

Pilot-scale production process for high internal phase emulsions: Experimentation and modeling

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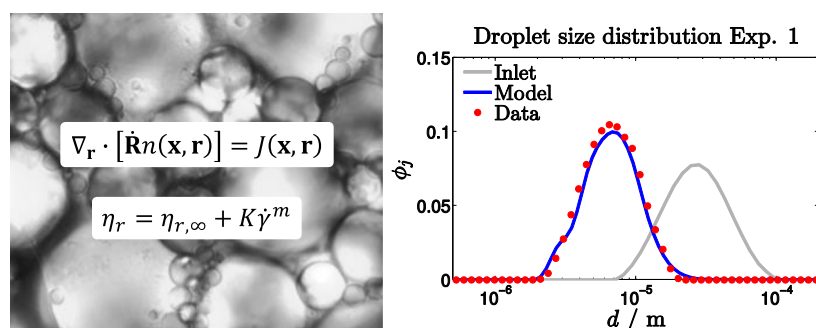
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HIGHLIGHTS

- A model was proposed to capture the droplet size-emulsion viscosity coupling.
- The two-way coupling was modeled by applying the mean-field viscosity approach.
- The emulsion viscosity model was validated with actual data at high shear rates.
- A variable number of fragments were included in the redistribution function.
- The model reproduced droplet size distributions measured under simple shear flow.

GRAPHICAL ABSTRACT



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ABSTRACT

The droplet size distribution determines some crucial properties for high internal phase emulsion (HIPE) products such as product structure, mouth feel and color. This research describes the development of a population balance model which aims at the prediction of the droplet size distribution. The objective is to find one set of parameters which is able to describe a well-planned experimental dataset. Two issues in particular are addressed in this paper. First, a redistribution function is introduced which is based on single droplet breakage. The number of daughter droplets is now a function of the system properties instead of an arbitrary constant. This approach significantly improved the model predictions. Second, the shear thinning viscosity of the HIPEs at high shear rates was measured and modeled using a semi-empirical model, which is based on the renowned Cross model. The modified Cross model was able to describe the viscosity of 18 different mayonnaises varying in oil content over a shear rate range from 10^{-1} to 10^4 s^{-1} with an average deviation of 16%. The population balance model was able to reproduce the Sauter mean diameter $D_{3,2}$ and the cumulative volume mean diameter $D_{v,10}$ with one set of fit-parameters but failed to estimate the larger droplet sizes.

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1. Introduction

For high internal phase emulsion (HIPE) products the droplet size distribution is an important variable to control, because it determines some crucial product properties such as structure, mouth

feel and color. Up to now a lot of research was dedicated to model the complete size distribution of droplets obtained after a high shear mixing process. The aim of this research was to control the droplet size distribution by changing operational variables. The droplet size distribution was modeled for high pressure homogenizers using population balance tools (see e.g. Raikar et al., 2009).

It was soon realized that product formulation also plays an important role in the outcome of a mixing experiment. By

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Nomenclature

Latin symbols

A	cross sectional area, m^2
A_H	Hamaker constant, J
$C(v, v')$	coalescence rate, $\text{m}^3 \text{s}^{-1}$
Ca	capillary number
d	droplet diameter, m
$D_{3,2}$	Sauter mean diameter, m
$D_{v,i}$	cumulative droplet diameter, m
F_v	volumetric flow rate, $\text{m}^3 \text{s}^{-1}$
$g(v)$	breakage rate, s^{-1}
$h(v, v')$	collision frequency, $\text{m}^3 \text{s}^{-1}$
J	source term population balance, $\text{m}^{-6} \text{s}^{-1}$
K	consistency, Pa s^n
K_{1-3}	fit parameters
K_f	Pal's viscosity parameter
m	fit parameter
n	number density, m^{-6}
p	number of daughter droplets
\mathbf{R}	fluid motion, m s^{-1}
R	droplet radius, m
Re_θ	rotational Reynolds number

\mathbf{r}	external coordinates, m
t_b	droplet breakup time, s
t_{res}	residence time, s
v	droplet volume, m^3
v_z	axial velocity, m s^{-1}
\mathbf{x}	internal coordinates, m
z	axial coordinate, m

Greek symbols

$\beta(v, v')$	daughter droplet size distribution function, m^{-3}
η_c	continuous phase viscosity, Pa s
η_e	mean field viscosity, Pa s
η_r	relative viscosity
$\eta_{r,\infty}$	high shear relative viscosity plateau
ϕ_j	dispersed phase volume fraction size class j
ϕ	dispersed phase volume fraction
ϕ_{max}	maximum packing volume fraction of solid spheres
$\dot{\gamma}$	shear rate, s^{-1}
$\Lambda(v, v')$	coalescence efficiency
λ	viscosity ratio
σ	surface tension, N m^{-1}
Ψ	objective function value

changing the concentration and/or the type of surface active components the final droplet size distribution could well be manipulated (see Maindankar et al., 2013, 2015). The breakage and coalescence of oil droplets were modeled as a function of the average energy dissipation. More refinement was achieved when more hydrodynamic features were incorporated into the models, such as taking the average energy dissipation in various zones of the mixer (Alopaues et al., 2002; Almeida-Rivera and Bongers, 2010; Dubbelboer et al., 2014) or fully integrating a Computational Fluid Dynamics simulation with a population balance in every grid cell (Becker et al., 2014). The population balance equation usually contains a couple of free parameters so that the model could be fitted to experimental data.

The models and experiments cited above were developed for dilute emulsions ($<0.10 v/v$ dispersed phase). Dilute, in the sense that the dispersed phase did not influence viscosity too much and that the macroscopic emulsion viscosity remained Newtonian which could be described by the famous suspension viscosity equation from Einstein (1906). However, when the concentration of the dispersed phase is increased the rheological emulsion properties change to non-Newtonian. The emulsion droplet size distribution starts to have an effect on the macroscopic rheological properties. The smaller the droplets the more viscous the emulsion becomes. This is because dispersions with smaller particles have larger Peclet numbers and Brownian forces keep dominating longer upon increasing the shear stress (Brady, 2001). For flocculating dispersions the decrease in droplet size increases the interfacial area and therefore the degree of flocculation which increases the viscosity (Barnes, 1994). Another explanation for the increase of the emulsion viscosity by decreasing the droplet size comes from the increase of the effective volume fraction, which is the sum of the film layer and the volume of all droplets (Princen, 1986). During an emulsification experiment the droplet size is reduced while simultaneously the viscosity of the emulsion increases. This viscosity increase influences the flow and as a consequence the rate of droplet breakup. Modeling this interaction is stipulated by Janssen and Hoogland (2014) as the top of the modeling hierarchy in terms of complexity. Therefore solving the development of the droplet size distribution in time remains far out of reach for practical industrial cases. Couette type

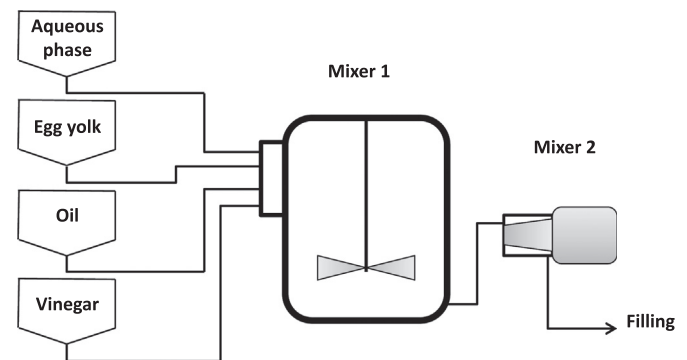


Fig. 1. Part of the production process for mayonnaises and dressings.

mixers such as the colloid mill operate in the laminar regime especially when processing very viscous liquids. Then the velocity gradient is constant between the rotor and the wall. When a mean field viscosity model is opted for, the interaction between flow and droplet breakup should be feasible, even in an optimization context.

There are many emulsified products available which contain a highly concentrated dispersed phase, for example, polymeric materials, paints, and foods such as mayonnaises and dressings. The work described in this paper presents the modeling results of an emulsification process for the manufacturing of mayonnaise. Mayonnaise is an oil-in-water emulsion stabilized by egg yolk. Other important ingredients are vinegar, salt, sugar, and mustard. Egg yolk is a mixture of complex biomolecules from which a few adsorb and unfold at the oil–water interface. A typical mayonnaise production process consists of two mixers, see Fig. 1. The first mixer is a fed batch system, where the oil phase is slowly added to the water phase to ensure the formation of an oil-in-water emulsion. The second mixer is usually a high shear mixer, which breaks up the coarse oil droplets to reach the required specifications.

In general, mayonnaises and high internal phase emulsions are shear thinning fluids which may show elastic properties as well. The fact that the droplets have to deform in order to pack together causes the Laplace pressure of the emulsion to increase. This excess surface energy translates into elasticity of the system as a

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