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Ultrasound assisted manufacturing of paraffin wax nanoemulsions: Process optimization

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ABSTRACT

This work reports on the process optimization of ultrasound-assisted, paraffin wax in water nanoemulsions, stabilized by modified sodium dodecyl sulfate (SDS). This work focuses on the optimization of major emulsification process variables including sonication time, applied power and surfactant concentration. The effects of these variables were investigated on the basis of mean droplet diameter and stability of the prepared emulsion. It was found that the stable emulsion with droplet diameters about 160.9 nm could be formed with the surfactant concentration of 10 mg/ml and treated at 40% of applied power (power density: 0.61 W/ml) for 15 min. Scanning electron microscopy (SEM) was used to study the morphology of the emulsion droplets. The droplets were solid at room temperature, showing bright spots under polarized light and a spherical shape under SEM. The electrophoretic properties of emulsion droplets showed a negative zeta potential due to the adsorption of head sulfate groups of the SDS surfactant. For the sake of comparison, paraffin wax emulsion was prepared via emulsion inversion point method and was checked its intrinsic stability. Visually, it was found that the emulsion get separated/creamed within 30 min. while the emulsion prepared via ultrasonically is stable for more than 3 months. From this study, it was found that the ultrasound-assisted emulsification process could be successfully used for the preparation of stable paraffin wax nanoemulsions.

1. Introduction

Nanoemulsions are metastable colloidal dispersion of immiscible aqueous and oil phase with droplet diameter in the range of 20–200 nm [1–3]. The nano sized droplet makes emulsion more flow able, less turbid or may be transparent and have unique rheological properties [4,5]. Their very small droplet diameter and large surface area makes nanoemulsion more stable against sedimentation and creaming for a long time [6]. There are two ways for the preparation of nanoemulsion, one with high-energy dissipation and another with low-energy dissipation approaches [1]. High energy approach is a mechanical method while low energy is chemical-based method for nanoemulsions preparation. Mechanical method include micro fluidization [7,8], high pressure homogenization [9,10] and ultrasound homogenization [1,11–13]. Low-energy methods make use of the chemical potential of the emulsion components [14].

Currently, ultrasonic cavitation assisted production of nanoemulsion has gained the interest mainly because of its energy-efficiency, low production cost, ease of system manipulation and better control over the formulation variables [1,2,13–18]. The ultrasonic irradiation causes acoustic cavitation, which result into the formation and subsequent collapse of microbubbles by the pressure fluctuations of a simple sound wave. The collapse of these voids creates high localized turbulence. This turbulence is used very effectively for disruption of dispersed oil into droplets of sub-micron size [19]. The final droplet size and dispersity of nanoemulsion droplets are influenced by a number of process and formulation variables [1]. Whereas, the type and concentration of surfactant will have an effect on the process of disruption of larger oil drops into nanosize droplets as well as the stability of the emulsion. The main function of surfactant is to reduce the interfacial tension so that the droplet disruptions occur at minimum energy and then adsorption onto the newly prepared droplets surface to prevent the droplet re-coalescence [20,21].

A lot of studies have been carried out on the oil in water nanoemulsion with liquid oil as dispersed phase, very few work have been done on the nano-emulsion with dispersed crystal phase at room temperature [22]. Since the dispersed phase becomes solid at room temperature, these nanoemulsions are very difficult to formulate and also very unstable in nature. It was found that no work have been reported on systematic ultrasonic cavitation assisted formulation of paraffin wax based nanoemulsion and process

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optimization. Paraffin wax is used as a phase change material (PCM) [23,24], it is hydrophobic in nature so used it is as a water repellent material [25]. Recently Janus colloidal particles have been synthesized using paraffin wax/water emulsion [26–28].

In the present work, we have formulated paraffin wax-in-water nanoemulsions using a combination of ultrasonic cavitation and low-energy emulsion inversion point (EIP) method. Aim of this work is to study the effects of major ultrasonic process-related parameters including ultrasonic power and sonication time, and formulation-related parameter i.e., surfactant concentrations on mean droplet diameter (MDD), zeta potential, viscosity, pH and stability of the emulsion.

2. Materials and methods

2.1. Chemical reagents

The paraffin wax was purchased from SD Fine Chemicals Ltd. Mumbai, India. Sodium lauryl sulfate (SDS) was obtained from SD Fine Chemicals Ltd. Mumbai Distilled water prepared using Millipore apparatus was used during all the experimental runs. All reagents were used as received without further purification.

2.2. Preparation of nanoemulsions

2.2.1. Ultrasonic emulsification

Nanoemulsion was formulated using paraffin wax as an oil phase, anionic SDS surfactant and distilled water. Coarse emulsion were prepared by homogenizing the mixture of surfactant (at various concentrations: 0, 0.01, 0.1, 0.5, 1, 5, 10 mg/ml of emulsion) and water, followed by the drop wise addition of paraffin wax keeping 0.2 oil-phase volume fraction (ϕ) using magnetic stirrer (Model RQ1210, Remi Metals Gujarat Ltd., India) at 1000 rpm. Initially the mixture of surfactant and water solution heated up to 65–70 °C then the molten paraffin wax was added drop wise into it with vigorous magnetic stirring. After complete addition of the required quantity of paraffin wax, the prepared coarse emulsion was subjected to ultrasonic irradiation using a 20 kHz Sonicator (Sonics and Materials, USA) with a maximum power output of 750 W. The sonication processes were carried out for different irradiation time and at different power density (W/ml). The formulated nanoemulsion was characterized and also the stability of the emulsion was investigated. All the characterization process was done at room temperature.

2.2.2. Emulsion inversion point method

Emulsions were prepared by drop wise addition (approximately 1.0 mL/min) of mixture of surfactant (1 gm/10 mg/ml of emulsion) and water into the paraffin wax ($\phi = 0.2$) with vigorous agitation (at 2000 rpm) using a magnetic stirrer (Model RQ1210, Remi Metals Gujarat Ltd., India) at a temperature of at 65–70 °C for 12 h.

2.3. Characterization of nanoemulsion

2.3.1. Mean droplet diameter (MDD) and zeta potential

MDD and zeta potential of the emulsion droplets were determined, using Zetasizer Nano ZS (Malvern Instruments, UK) equipped with dynamic light scattering (DLS) technology. Samples were diluted (1:100) prior to the measurement studies in order to ensure the free Brownian motion of the droplets. Samples were equilibrated at 25 °C for 1 min. The surface charge of the nanoemulsion droplets was determined by measuring the electrophoretic mobility at 25 °C and values of zeta potential were expressed in mV.

2.3.2. Emulsion morphology

Scanning electron microscopy (SEM) (Quanta 200 ESEM) was also used to observe the surface morphology of nanoemulsion. Initially, the nanoemulsion were diluted with water and then drop coated onto the sample stub. Prior to imaging, a thin layer of gold sputtering was done to prevent surface charging in the electron beam.

2.3.3. Physicochemical characteristics

The FTIR spectrum of the bulk paraffin wax and emulsion were recorded using Shimadzu FTIR spectrophotometer using ATR mode of operation and scanning of the FTIR spectrophotometer was carried out from 4000 to 600 cm^{-1}. The thermal behavior of the bulk paraffin wax and emulsion was characterized using Differential Scanning Calorimetry, Shimadzu DSC 60. Brookfield viscometer (model:DV-E) was used to measure the viscosity of the emulsion. pH of the emulsion were recorded using a pH meter (Eutech Instruments, Singapore) at room temperature. Conductivity of the emulsion were measured using conductivity meter (EQUIPTRONICS instruments).

2.4. Stability of emulsion

The stability of formulated nanoemulsion was measured after 2 weeks. The creaming of the nanoemulsion was assessed as follows:

$$f(\%) = \left(\frac{h_e}{h_t}\right) \times 100 \tag{1}$$

where $f(\%)$ represents the fraction of emulsion phase, h_e represents the height of emulsion phase, h_t represents the total height of the emulsion system (including the height of emulsion phase and the height of the separated water). Intrinsic stability of nanoemulsion was studied by storing the emulsion at room temperature for two months and subsequent increase in droplet diameter was recorded.

3. Result and discussion

3.1. Effects of surfactant concentration on paraffin wax emulsion

To study the effect of surfactant concentration on the physicochemical properties of paraffin emulsion, the emulsion were prepared with varying concentrations of surfactant. Table 1 shows the surfactant concentration and physico-chemical properties of the paraffin wax emulsion. The MDD of the paraffin wax emulsion was found to be reduced as the surfactant concentration increases, due to an increase in the ratio of surfactant film thickness to droplet radius [29]. The thicker surfactant film can provide better steric stabilization against flocculation, resulting into lowering of the impact of flocculation on the instability process. Also, higher surfactant concentration can increase in the interfacial area plus

Table 1
Effect of surfactant concentration on physico-chemical properties of paraffin emulsion formulated via US.

SDS (mg/ml of emulsion)	Fraction of emulsion phase (f) (%)	Mean droplet diameter (nm)	Zeta potential (mV)	Viscosity (cP)	pH
0.00	46	623.1	−38.1	1.48	5.82
0.01	47	614.1	−38.3	1.79	6.33
0.10	56	515.9	−38.4	1.85	6.50
0.50	78	327.6	−38.6	3.27	7.25
1.00	98	291.1	−38.7	4.11	7.61
5.00	99	182.9	−38.8	7.10	7.75
10.0	100	160.9	−38.9	7.40	7.98

decreasing interfacial tension which results in a reduction in MDD [30–33]. Significant decreases in MDD of paraffin emulsion were obtained at surfactant concentration up to 10 mg/ml (Table 1 and Fig. 1).

As the surfactant concentration increases from 0 to 10 (mg/ml), the zeta potential drops down from −38.1 to −38.9 mV and the original emulsion pH increases from 5.82 to 7.98 (Fig. 1). The maximum negative charge on the paraffin wax droplets covered by SDS was a result of the adsorption of the sulfate ion at the O/W interface. Increasing of the surface charge can improve the stability of emulsions because of the increasing repulsive forces between the droplets against flocculation and coalescence.

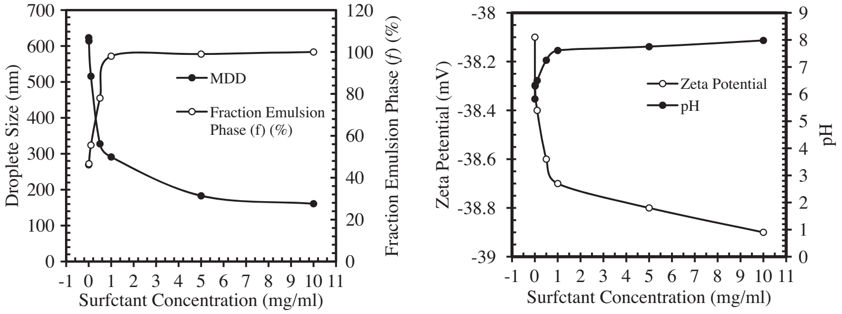
It was found that, as a surfactant concentration increased from 0 to 10 (mg/ml) at constant ϕ (0.2), the viscosity of the emulsion also increases (Table 1). This is because of as the surfactant concentration increases, the increase in effective volume fraction due to the contribution of the electric double layer of surfactant around the emulsion droplets, as well as with an elevated surfactant concentration water molecule gets trapped in cross-linking chains of surfactants, resulting in increased viscosity of the emulsion [34].

3.2. Effect of acoustic cavitation on paraffin wax emulsion

To find, the likely possible chemical effects of the ultrasonic irradiations on to paraffin wax during the emulsification process, the thermogram and FTIR spectra of emulsion were compared with bulk paraffin wax. Fig. 2 shows the thermograms of emulsified (a) and bulk paraffin wax (b). The emulsion was heated from 25 to 300 °C, and the dispersed paraffin wax melted at 63.75 °C, which is very close to the melting temperature of the pure paraffin wax (62.21 °C) obtained from thermogram of bulk paraffin wax. The peak at 105.65 °C in emulsified paraffin wax thermogram shows the boiling point of water. From this thermogram, it is confirmed that no chemical changes occur in the paraffin wax during the emulsification process. Fig. 3 shows the FTIR spectra of emulsified (a) and bulk paraffin wax (b). From FTIR spectra of emulsified and bulk paraffin wax, it was observed that only one extra peak is present in emulsion at 3429.20 cm^{-1} corresponding to H–OH stretching indicating the presence of water. The remaining peaks observed with both the samples are same, confirming that, no chemical changes occur in paraffin wax during emulsification process. Hence, the observed effects have been described using only the physical effects of the ultrasonic irradiations.

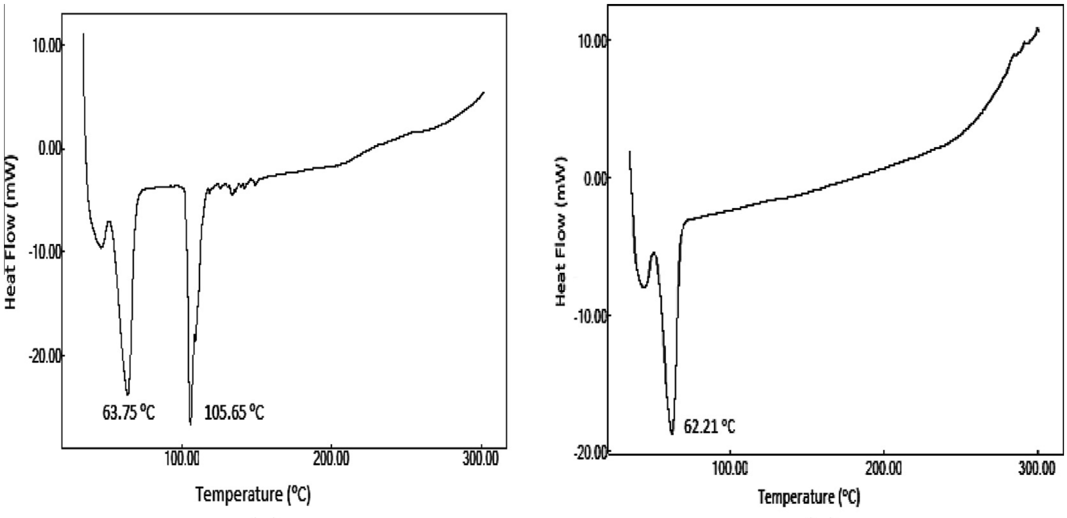
3.2.1. Effect of irradiation power at constant time

Fig. 4(a) shows the effect of irradiation power on the dispersed phase droplet size at a constant time. It was observed that as the irradiation power increase, the mean droplet diameter decrease. This is because of the fact that as the irradiation power increases, the pressure amplitude of the applied sound pressure increases,



(a) (b)

Fig. 1. Effect of surfactant concentration on (a) MDD (nm) and fraction emulsion phase (*f*) (%). (b) Zeta potential (mV) and pH of emulsion formulated via ultrasonic emulsification method. Composition: O/W emulsion (100 mL sample); ϕ: 0.2, prepared at 40% of applied power and 15 min sonication time.



(a) (b)

Fig. 2. Typical thermogram of (a) emulsified and (b) bulk paraffin wax. Samples were heated from 10 to 300 °C.

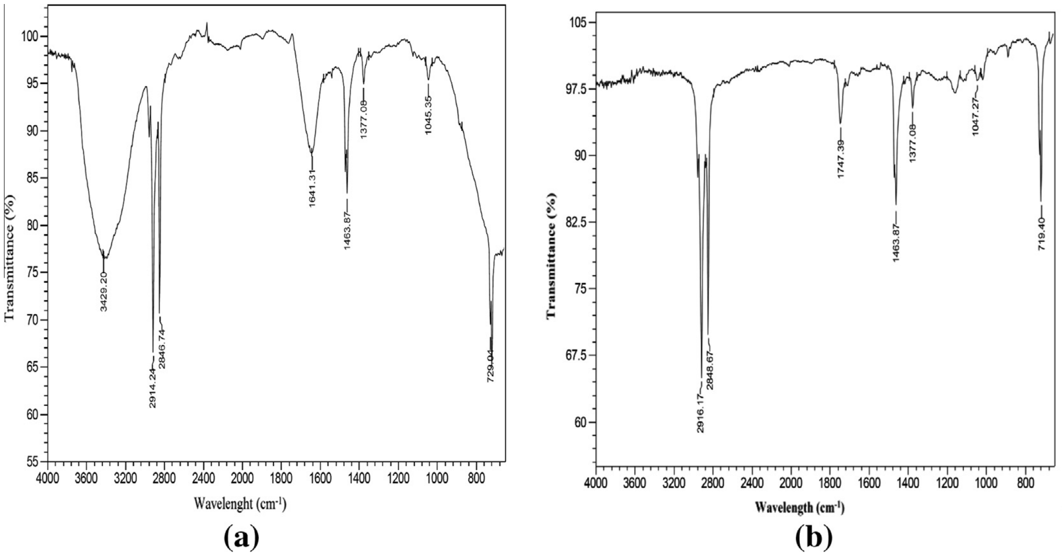


Fig. 3. FTIR spectra of (a) emulsified and (b) bulk paraffin wax.

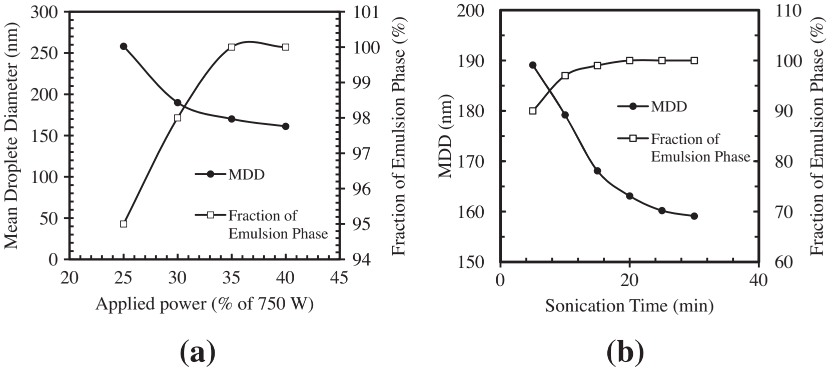


Fig. 4. (a) MDD and fraction of emulsion phase at different applied powers for 15 min sonication time. (b) Effect of sonication time on MDD and fraction of emulsion phase at constant power (40 % of maximum of 750 W). Composition: O/W nanoemulsion (100 mL sample), oil ϕ: 0.2, SDS: 10 (mg/ml) as surfactant concentration.

which will increase the cavitation phenomena (number of events and the cavity collapse intensity). When the irradiation power increases, the rate of energy dissipation in the system is also increases which also increase the rate of rise of temperature and with an increase in the temperature, the interfacial tension and viscosity is observed to decrease, which increases the ease of dispersion of one phase into other. Thus, as the increased power dissipation increases the phase hold-up of the dispersed phase at a constant irradiation time.

As discussed earlier, the breaking of large size droplet into nanosize droplet is dependent on the amount of disruptive forces or energy delivered to the liquid sample. Ultrasonic (acoustic) cavitation produces the shear force which causes the disruption of large droplets into smaller droplet and these newly formed droplets get stabilized by the added surfactant. For applied powers of 25%, 30%, 35% and 40% of the maximum power (750 W), their corresponding power densities were determined (Table 2). Table 2 also shows the applied power and physico-chemical properties of the paraffin wax emulsion. As shown in Fig. 4(a), an increase in the applied power from 25% to 40% (power density increased from 0.29 to 0.61 W/ml) of maximum power resulted in the decrease of MDD at a fixed time.

3.2.2. Effect of sonication time at constant irradiation power

The effect of sonication time on MDD and the stability of emulsion were checked by preparing the emulsion with varying sonication time, at fixed applied power of 40%, 0.2 oil fraction and 10 mg/ml of surfactant. Effect of different sonication times, i.e., 5, 10, 15, 20, 25 and 30 min on MDD and stability is shown in Fig. 4(b). It was observed that as the sonication time increases, the droplet diameter of emulsion goes on decreasing and the fraction of emulsion phase goes on increasing. There was a significant decrease in MDD of emulsion with an increase in sonication time from 5 to 15 min as well as increase in the fraction of oil in the emulsion phase. For different sonication times, i.e., 5, 10, 15, 20, 25 and 30 min at a fixed power density, their corresponding energy input values were determined as 3.82, 8.27, 12.33, 16.63, 22.20 and 27.38 kJ, respectively. This result shows that, total energy dissipated into the system was the function of time at a constant power density. The optimum sonication time was found to be 15 min, after that sonication time had little effect on the MDD reduction as well as the fraction of oil/wax in the emulsion phase.

3.3. Morphology of nanoemulsion

The morphology of paraffin nanoemulsion was visualized using SEM. Fig. 5 shows the SEM image of paraffin nanoemulsion. The droplets were spherical in morphology and were in the range of 100–175 nm. The data pertaining to droplet size obtained by SEM analysis correlates well with the range of droplet diameter obtained using particle size analyzer.

Table 2
Energy dissipated and power input (power density) supplied to emulsion and their physico-chemical properties.

Applied power (% of 750 W)	Sonication time (min)	SDS (mg/ml of emulsion)	Energy dissipated (kJ)	Input power (W)	Power density (W/ml)	Fraction of Emulsion Phase (*f*) (%)	Droplet size (nm)	Zeta potential (mV)	Viscosity (cP)	pH
25	15	10	13.27	14.74	0.29	95	258.1	−36.7	3.25	7.23
30	15	10	18.72	20.80	0.42	98	189.9	−31.6	3.45	7.24
35	15	10	22.85	25.39	0.51	100	170.1	−34.8	3.20	7.24
40	15	10	27.38	30.42	0.61	100	161.1	−38.7	3.27	7.25

3.4. Ultrasonic emulsification vs. emulsion inversion point method

To compare the result obtained from ultrasonic emulsification method, the paraffin wax emulsion was prepared by EIP method. The EIP method is a catastrophic phase inversion process, which is induced by a change in the water-to-oil ratio. Fig. 6 shows the emulsion conductivity with water weight fraction. The conductivity was low at low water fraction and increased sharply at 35 wt% of water, indicating the phase inversion from W/O (oil as continuous phase) to O/W (water as continuous phase). It was found that, the emulsion prepared using ultrasound with 10 mg/ml of surfactant having intrinsic stability (assessed by storing the emulsion at room temperature and then observing it for any phase separation or creaming) of more than 3 months while the emulsion prepared by EIP method gets separated/creamed within 30 min. of storage at room temperature as shown in Fig. 7. From results it was concluded that, the formulated paraffin wax emulsion via EIP method is very prone to destabilization while ultrasonic emulsification is very effective method for the production of stable paraffin wax emulsion.

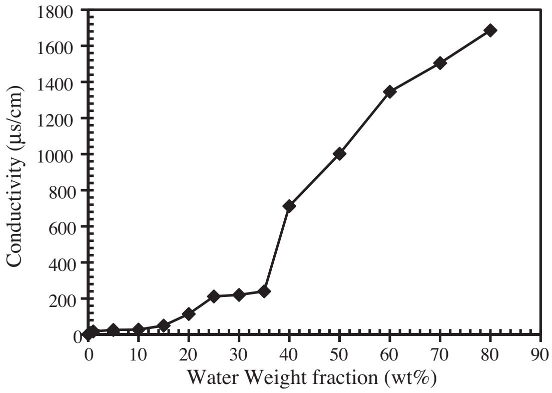


Fig. 6. Variations in the conductivity with water weight fraction containing surfactant concentration of 10 mg/ml of emulsion for emulsion system prepared at 65–70 °C.

In the emulsification either via ultrasonication method or EIP method, typically two opposing processes take place (a) droplet break-up and (b) droplet re-coalescence. The frequencies of occurrence of both the processes depend on the intensity of shear provided to the two phase materials (paraffin wax and water). The droplet break-up occurs when the applied shear is greater than the Laplace pressure within the emulsion drop [2]. The efficiency of droplet break-up is also controlled by the nature and the intensity of the shear. The efficiency of droplet break-up is also triggered using surfactant [35]. The surfactant can reduce the interfacial tension which results in decreases the resistance (reduction in Laplace pressure) to droplet deformation. As soon as, new droplet form surfactant gets adsorbed onto the surface and prevent the immediate re-coalescence of newly formed droplets, and stabilizes the newly formed interface. The coalescence is the irreversible de-emulsification process, it is the result of drop collision; it take place if the continuous-phase film between colliding drops drains and ruptures and the drops finally merge to form a single drop. Poorly enclosed drops by surfactant or the presence of large sized drops in the emulsion increases the probability of drop coalescence. Finally, the more stable nanoemulsion can be formed if the droplet break-up process is more dominant over the droplet re-coalescence as

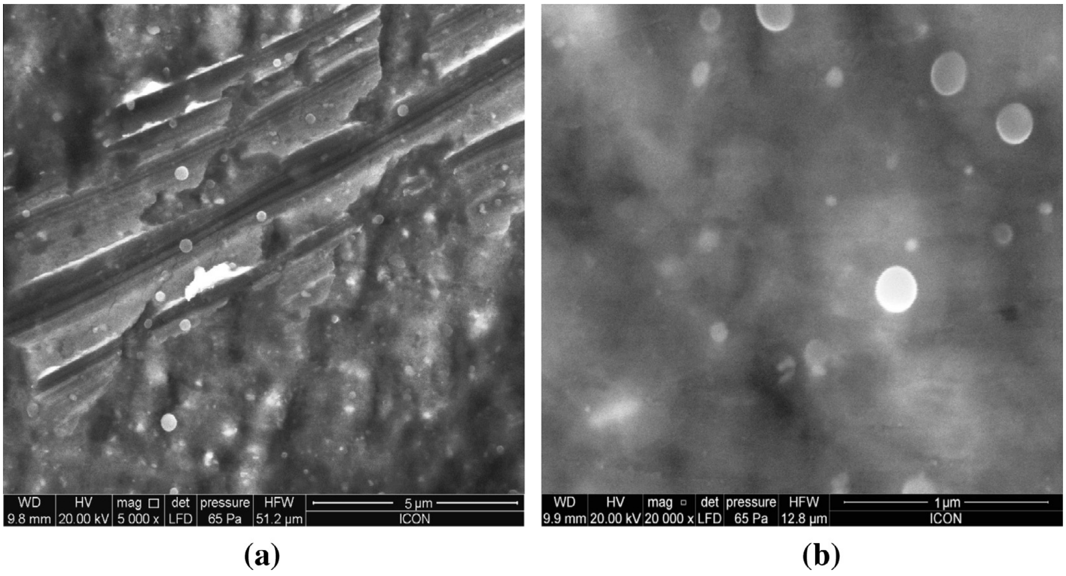


Fig. 5. SEM images of paraffin wax emulsion at (a) 5000×, (b) 20,000×.

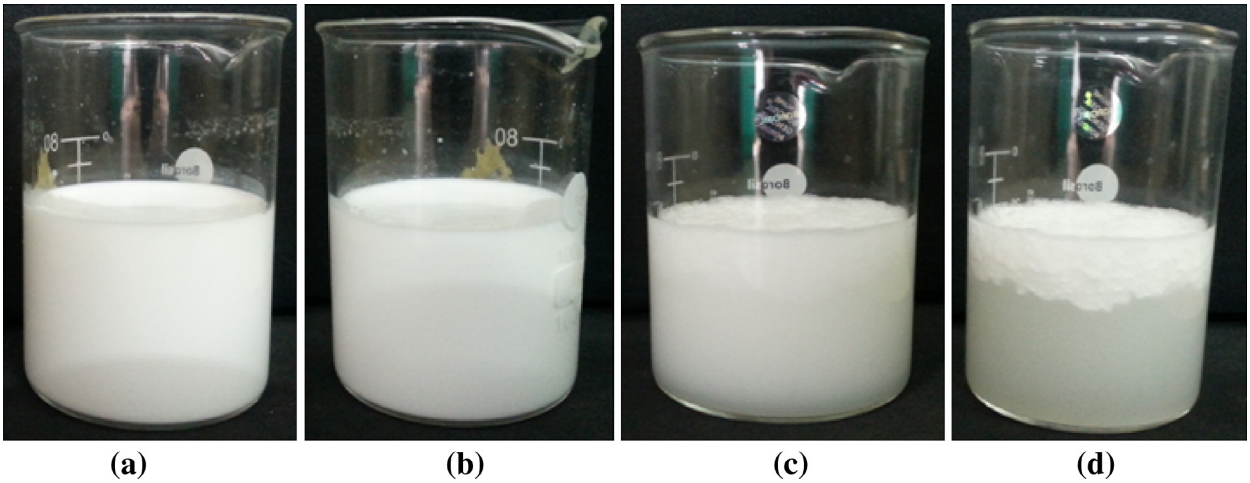


Fig. 7. Visual appearance of emulsions prepared by (a) sonication, after 1 min., (b) sonication, after 3 months (c) emulsion inversion point method, after 1 min. (d) emulsion inversion point method, after 30 min.

well as simultaneous adsorption/coverage of newly formed droplet surface with surfactant [36].

Ultrasonic emulsification process is mainly occurring through two mechanisms. Initially, an acoustic field creates interfacial waves which erupt the oil phase into the aqueous phase in the form of droplets. Then, the ultrasound causes acoustic cavitation, that is, the formation, growth and subsequent collapse of microbubbles caused by the pressure fluctuations of the acoustic wave. Each bubble collapse event causes extreme levels of highly localized turbulence and acoustic streaming induces micromixing and increased diffusivity of the surfactant causes better surface coverage stabilizing newly formed drops. The turbulent micro-implosions act as a very effective method of breaking up primary droplets of dispersed oil into droplets of sub-micron size [19] and the simultaneous adsorption of surfactant on to the newly formed droplet stabilizes the interface and makes the emulsion more stable.

In EIP method, the droplet break-up process is less dominant than the droplet re-coalescence process. Also, the adsorption of surfactant on to the surface of droplet is molecular diffusivity dominated and cannot stabilize the droplet due to the larger size of the droplet. Because of larger droplet size and partially coverage of droplet surface by surfactant, this type of emulsion is less stable.

The amount of energy required for the formulation of paraffin wax emulsion US and EIP methods has been compared in Appendix A. The energy utilized for the formulation of paraffin wax emulsion is the total energy supplied (kJ) per unit weight of the material (final emulsion) obtained (g). The total time required for the formulation of paraffin wax emulsion by US and EIP was 20 min and 180 min respectively. Total energy required per unit weight of the emulsion is 4.939×10^{-2} (kJ/gm) for US method and 90.998×10^{-2} (kJ/gm) for EIP method.

4. Conclusions

Ultrasound-assisted paraffin wax nanoemulsion stabilized by SDS was successfully prepared. Although, nanoemulsions were produced at all levels, optimum process and formulation parameters values have been identified for the preparation of very stable nanoemulsion with smallest mean droplet diameter at lowest possible delivered power, sonication time and minimum surfactant concentration for maximum oil fraction loading. It was found that 40% of applied power (power density: 0.61 W/ml) and 15 min of sonication time was optimum. Mean droplet diameter of the emulsion and fraction of emulsion phase were found to be 160.9 nm and 100%, at a surfactant concentration of 10 mg/ml of emulsion.

There was no chemical effect of acoustic cavitation on paraffin wax during the emulsification process confirmed using DSC and FTIR analysis of the emulsified and bulk paraffin wax. The observed effects have been described taking into consideration only the physical effects of the ultrasonic irradiations.

Paraffin wax used as the oil phase of the emulsion is a crystallizable oil phase, so we used scanning electron microscopy (SEM) to visualize the morphology of the emulsion. The results show that the emulsion droplets are solid and with a regular spherical shape, which is better for emulsion stability. To clarify the stability mechanism of the emulsion, the electrostatic properties were also investigated. The zeta potential increased from −38.1 to −38.9 mV, as the surfactant concentration increased from 0 to 10 mg/ml of emulsion. The origin of the negative surface charge on paraffin wax droplets is thought to be a result of the adsorption of head sulfate groups of the SDS surfactant on the O/W interface.

Emulsion inversion point (EIP) method was used to prepare paraffin wax-in-water emulsions stabilized by SDS surfactant. The emulsion prepared was very unstable, it got separated/creamed within 30 min. of storage. Thus, the ultrasonic emulsification is an excellent method for formulation of paraffin wax nanoemulsion.

This process is environmental benign, easy to handle also promises us future large scale manufacturing using hydrodynamic cavitation.

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Appendix A. Energy calculations

A.1. Energy delivered during sonication

- Energy delivered during sonication = energy required to formulate paraffin wax emulsion.
- Electrical energy delivered during sonication using horn for 20 min (indicated by the power meter) = 16.631 kJ.
- Efficiency of horn taken for the calculation = 30% (estimated independently using calorimetric studies).
- Actual energy delivered by horn during sonication = energy delivered during sonication using horn in 20 min

- Efficiency of horn = $16.631 \times 30/100 = 4.989$ kJ.
- Quantity of material processed = quantity of water + quantity of paraffin wax + quantity of sodium dodecyl sulfate = 80 g + 20 g + 1 g = 101 g.
- Net energy supplied for processing of material using sonochemical method = actual energy delivered by horn during sonication/quantity of material processed $= 4.989\,(\mathrm{kJ})/101\,(\mathrm{g}) = 4.939 \times 10^{-2}\,(\mathrm{kJ/g}) \quad (\mathrm{A})$

A.2. Energy delivered during conventional method

- Voltage input in magnetic stirrer (Model RQ1210, Remi Metals Gujarat Limited, India) = 230 V.
- Current measured using digital multimeter (KUSAM-MECO Model 2718, Kusam Electrical Industries Ltd., Mumbai, India) = 37 mA = 37×10^{-3} A.
- Power input in overhead stirrer = voltage input × current measured = $230\,(\mathrm{V}) \times 37 \times 10^{-3}\mathrm{A} = 8.51$ W (J/s).
- Time required for completion of reaction = 180 min (10,800 s).
- Net energy delivered during EIP method = power input in magnetic stirrer time required for completion of reaction = 8.51 J/s × 10800 s = 91,908 J = 91.908 kJ.
- Quantity of material processed = quantity of water + quantity of paraffin wax + quantity of sodium dodecyl sulfate = 80 g + 20 g + 1 g = 101 g.
- Net energy supplied for processing of material using EIP method = net energy delivered during EIP method/quantity of material processed = $91.908\,(\mathrm{kJ})/101(\mathrm{g}) = 90.998 \times 10^{-2}\,(\mathrm{kJ/g}) \quad (\mathrm{B})$.

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