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Towards an Understanding of Optimum Ultrasonication Process Time on Size Reduction of Hempseed Oil Nanoemulsions

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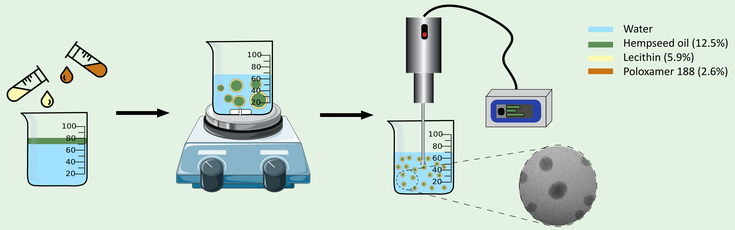
Purpose

To explore an optimal ultrasound processing time for a range of different formulations and volumes of hempseed oil (HSO) nanoemulsion produced by various ultrasonic amplitudes.

Methods

Hempseed oil nanoemulsion was prepared through a two-step process:



• Pre-emulsions were formulated based on previous optimal ratios found using stirring. Next, different volumes of pre-emulsions (5-50 mL) were subjected to various ultrasonications at 20 kHz at different amplitudes (25-100% full power) for various process times (1-60 min).

• The hydrodynamic diameters of the emulsion droplets were measured with dynamic light scattering (DLS) technique.

• The morphology of the nanoemulsions was observed by Transmission Electron Microscopy (TEM).

• Models created by Matlab Curve fitting tool to find the relationship between particle size (Ps) and ultrasound time (t):

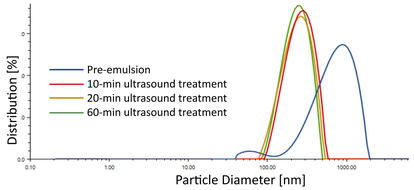
$$Ps(t) = at + b \quad \text{Zero-order}$$
$$Ps(t) = ae^{-b*t} + c \quad \text{First-order}$$
$$Ps(t) = \frac{1}{\frac{1}{a}+(b*t)} + c \quad \text{Second-order}$$

Optimal ultrasound time (Ps_{opt}) for maximum size reduction (τ %) was calculated as:
$Ps_{opt} = Ps_{before} - \tau\% \, (Ps_{before} - Ps_{after})$

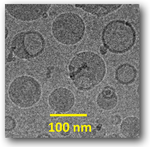
Emulsion Characterization Results

• The average droplet size HSO nanoemulsion prepared by optimized formulation was 176.2± 5.04 nm.

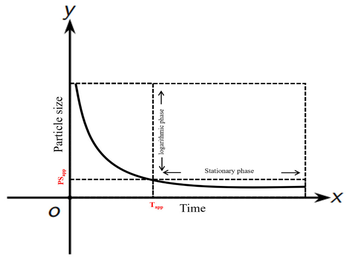
• Particle size distribution by intensity showed no significant difference ($p > 0.05$) between emulsion samples with different processing times.



• The morphology study of the optimal emulsion showed spherical appearance with uniform size.

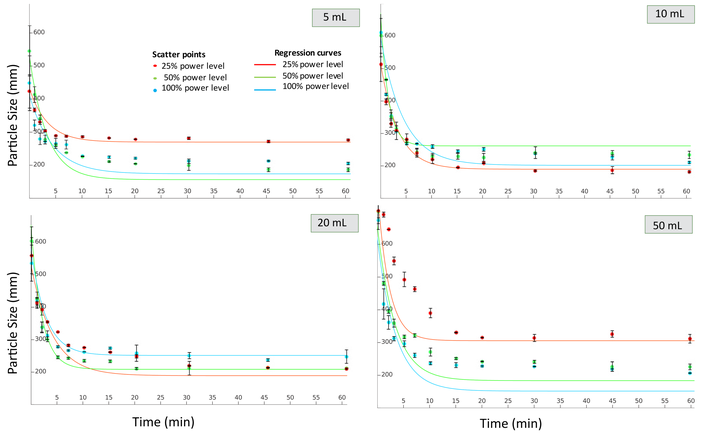


• Optimal nanoemulsion process time model for the maximum particle size reduction (τ%):



Modeling Studies Results

• The correlation curves between processing time (min) and particle size (nm) of samples with different volumes under various ultrasonic amplitudes:



• The relation between Ps and process time (t) was best fitted to first-order model (R^2 = 0.95 ± 0.04).

$$Ps(t) = ae^{-b*t} + c \quad (1)$$

- When t → ∞, $Ps(t)$ → c, c = final particle size after 60 min process time
- When t = 0, $Ps(t) = a + c \rightarrow Ps_{before} = a + c$, $Ps_{after} = c$

• The relationships between the optimal time (T_{opt}) and Ps (t), and maximum reduction time (τ %) were:

$$Topt = \frac{-2.303}{b} \times log\left(\frac{(1-\tau\%)\,Ps_{before} + \tau\%\,Ps_{after} - c}{a}\right) \quad (2)$$

Equation (2) simplifies to:

$$Topt = \frac{-2.303}{b} \times log\,(1-\tau\%) \quad (3)$$

• The T_{opt} tended to be the same once τ% was close to 1. Thus, τ% was defined as 99% to calculate the T_{opt}. The T_{opt} was found ~ 10 min for nanoemulsion samples prepared in different volumes and with various amplitudes.

Conclusion

• The optimum formulation of hempseed oil nanoemulsion prepared with ultrasound process contained particles with average size of 176.2± 5.04 nm .
• Particle size reduction was not significantly (p> 0.05) impacted by the ultrasonication process time.
• The first-order model was the most suitable for modeling the relationship between the process time and the particle size reduction.
• The T_{opt} of all samples was around 10 min independent of volume (mL) and processing amplitude (%).
• The results of this study provide insight into the ultrasonic process optimization for nanoemulsions prepared for drug delivery purposes.

Acknowledgment

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