Experimental study of the stratification of polydisperse emulsions in a cell with heated walls

V I Valiullina*, A I Mullayanov, A A Musin and L A Kovaleva

Bashkir State University, 32 Zaki Validi Street, Ufa 450076, Russia

*E-mail:valiullina.vilena@mail.ru

Abstract. Experimental studies of the gravitational deposition of a polydisperse water-in-oil emulsion under heat influence are carried out. When the rate of thermal convection exceeds the rate of precipitation, partial delamination of the emulsion is found to occur. The viscosity of the dispersion medium decreases with increasing temperature, which contributes to an increase in the deposition rate of water droplets in the emulsion. In the presence of a temperature difference, convective flows occur in the liquid, while the drops of the emulsion coagulate and form larger agglomerates that settle faster to the bottom of the cell.

1. Introduction

Natural convection is the result of the tendency of most liquids to expand when heated, that is, to become less dense and rise as a result of increased buoyancy. The circulation caused by this effect explains the uniform heating of the liquids. Thermal motion of liquids with inhomogeneous heating is the most common type of convection. It is found not only in such complex processes as those associated with all kinds of movements in the atmosphere and hydrosphere, and those that occur underground, for example, in the mantle and core of the Earth, but also in the oil industry, for example, in the application of thermal methods for the destruction of oil-water emulsions.

An adequate understanding of the hydrodynamics of polydisperse system droplets is a prerequisite for rationalizing the overall transport processes and the stability of emulsions [1]. The stability of the emulsions is affected by a large number of variables, and the deposition rate of the dispersed phase under static conditions is a key parameter in determining the overall stability characteristics of the emulsion. Under variable external conditions, additional processes that affect the stability of the emulsions are imposed. One of these processes is the thermal convection of a liquid.

There is a large number of numerical and experimental works in the field of studying the development of convection in liquids [2, 3]. For example, in [4], the influence of thermal action on the behavior of convective flows in dispersed systems is investigated. It is found that the velocity of convective flows significantly exceeds the value of the sedimentation rate. In this case, the bulk of the particles is concentrated in the core of the flow. It is noted that the resulting convective flows prevent the separation of the suspension. In the central vertical section of the cell, a sparse region with a low concentration of particles is formed. The numerical results obtained for various Rayleigh numbers are in good agreement with the results of experimental studies. In [5], the characteristics of natural convection of oil droplets in an oil-in-water emulsion inside a rectangular vessel heated from one vertical wall and cooled from the opposite wall were experimentally studied. It was established that a set of convection layers is formed inside the emulsion, depending on the temperature difference of the walls, the size of the oil droplets and the volume fraction of the oil.
The greatest problems from the point of view of destruction arise with water-oil emulsions of the reverse type in the oil industry. It is important to take into account the contribution of thermal convection to the process of settling water droplets in the emulsion when using thermal methods of emulsion separation [6]. This work is devoted to the experimental study of the dynamics of the water stratification in oil emulsions under different methods of thermal action, including under the conditions of the occurrence of free convective flows in the emulsion.

2. Experimental procedure

Experimental studies were carried out on simplified model water in oil emulsions. Vaseline oil was used as the dispersed phase. The dispersion medium was water. The emulsion was stabilized with a surfactant Span 80. A fine emulsion consisting mainly of droplets up to 15 µm in size with an average calculated radius of water droplets in the emulsion of 16.9 µm, and a coarse emulsion consisting of droplets of 15-30 µm in size with an average calculated radius of 38.1 µm were used in the experiments.

![Photo of emulsions under a microscope and histograms of size distribution of water droplets in the emulsion: fine (upper row); coarse (lower row).](image)

Figure 1. Photo of emulsions under a microscope and histograms of size distribution of water droplets in the emulsion: fine (upper row); coarse (lower row).

Experimental studies of the dynamics of emulsion stratification under different methods of thermal exposure are carried out on an experimental setup, shown in Figure 2. The main elements of the setup are a convective cell (1), a light source (2), a camera with a macro lens (3), a videographic temperature recorder (4) and thermostats (5). The frame of the experimental cell (1 in Fig. 2) is made of polycarbonate, and the front and back walls of the cell are made of mineral glass. The dimensions of
the internal cavity in which the objects under study are placed (the emulsion) are 50x50x12mm. On the side faces of the cell there are tubes for filling the inner cavity with the emulsion and draining it, as well as five thermocouples for temperature control.

An inhomogeneous temperature field in the inner cavity is created by two aluminum tubes with a square cross-section of 12x12 mm, through which water of different temperatures is pumped using thermostats. The water temperature can vary in the range of 0-50°C. The experimental area of the cell was photographed using a Canon EOS 250D camera complete with a Tamron SP 90mm macro lens. The resulting images were processed in the MATLAB application software package according to the method described in the article [6].

3. Results

To compare the process of emulsion separation in the presence of convective flows with the isothermal process of gravitational deposition, three series of experiments were carried out. In the first series, the upper and lower faces of the convective cell had the same temperature \( T_0 \), and the isothermal case was realized. In the second series of experiments, the upper face of the cell was heated to a temperature of \( T_0 + \Delta T \) and the lower face was maintained at a temperature of \( T_0 \). In this case, an inhomogeneous temperature field was formed in the cell, and no convective flows were observed. In the third series of experiments, the lower face was heated to a temperature of \( T_0 + \Delta T \), and the upper face had a temperature \( T_0 \). In this series of experiments, thermal convection occurred in the cell.

The results of digital image processing obtained during all three series of experiments are shown in Figure 3 in the form of dependences of the height of settled water droplets on time. Polydisperse emulsions with average radii of 16.9 \( \mu m \) and 38.14 \( \mu m \) (Fig. 1) with a volume content of the aqueous phase of 5\% were used as the object. The initial temperature was \( T_0 = 23^\circ C \), the temperature difference was \( \Delta T = 5^\circ C \).
Figure 3. The dependence of the height of the settled water droplets on time for different methods of heating polydisperse emulsions with average radii: a) $r=16.9 \mu m$; b) $r=38.14 \mu m$.

The figure shows that heating the upper face of the cell for both emulsions increases the volume of separated water in comparison with the isothermal case, which is associated with a decrease in the viscosity of the carrier phase during heating (Curve 2 in Fig. 3). When the lower face of the cell is heated, the result depends on the size of the emulsion droplets. For a fine emulsion, heating the cell from below slows down the emulsion separation process (Curve 3 in Fig. 3a). This is due to the fact that when heated from below, a thermal movement of the liquid occurs in the medium. In this case, the convective flows prevent the precipitation of the emulsion droplets, and the ongoing processes of droplet coagulation do not result in the formation of sufficiently large agglomerates.

The stratification of the coarse emulsion occurs more intensively when the cell is heated from below (Curve 3 in Fig. 3b). This is because the convection that occurs in the medium involves large drops in the direction of the action of the gravity vector, that is, from top to bottom. At the same time, the flow force for lifting large drops up is insufficient. In addition, convective flows can contribute to the formation of large agglomerates of droplets, which then quickly settle without being involved in the convective flow.

In addition, the separation of a fine emulsion with different initial concentrations of 1% and 5% was studied when the cell was heated from below with a temperature difference of 5 and 10 °C. At the initial time, the water droplets in the emulsion gradually settled. The concentration of water droplets in the upper part of the cell decreased, while at the bottom it increased. Partial delamination of the emulsion was observed. However, the rate of thermal convection of the liquid increased over time, and the convective flows began to interfere with the deposition of water droplets in the emulsion. The convective fluid flow intensively mixed the emulsion system. The drops of the emulsion were drawn into the liquid flow and created a translational motion along the velocity vector of the thermal motion of the liquid.

At the initial time $t = 0$, the emulsion droplets were fairly evenly distributed over the volume of the convective cell. Over time, the emulsion was separated, and a layer of water droplets formed at the bottom of the cell. The dependences of the height of the separated water layer on time were constructed, as shown in Figure 4 as a result of photo processing.
Figure 4. Layer growth dynamics of the settled dispersed phase of the fine emulsion (r = 16.4 μm) at different temperature differences (ΔT = 5°C and 10°C) and different concentrations of the emulsion: a) C = 1%, b) C = 5%

The figure shows that for a finely dispersed emulsion with an initial concentration of 1%, an increase in the temperature difference results in a small increase in the volume of separated water (Fig. 4a). A much greater effect of the change in the temperature gradient on the process of emulsion separation is observed for an emulsion with an initial concentration of 5% (Fig. 4b): an increase in the temperature difference between the lower and upper faces of the convective cell leads to more than a twofold increase in the thickness of the settled water layer. The rate of separation of the emulsion also increases. This is due to a decrease in the viscosity of the surrounding liquid droplet in the process of the temperature increase. The rate of droplet deposition increases faster than the intensity of convective flows in the liquid, which involve the droplets in the flow.

Conclusions
Experimental results of the study of the separation of emulsions under the thermal influence have been obtained. It has been observed that at small sizes of water droplets in the emulsion, when the rate of thermal convection exceeds the rate of precipitation, a partial delamination of the emulsion occurs. This is due to the fact that convective flows begin to prevent the deposition of water droplets in the emulsion. At large sizes of water droplets in the emulsion, the sedimentation rate always exceeds the speed of convective flows, while there is an unhindered deposition of water droplets in the emulsion. The increase in the efficiency of the emulsion delamination under thermal influence occurs for two reasons. Firstly, with an increase in temperature, the viscosity of the dispersion medium decreases, which contributes to an increase in the deposition rate of water droplets in the emulsion. Secondly, in the presence of a temperature difference, convective flows occur in the liquid, while the emulsion droplets coagulate and larger agglomerates are formed and settle faster to the bottom of the cell. Therefore, the effect is more noticeable for emulsions with a high concentration at the initial stage of the delamination process.

Acknowledgments
The reported study was funded by the grant of the Russian Science Foundation (project No.19-11-00298).

References
[1] Nigmatulin R I 1990 CRC Press 369.
[2] Tamar G and Silverstein M S 2011 Polymer 52(1) 107–15
[3] Dragsted J et al. 2017 Solar Energy 147 414–25
[4] Tukhbatova E R et al. 2019 IOP Publishing 1359(1) 012112
[5] Morimoto Takashi, Toshiki Ikeda and Hiroyuki Kumano 2018 International Journal of Heat and Mass Transfer 127 616–28
[6] Valliullina V I et al. 2020 IOP Publishing 1675(1) 012025