various weight percentages

Sample	1	2	3	4	5	6
PIT (°C)	69	52	81	82	78	73

PIT in emulsions exactly reflects on changing HLB of emulsifiers due to additives which provide clear information about the effect of quantity and types of additives.

Various processes of emulsification based on the PIT emulsification were studied to identify the most effective way for studied systems.

One process which produced more transparent nanoemulsions with highest transmittance was emulsification at 15 °C above the PIT which has been shown in Table 2 for those systems with various concentrations of NaCl in aqueous phase and consequently cooling down the emulsion until PIT temperature and immediate cooling down it below 20°C (Table 5).

The resulted emulsions have transparent and semi-transparent appearance and the droplets size were in the range of nanoemulsions (Figure 2).

Fig. 2. The prepared nanoemulsions (right : nanoemulsions with NaCl 0.1) M and left: nanoemulsions with NaCl 0.5 M

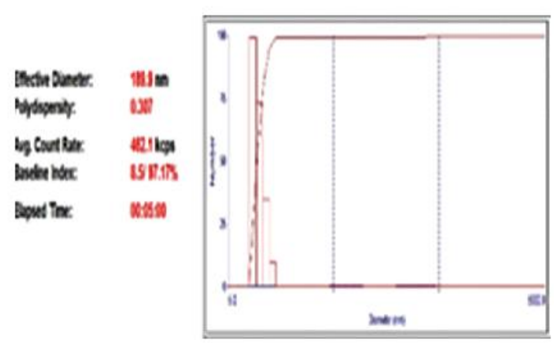

Fig. 3. The chart of nano-ZS for a sample of nanoemulsion (weight percentage: 20% oil, 7.5% surfactant and 72.5% NaCl 1 M

Table 5. Transmittance percentages at different temperatures and concentrations

T(^{0}C)	Concentration (M)				
	0.1	0.25	0.5	0.8	1
14	19.5%	0.9%	7.3%	2.8%	0.6%
19	16.7%	0.8%	5.4%	2.1%	0.5%
34	20.1%	1.2%	9.5%	3.8%	0.9%
37	18.6%	1.2%	8.5%	3%	0.9%
50	14.7%	1%	6.4%	6.5%	0.8%

CONCLUSION

In this paper, lime oil nanoemulsions were prepared in water using PIT method. Tween 40 was used as the surfactant and the effect of major parameters in the aqueous phase including pH and weight percentage of surfactant and aqueous phase were studied on the PIT and particle size. The results showed that average particle size for 0.1, 0.5 and 1 M NaCl equal to 497.3, 308.1 and 189.9 nm in 25^{o}C, respectively and dispersion index of 0.348, 0.334 and 0.307, respectively. The prepared nanoemulsions could be considered as suitable reaction medium for polymerization of cosmetics and pharmaceuticals according to transparency, solution stability and droplet size.

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