



Review

Experimental methods for measuring coalescence during emulsification – A critical review



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ABSTRACT

Emulsification is a common process in the production in many non-solid foods. These food-emulsions often have high disperse phase volume fractions and slow emulsifier dynamics, giving rise to substantial coalescence during emulsification. Optimal design and operation of food-emulsification requires experimental methods to study how emulsification in general and coalescence in particular progresses under different conditions. Methods for coalescence quantification during emulsification has been suggested in literature but they are rarely used in food-emulsification research. This contribution offers a critical review of the different methods that have been suggested with special emphasis on their applicability to technical food-emulsification. The methods are critically compared in terms of design limitations, degree of quantification and applicability. A state-of-the-art in the form of two methods is identified and guidelines for their application are suggested.

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1. Introduction

Many non-solid foods (e.g. dairy products, sauces, dressings and beverages) are emulsions (i.e. liquid-liquid dispersions consisting of drops of one phase dispersed in the other). The drop size distribution (DSD), volume fraction of disperse phase and surface properties all contribute to the structure and perceived quality of emulsion foods (Harrison and Cunningham, 1985; McClements, 2005). Designing and producing high quality emulsion-foods thus requires a high degree of control and predictability in the industrial production of emulsions.

Emulsion foods are formed through *emulsification*, the process of dispersing one liquid phase in the other. Several different techniques are applied for food emulsification, and comprehensive reviews describing the principles and mechanisms are available elsewhere (McClements, 2005; Rayner and Dejmeck, 2015; Santana et al., 2013; Walstra, 2005). Emulsification in general can be described as a combination of at least three processes (McClements, 2005; Tcholakova et al., 2004; Walstra, 2005): (i) fragmentation of drops, (ii) adsorption of emulsifying molecules or particles to the droplet interface and (iii) coalescence of insufficiently covered drops.

1.1. An empirical shift: from resulting drop sizes to dynamics of fragmentation and coalescence

The empiric study of (food-) emulsification has traditionally been based on measuring average drop size or DSD as a function of operating conditions and chemical composition (Boxall et al., 2012; Grace, 1982; Hinze, 1955; Rueger and calabrese, 2013; Sjöo et al., 2015; Tcholakova et al., 2004; Walstra, 1975). These empirical investigations were made possible by advances in rapid and reliable method for measuring DSDs based on light scattering (Goulden, 1958; Walstra, 1968). For ideal system such as fragmentation dominated emulsification (e.g. low volume fraction with excess of low-molecular emulsifier), this approach has allowed for substantial mechanistic insight (e.g. Boxall et al., 2012; Walstra, 1969; Vankova et al., 2007a). However, many food-emulsions are more complex, e.g. mayonnaises, creamy sauces and cake batters. Their emulsifiers are complex macromolecular systems giving rise to slow adsorption and/or surface dynamics (Dalglish, 1997; Kralova and Sjöblom, 2009; Walstra, 2005). Moreover, the volume fraction of disperse phase is high. Together this gives rise to significant coalescence during emulsification (Lobo et al., 2002; Niknafs et al., 2011). For such systems, resulting DSDs convey little mechanistic insight since they do not allow for separately studying fragmentation and coalescence. Thus, the mechanistic understanding of food emulsification needed in improving design requires new methodological advances. One suggestion to further our understanding is to shift the focus of empirical efforts from DSDs to the primary rates of fragmentation and coalescence during emulsification.

1.2. The relevance of measuring coalescence rates

Fragmentation rates can be estimated relatively straight forward by designing fragmentation dominated conditions and measuring the drop size decrease rate (e.g. Andersson and Andersson, 2006; Tcholakova et al., 2007; Vankova et al., 2007b). Measuring coalescence rates during emulsification is more challenging and requires special techniques. Although relatively rarely employed in food-emulsification research, a large number of methods have been suggested (e.g. Curl, 1963; Howarth, 1967; Karbaschi et al., 2014; Lobo et al., 2002; Madden and Damerell, 1962; Taisne et al., 1996). This contribution offers a critical review of these methods for measuring coalescence rates during emulsification and offers

guidelines for food-emulsification researchers in choosing and applying coalescence rate determination methods to improve the understanding of technical food-emulsification.

Emulsification processing is designed and operated under conditions dominated by fragmentation; the net effect is a reduction in drop size. However, the effect of coalescence, i.e. the collision and subsequent fusion of drops, is significant, as could be seen from direct measurement (Håkansson and Hounslow, 2013; Narsimhan and Goel, 2001; Niknafs et al., 2011) as well as from a number of studies on emulsification modeling concluding that coalescence must be included to describe emulsification of complex food-like emulsions (Håkansson et al., 2009; Janssen and Hoogland, 2014; Maindarkar et al., 2015).

A wealth of theoretical models for calculating coalescence rates have been presented in literature (see Liao and Lucas, 2010 for a review). However, as of yet, they offer no viable alternative for food-emulsification researchers interested in characterizing non-ideal systems. As an example, the proposed treatments of macromolecular emulsifier adsorption and transport thereof in these theoretical models have not obtained sufficient empirical support to become an alternative to measurement (c.f. Håkansson et al., 2009; Maindarkar et al., 2015).

1.3. Objective and limitations

Despite that a large number of methods have been suggested for measuring coalescence, from the 1960s and onwards, no comprehensive review comparing the methods, their assumptions and applicability has been offered since Shah et al. (1972). The intention with this paper is to critically review the previously suggested methods for measuring coalescence during emulsification, with special emphasis on principal design, quantification and applicability for forwarding food emulsification research.

It should be noted that coalescence can take place both during emulsification and during subsequent storage. Measuring during emulsification is more challenging due to the shorter time scales and limited accessibility. This review is therefore based on evaluating the ability of the methods during high-intensity emulsification only. Furthermore, studies measuring coalescence through individual drop-drop visualizations (e.g. Liao et al., 2008) have been excluded from the review. These studies are valuable for building fundamental knowledge but less so for quantifying coalescence under technical food-emulsification conditions due to assumptions imposed on volume fraction of disperse phase and optical properties of equipment and phases. Adoption of these methods to technical conditions are thus scarce (see Park and Blair, 1975 for an exception). The review is also limited to methods presented in peer-reviewed journals (excluding e.g. conference abstracts and proceedings). This limitation is not expected to influence the relevance of the review. A preliminary review of conference literature showed no methods, comparisons or development that did not occur in later journal publications.

This paper is organized as follows: A brief background to standardized measures of coalescence is given in Section 2. Previous studies suggesting, evaluating, applying or developing methods for quantifying coalescence during emulsification are reviewed and compared in Section 3. Section 4 discuss common themes, give guidelines for applications of coalescence rate quantification methods and perspectives for further research. The discussion is summarized in Section 5.

2. Quantifying coalescence – rates, frequencies and non-standardized measures

Assume that an emulsification experiment has been designed

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