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## **Review Article**

# Techniques and methods to study functional characteristics of emulsion systems



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#### ABSTRACT

With the growing popularity of the functional food market, bioactive ingredients from natural sources are discovered one after another for their ability to promote better health and prevent chronic diseases. Emulsion, widely occurring in many food systems, has become a popular vehicle to facilitate the incorporation of bioactive components into the food system. Depending on the designated functionality, an emulsion can be developed with various physical and chemical properties. To ensure the successful development of a high-quality emulsion-based system to serve their purpose in food, knowledge of the analytical methods that could efficiently evaluate their quality parameters is important for investigators who work in this field. In this work, important emulsion properties are overviewed, and techniques that are commonly used to assess them are provided. Discussions and recommendations are also included to make suggestions on advantages and disadvantages when selecting suitable techniques and methods to characterize these quality parameters of emulsion systems.

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#### 1. Introduction

As food systems are complex matrices of various components that are not always well mixed with one another, oil—water and/or air—water interfaces are commonly found and could be stabilized using proper emulsifying or surface stabilizing agents. Thus, understanding the science and technology of emulsion systems is vital for food-related investigators because many natural and processed foods such as milk, salad dressing, ice cream, soft drinks, and cakes are partially or exclusively made of emulsion [1]. Emulsion formed with various ingredients and processing conditions exhibit a broad spectrum of physical and chemical characteristics that allow them to provide food systems with important functionalities where stability, texture, taste, smell, appearance, and biological response can be effectively fine-tuned to meet target requirements for any specific product.

Because of its wide occurrence in food systems, emulsion has now become a popular delivery vehicle for functional

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ingredients including color, flavor, preservatives, vitamins, minerals, and nutraceuticals [2]. The stability and efficacy of the abovementioned components could be effectively improved after being contained in the emulsion system, from which it could be protected and released into the biological system at optimum condition and timing. Depending on the type of ingredients used and requirement for different applications, emulsion is a highly versatile system that could be engineered to various sizes, surface structures, and electrochemical properties. Many studies have successfully demonstrated the development of emulsion-based systems, and results have already been summarized in many published reports [3–5].

The process of producing emulsion is called homogenization, which is achieved by applying sufficient energy to the oil/ water interface that breaks up the bulk oil into smaller droplets. In general, the smaller the size to be achieved, the larger the energy input required [6]. As in the case for macroemulsion, simple high-speed stirring is capable of forming droplets of a few micrometers. To further reduce the droplet size to submicron range, a larger energy input is required and could be supplied by various breakup mechanisms including rotor-stator, high-pressure, membrane, and ultrasonic systems [7]. Another important aspect of emulsion system is the selection of surfactant (emulsifier), which may determine not only the lowest achievable size, but also the stability and surface characteristics of emulsion droplets-that is, by alternating the combination of surface active materials and processing conditions, an emulsion system may contain different physical and chemical characteristics that would meet the requirement of target applications in food or many other fields [8].

For investigators to effectively design and produce emulsion systems that could meet their specific application needs, it is essential to realize the emulsion matrix and their droplet characteristics, which could strongly influence the physicochemical, functional, and sensory properties of such a system. Characteristics such as size, morphology, rheology, charge, and encapsulation capacity could be studied and evaluated using analytical instruments and standardized methodologies [9]. In this review, we aim to summarize and compare various analytical techniques and methods that are commonly used to assess the appearance, morphology, surface charge, and rheology of the emulsion system. The characteristics selected in this work are the parameters that are most commonly evaluated when determining the quality and predicting the suitability for a potential application. The purpose of this work is to provide a latest summary of general characterization for emulsion systems, and results can be used as a reference by investigators working in the field of emulsion science.

#### 2. Emulsion stability

An emulsion is composed of two immiscible liquids, in which one of them is dispersed as droplets into the other liquid named the continuous phase [1]. In the food industry, manufacturers develop many products, such as milk, cream, butter, and margarine, which contain emulsion as part of or the entirety of its matrix. As noted earlier, all emulsions are unstable by nature, and the two phases will eventually separate when they are allowed to stand for long enough time. The instability of emulsions may result in some undesirable effects in food including oiling off and sedimentation, which decrease the product quality and shorten shelf life. Therefore, it is important for investigators to understand the mechanisms that cause emulsion instability and to accurately evaluate the stability of such system.

Emulsion stability refers to the ability of emulsions to resist changes in its physicochemical properties over time [1]. The mechanisms that lead to emulsion instability include gravitational separation (creaming/sedimentation), flocculation, coalescence, Ostwald ripening, and phase inversion (Figure 1). The stability of emulsions is influenced by their compositional materials and processing conditions, from which different characteristics of their containing droplets would be developed. Important droplet characteristics include their concentration, size, charge, interactions, and rheological behavior [1,10]. For this reason, analytical instruments and experimental methodologies are necessary to provide information on these characteristics. In the following section, the common methods used to assess emulsion stability are summarized and compared.

#### 2.1. Visual observation

Emulsion stability affects the products' appearance, and most of the time emulsion instability can be observed directly by the naked human eye. In this sense, visual observation is probably the simplest, cheapest, and quickest method to assess the gravitational separation of the emulsion without expensive analytical instruments [11]. Gravitational separation could be classified into two mechanisms: creaming and sedimentation [1,10]. Creaming happens when the dispersed phase that has a lower density than the continuous phase moves upward and results in a thick separated layer. Conversely, sedimentation happens if the dispersed phase has a higher density than the continuous phase, causing the droplets to move downward. The extent of creaming or sedimentation can be assessed by observing the thickness of creaming or sedimentation layer with naked eyes, and then being instrumentally measured and recorded. However, visual observation, despite its convenience, is not suitable for use to study other instability phenomena, such as flocculation, coalescence, and Ostwald ripening. More often, the creaming layer is only observed when the extent of creaming is considerable. Thus, the initial stage of emulsion stability usually requires the aid of other analytical instruments to make a precise observation.

#### 2.2. Microscopy observation

As visual observation is not sufficiently capable of studying most instability mechanisms as well as droplets smaller than 100  $\mu$ m [12], microscopy is used to observe the droplets that cannot be viewed by unaided eyes and to examine the factors that influence the stability of the emulsion system. For example, using microscopy, one could easily observe the distribution and dimensions of droplets, and thus obtain information on the cause of the emulsion system's instability.

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