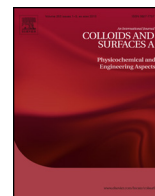




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The effect of oils, low molecular weight emulsifiers and hydrodynamics on oil-in-water emulsification in confined impinging jet mixer

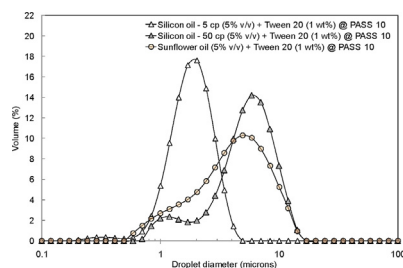
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HIGHLIGHTS

- Emulsification proceeds in turbulent-inertial regime at low o/w interfacial tensions.
- Wide range of oils (triglycerides, silicones and alkanes) and small emulsifiers studied.
- Complex interplay of process variables during emulsification process.
- Droplet coalescence effects can be removed by re-circulation.
- High molar-volume oils like triglycerides produce stable emulsions.

GRAPHICAL ABSTRACT



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ABSTRACT

Oil-in-water emulsification is carried out in turbulent-inertial regime in confined impinging jet mixer (CIJM). Sunflower oil (triglyceride, 50 cP), silicon oils (5 cP and 50 cP) and *n*-alkanes (heptane 0.56 cP and dodecane 1.34 cP) are dispersed in aqueous phase in presence of low molecular weight emulsifiers - anionic sodium dodecyl sulfate (SDS) and nonionic Tween20 over a range of Reynolds numbers (Re) and Weber numbers (We). To prevent droplet coalescence the experiments are performed at high emulsifier concentration. Relative influence of disperse oil-phase viscosity, o/w interfacial tension, local hydrodynamics and multi-pass circulation on formation of emulsions as small as 1–2 μm (mean Sauter diameter) is investigated. Results show that higher viscosity oils favor production of bigger emulsion droplets when oil/water interfacial tensions are similar likewise low oil/water interfacial tensions favor formation of smaller droplets when oil phase viscosities are identical. Also more viscous and higher molar volume sunflower oil (triglyceride) emulsions record higher emulsion stability than *n*-alkane emulsions. Emulsions where oil viscosities and o/w interfacial tensions vary simultaneously are also investigated and effects documented.

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1. Introduction

In the last decade or so there has been a lot of interest in emulsions particularly in food, beverage and pharmaceutical industry.

Emphasis is on producing emulsions with narrow droplet size distributions, lower capital and operating costs and using equipment that require minimal maintenance.

Emulsions, in general, are produced using mechanically stirred tanks or continuous in-line static mixers but high-shear rotor–stator devices and homogenizers are extensively used in specialty industries like food and beverages. Commonly used stirred tanks see highly non-uniform mixing across the mixing volume where energy dissipation rates may vary by 2 orders of magnitude [1]. A non-uniform mixing field thus requires addition of feed

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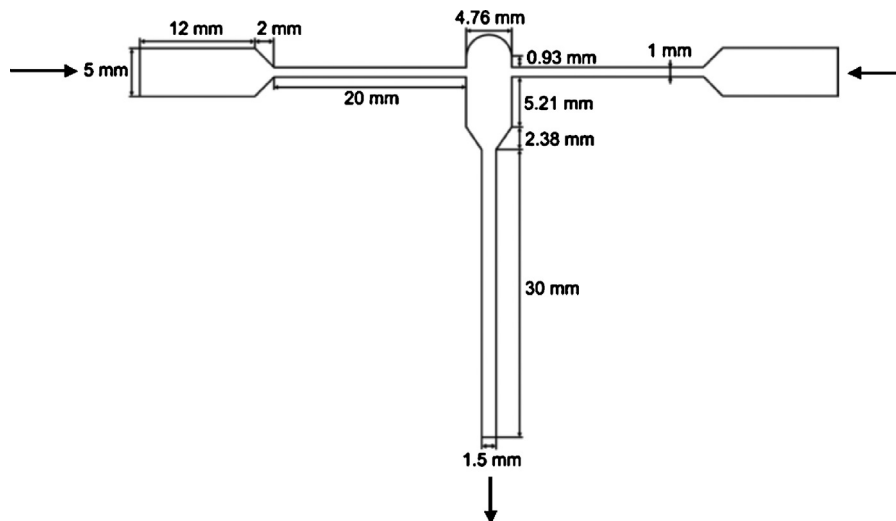


Fig. 1. Dimensions of confined impinging jet mixer (CIJM) with liquid jet entry and exit points marked (reproduced from Siddiqui et al. [1,3]).

stream(s) into zone of high turbulence and high energy dissipation to limit consequences of poor mixing. Whereas poor mixing of liquid streams may cause formation of by-products in mixing-sensitive chemical reactions [1,2]; poor mixing may also end up producing emulsions with broad droplet size distributions. On the other hand, local turbulence and energy dissipation rates in high pressure homogenizers are several orders of magnitude higher than in stirred tanks. The valve design in homogenizers allows development of high turbulence zone near the valve edges where most of the droplet breakage occurs. However away from the valve edges, low shear zones may exist favoring droplet coalescence [3]. High-pressure homogenizers may thus be easily scaled-up but tight control over emulsion quality remains a challenge. By contrast, confined impinging jet mixer (CIJM) is a proven novel mixer design to achieve good micromixing and high energy dissipation (10^3 – 10^4 W/kg) as required for highly mixing-sensitive systems [1,2]. Siddiqui and Norton [3] have used CIJM (Fig. 1) to produce food-grade emulsions of average size 3–4 μm . Present study is one of the earliest ones to demonstrate suitability of confined impinging jet mixer (CIJM) for producing smaller emulsions (< 2 μm) with controlled droplet size distributions for various speciality applications.

Working of confined impinging jet mixer (CIJM) is based on impingement of two high-velocity liquid jets within a confined space ($\sim 10 \mu\text{L}$) followed by generation of a localized region of very high turbulence (characterized by peak energy dissipation at the impingement point) causing rapid reduction of segregation between the colliding streams. It approaches a plug flow device with very narrow residence time distribution and very high local energy dissipation rate with increase in inlet stream velocities [1]. These mixing characteristics make CIJM attractive for mixing-sensitive systems with high product quality requirements, yet requiring low input energy for high product throughput [4].

In emulsification process, irrespective of the mixing device being used, it is important to consider droplet formation step as a dynamic process between droplet breakup and re-coalescence steps. These duo-competing process steps are influenced by emulsifier adsorption kinetics and interface stabilization step [3,5,6]. Siddiqui and Norton [3] have shown that local mixing conditions influence various emulsification steps including dispersion of fluid blobs to smaller droplets, droplet–droplet collisions (hence droplet coalescence) and convective transport of emulsifier to the droplet interface, however; close to the droplet interface, diffusion of emulsifier is the dominant mass transfer mechanism which depends

on emulsifier size besides other factors. Thus smaller the emulsifier molecule, faster it diffuses to the interface, and smaller will be dynamic interfacial tension [6]. When emulsifier concentration is sufficiently high and the type right, droplet coalescence is restricted making droplet breakage step the size determining step [5,7]. Thus mixing characteristics of the device besides other physical physicochemical properties must be considered when designing emulsification processes [3].

During emulsification droplet breakage may proceed in planar shear, elongational, turbulence or cavitation field [8,9]. In a turbulent field, droplet breakage may occur either in ‘turbulent-inertial regime’ or ‘turbulent-viscous regime’ depending on the dominant –viscous and inertial stresses acting on the droplet. Which one of these stresses dominates depends on the ratio of the droplet size to the smallest turbulent eddy size i.e. Kolmogorov microscale (λ_k) [10,11]. Thus when emulsification proceeds in turbulent-viscous flow regime resulting emulsion droplets are smaller than Kolmogorov microscale while if droplet break-up proceeds in turbulent-inertial regime emulsion droplets larger than Kolmogorov scale are produced. In this work, emulsification is carried out in turbulent-inertial regime and Sauter mean diameter D_{32} and droplet size distributions distributed in volume are measured and reported at each step.

$$\lambda_k = \left(\frac{v_c^3}{\varepsilon} \right)^{1/4} \quad (1)$$

In Kolmogorov microscale (λ_k), v_c is the continuous phase kinematic viscosity ($=\mu_c/\rho_c$), and ε is the mean or peak energy dissipation rate (m^2/s^3 or W/kg) within the mixer [10]. Energy dissipation rates used in this work were experimentally and numerically determined by Siddiqui et al. for the range of hydrodynamic and mixing conditions [1,2].

According to Kolmogorov–Hinze emulsification theory, in turbulent-inertial regime, maximum stable disperse phase (comparable viscosity to water) droplet size d_c is estimated by equating hydrodynamic pressure fluctuations in the flow $\{=C_1 C_2 \rho_c (\varepsilon d_c)^{2/3}\}$ with the capillary pressure (of the droplet) opposing droplet deformation $\{=4\sigma/d_c\}$ [10,11]. Here C_1 and C_2 are constants of the order of unity. Kolmogorov–Hinze theory however doesn’t clearly account for the influence of disperse phase viscosity (μ_D) on droplet size and is only valid for low or comparable disperse phase viscosities to water i.e. $\mu_d \leq \mu_c$ [7]. Davies [12] and Berkman and Calabrese [13] therefore proposed to account for viscous disperse phase ($\mu_d > \mu_c$)

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