Process Intensification of Oily Wastewater Treatment by Ionic Microbubble in a Plant Prototype

Subrata Kumar Majumder and Gulshan Kumar

***Abstract*—**The present work reports the efficiency of ionic micro bubble in a plant prototype developed to separate the oil from oily wastewater. It is observed that percentage of oil recovery increases with decrease in bubbles size. Oil recovery firstly increases then decreases by addition of floating agent. The efficiency is interpreted based on the mixing of the fluid particles with the different operating variables. A model is also proposed to analyze the process efficiency based on the mixing characteristics.

***Index Terms*—** Ionic microbubble, Mixing, Oil, Wastewater.

I. Introduction

Any chemical engineering development that leads to a substantially smaller, cleaner and more energy efficient technology is process intensification. The aim of intensification is to optimize capital, energy, environmental and safety benefits by reduction in the physical size of the plant. Mining, metallurgical, petroleum and chemical industries generate huge amounts of wastewater which are usually polluted by solids, process chemicals, organic and other compound. During crude oil exploration and production large volumes of petroleum hydrocarbon bearing effluents, the so-called produced waters, are concurrently recovered. These waters usually contain high salinity, suspended solids (clay, sand, scale corrosion products) and oils. These oils can recover by various means. Oily wastewater can be cleaned by gas microbubble flotation. While using microbubbles it is seen that oil separation efficiency is increased substantially. It happens because of increase in air and oil interfacial area. Properly operated gas microbubble flotation units can reduce oil concentrations of wastewater effluents below 40 mg/l. Gas microbubble flotation is particularly valuable for heavy oils (oils having a density close to that of water). The flotation/oil separation process relies on the attachment of gas bubbles to the dispersed oil droplets. This attachment is heavily dependent on the complex processes involving the surface characteristics of the oil droplets and their interaction with gas. It can only be optimally achieved if the surface science conditions are properly understood. The attachment mechanisms include the oil/bubble contact, the interactions of chemical additives (usually surfactants) and the spreading of the oil around the gas bubble.

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Various authors reported their different studies done on the mixing characteristics and oil separation in flotation column. Strickland [1] studied the use of induced air flotation to recover oil from sludge containing oil. The oil recovery increased by a maximum of 12% when the surfactant amount added increased from 5 to 20 g. Xu et al. [2] applied the axial dispersion model for fitting residence time distribution in flotation columns and analysis of flotation rate to effect on the recovery efficiency of effluent based on the intensity of mixing. Mavros and Daniilidou [3] studied the mixing characteristics and axial dispersion coefficient in flotation columns. They observed that dispersion number mostly depend on column diameter and liquid velocity. Intensity of mixing is less for narrow column as there is less free space to disperse while intensity of mixing is more for large column diameter due to intense recirculation phenomena which governs the efficiency of the separation. They also determined the dispersion number and characteristic of mixing in flotation column to analyze the recovery efficiency of the effluent in flotation column. Zouboulis and Avranas [4] studied about treatment of oil-in-water emulsions by coagulation and dissolved air flotation. At optimum experimental condition (recycle ratio: 30 %, pH: 6, sodium oleate: 50 mg/l) more than 95% of the emulsified oil was effectively separated from an initial concentration of 500 mg/l. Al-Shamrani et al. [5] studied oil recovery from industrial effluent containing oil content by dissolved air flotation. Operating parameters including saturator pressure, recycle ratio, and air to oil ratio, were investigated to find conditions that would enhance the efficiency of the DAF separation. A maximum of 90 % saturator efficiency was observed. Xiao-bing et al. [6] studied oil separation from waste water by dissolved air flotation column. Dissolved air release occurred within the column separation system. A high separation efficiency (more than 90%) was obtained in a series of tests. Bande et al. [7] studied oil field effluent treatment by electro flotation. Oil removal efficiency was 90% at 4.72 pH in 30 min treatment time for 50 mg/l concentration of oil and 94.44% of oil removed within 30 min at 4 mg/l of salinity. Beneventi et al. [8] measured the mixing characteristics and the bubble size of a Venturi aerated laboratory flotation column in the presence of simplified model systems and of industrial pulp slurry. It was observed that surfactants dissolved in the pulp slurry stabilized air bubbles while cellulose fibres promoted coalescence and affect the separation process. Yianatos et al. [9] studied the measurement of the axial gas holdup profile and gas residence time distribution (RTD), in self-aerated flotation cell. Shukla et al. [10] studied gas holdup and pressure characteristics in a column flotation cell to separate the coal particles. Xiaohui et al. [11] showed oil removal technique by dynamic state micro bubble flotation which is used for water treatment on offshore oil platform. Experimental result shows that the micro bubbles generated by micro pore membrane reduce oil content by more than 60%. For the separation of oil, hydrodynamics like gas holdup, mixing of fluid element and its residence time are very crucial. Le et al. [12] measured the oil separation from oil in water emulsions with the help of micro bubbles enhanced with normal cyclone bubbles. The treatment by a combination of MBs with NBs was more efficient for emulsified oil (EO) separation than was treatment by MBs alone.

Based on literature review it is observed that oil separation can be improved by various parameters and by adopting various methods. The separation by microbubble is one of the efficient process to separate the oil from wastewater due to its huge interfacial area. Process can be intensified by modifying parameters and adding new parameters. In the present study an attempt has been made to study the efficiency of the microbubble to recover the oil from an oily wastewater effluent in a plant prototype.

II. Experimental

The experimental setup consists of a glass flotation column (150 cm × 20 cm × 20 cm), microbubble generator, liquid rotameter, gas rotameter, manometer and conductivity meter as shown in Fig. 1. Glass flotation column consists of three holes in left side are made for input of feed. On the top of glass column a provision is made for collecting the froth/oil content. A manometer has been attached in back portion for measuring the pressure in column. One liquid rotameter is attached in inlet line. A microbubble generator has been attached in bottom portion of flotation column and it is connected with compressor. A gas rotameter is attached in incoming compressed air line to measure the gas flow rate. Liquid/Oil-water mixture is sucked from feed tank through pump and sent inside flotation column. An exit line is made inside the bottom of flotation column. Once steady-state was reached and when the flow is fully developed, the tracer, 40 ml of Sodium chloride solution of concentration 1 M was injected at a point 30cm below the top of the column. Liquid was collected in test tubes at every 9 s interval at some downstream position of the column just after the injection of the tracer and measured by conductivity probe. The experiments were conducted at four different liquid flow rates 400, 600, 800, 1200 lph and three different gas flow rates 1, 2 and 3 lpm. At steady-flow of gas and liquid pressure readings were noted from the manometers connected to the column. Materials used for preparing emulsion was lube oil (density: 0.870 g/l) and tap water. EO samples with varying concentrations of 793.79 – 3146.47 mg/l were prepared firstly by mixing method using common mixer grinder having 18000 rpm measured by a digital Tachometer.

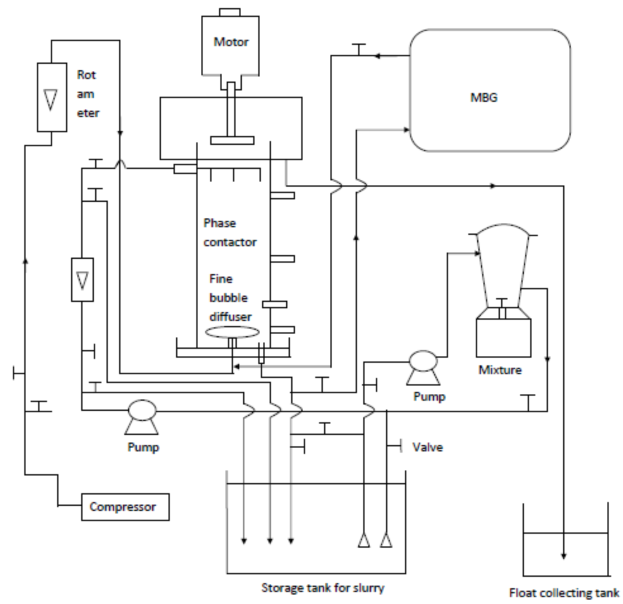

Fig. 1 Plant prototype of oily water cleaning

Oil was mixed with water for nearly 25-30 minute inside mixer grinder. Its color changes are almost whitish. Then this emulsion was poured inside feed tank which contains fixed amount of water (54.6 l). It was circulated vigorously throughout column and feed tank up to 5 minutes so that it was properly mixed with water. Experiments were carried out by varying EO concentrations, gas flow rates, surfactants. Surface phenomenon occurs between oil particles and air bubbles. Oil particles spread over air bubbles and slowly moves towards upward direction. Oil was recovered from top at different time intervals and collected in graduated cylinder which gives an idea about the trend of oil recovery with time and other parameters. Four different types of emulsion concentrations were prepared by varying parameters.

Initially surfactant concentration was fixed and gas flow rates were varied. Later experiments were carried out by varying amount of surfactant. Oil separation efficiency was also measured by varying salt concentration. Oil droplet size is one of the important factors in oil separation that affects oil separation efficiency. Oil droplet size was measured for different concentrations. It was found that by varying concentration of emulsion from 793.8 mg/l to 3146.47 mg/l, oil droplet size varies from 30 μm to 70 μm. It has been observed that rising velocity is very much affected by droplet size. Rising velocity increases by increasing droplet size.

III. Theory

Intensity of axial dispersion of liquid expressed as Peclet number (Pe) which can be estimated using residence time distribution (RTD) technique. The normalized RTD curves have been proposed by Levenspiel by using the open boundary conditions as follows

$$\frac{C_T}{C_{T0}} = \frac{1}{2\sqrt{\pi\theta / Pe}} \exp\left[\frac{-(1-\theta)^2}{4\theta / Pe}\right] \quad (1)$$

C_T is the tracer conentration in kg/m^3. C_{T0} is the initial tracer conentration in kg/m^3. The parameter θ is the dimentionless time, t/t_m. t_m is the mean residence time. The Peclet number, Pe is calculated using the following equations:

$$\frac{\sigma^2}{t^2{}_m} = \frac{2}{Pe} + \frac{8}{Pe^2} \quad (2)$$

where

$$t_m = \sum (t_i C_{Ti}) / \sum C_{Ti} \quad (3)$$

$$\sigma^2 = \frac{\sum (t_i - t_m)^2 C_{Ti} \Delta t_i}{\sum C_{Ti} \Delta t_i} \quad (4)$$

The variance (σ^2) represents the square of spread of the distribution as passes the column exit. Longitudinal dispersion coefficient E_z in m^2/s was calculated from equation

$$E_z = \frac{u_{SL} z}{(1-\varepsilon_g) Pe} \quad (5)$$

where z is the length between tracer injection and tracer collection port. Percentage of oil recovery can be estimated by

$$\% R = \frac{C_f}{C_i} \times 100 \quad (6)$$

where C_f is volume of oil at different time interval and C_i is the initial oil volume making emulsion, R is the removal capacity.

IV. RESULTS AND DISCUSSION

Analysis of Residence time Distribution

Experimentation on RTD for mixing phenomena has been done in air-water liquid system with NaCl tracer. The result has been analyzed with axial dispersion model. It is found that the intensity of the mixing in the flotation column depends on different operating variables like gas flow rate, liquid flow rate, surfactant concentration, emulsified initial oil concentrations and operation time. In the Fig. 2, the effect of liquid velocity on the residence time of tracer particles has been shown. It is found that just after the injection of tracer, up to a certain time the concentration of the tracer particles is zero in the sample collected. It is also observed that the residence time of the tracer particle decreases when the gas velocity is increased. This is due to the fact that at high gas velocity the momentum energy in the liquid-gas mixture increases which increases the dispersion and reduces the residence time. The residence time of the fluid decreases with increase in gas velocity. This is due to the increase in axial dispersion with the increase in gas velocity. When liquid flow rate increases the turbulence in the column increase and momentum exchange in the fluid element also increase. The elemental fluid velocity also increases with increasing flow rate, so the high velocity fluid element tends to pass the column very fast, so their spent in column decreases and hence residence time decreases. The variation of longitudinal dispersion coefficient with the liquid flow rates is shown in Fig. 3 and it is observed that with the increase of liquid velocity the dispersion coefficient increases. Longitudinal dispersion coefficient is proportional to fluid velocity and inversely proportional to the Peclet number ($Pe = u_{iL}/E_z$).

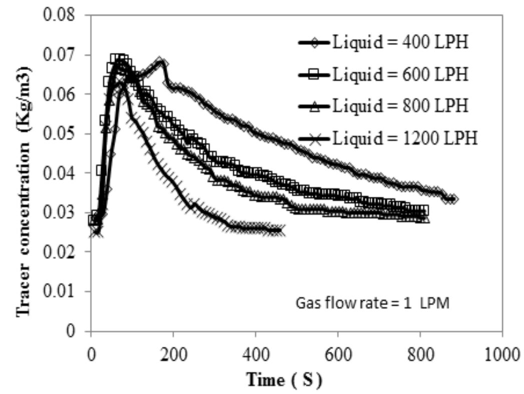

Fig. 2 RTD of tracer particle with liquid velocity

At higher liquid velocity, the gas holdup increases which enhances the turbulence of the gas-liquids mixture in the column. The more gas holdup results more bubbles. More bubbles increase the interactions among them which results more turbulence of gas liquid mixture in the column and consequently increases the dispersion.

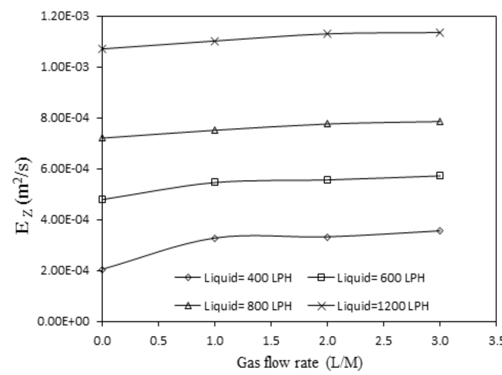

Fig. 3 Variation of longitudinal dispersion coefficient with gas flow rate

Oil Recovery Analysis

Oil recovery increases by increase in EO concentration. At the EO concentration of 793.7956 mg/l, 1582.818 mg/l, 2368.421 mg/l and 3146.474 mg/l, EO removal efficiency increases for a particular gas flow rates after 2 hours of treatment as shown in Fig. 4. This indicated that the performance of flotation method is effective even under a high load of EO present in column. Effect of flotation time on amount of oil removed has been studied at different gas flowrates and SDS concentrations. Trend of results show that oil recovery increases as time passes. It has also been observed that oil separation is more in initial stages and rate of separation decreases with time. Oil droplets are present in various sizes in EO so it is easier to separate the larger oil droplets in comparison to smaller one. For gas flow rates of 1, 2.5, 3.5 & 6 l/m recoveries are almost more than 52% in first 30 minutes. For gas flow rates of 1, 2.5, 3.5 & 6 l/m recovery are almost more than 70% in first 30 minutes for an initial emulsified concentration of 1.58 g/l as shown in Fig. 5.

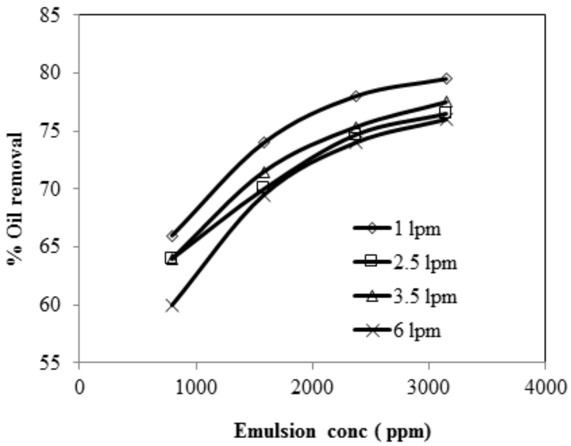

Fig. 4. Effect of EO concentration on oil removal

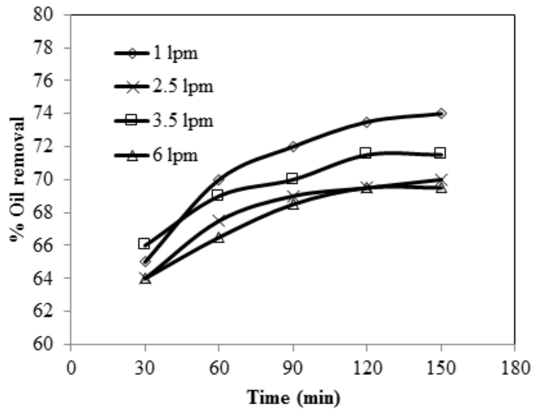

Fig. 5. Effect of time on oil removal at 1.58 g/l EO concentration

It is also observed that percentage recovery increases with time of operation. Rate of recovery is higher at initial stages then it decreases and becomes almost constant after 90 minutes in almost all cases. Addition of surfactant to EO affects size, number and adhesion firmness of fine bubbles. Water surface tension is reduced by adding surfactant. The surface energy of fine bubbles decreases when surfactant is present at the interface between gases and liquid. Beside this, size of fine bubbles is further reduced and stability of froth increase as bubbles tend not to agglomerate. It can be seen from the Fig. 6 that oil removal efficiencies were 63.3 %, 67.3 %, 75.3% when sodium dodecyl sulfonate present in EO were 0, 7.4, 10 ppm respectively.

Model Development for Intensity of Mixing

Axial Dispersion coefficient in dissolved air flotation column is found to be affected by many parameters such as gas velocity, liquid velocity, column diameter, pressure etc. In the present work liquid velocity and gas velocity are the main variable affecting the axial dispersion coefficient. Hence an attempt has been made to correlate the effect of these variables on dispersion coefficients. By Taylor's theory, dispersion due to velocity profile for single phase flow is expressed by an equation,

$$Pe = 1.3\,\mathrm{Re}_l^{\,0.1}(z/d_{eq}) \quad (7)$$

The parameter Re_l is the liquid Reynolds number.

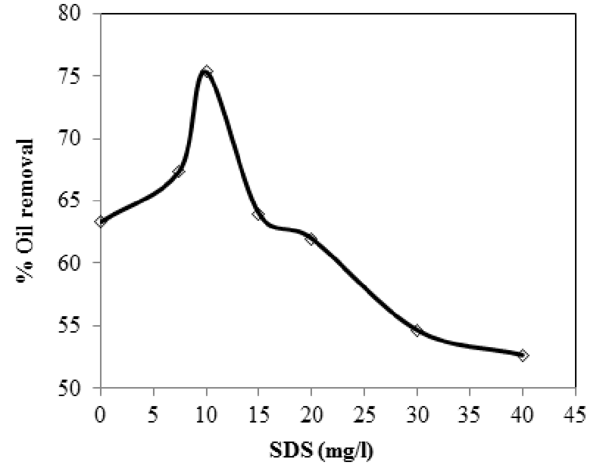

Fig. 6. : Effect of SDS on oil recovery

The equivalent column diameter for square crosssectional area is denoted by d_{eq}. For the Reynolds number >10^4 dispersion coefficient can written as [13]

$$E_z = 0.25 d_{eq} u_{sl} \quad (8)$$

The u_{sl} is the superficial liquid velocity. The Peclet number for single phase flow is expressed as

$$Pe = \frac{u_{sl} z}{E_z} \quad (9)$$

From equation (7) and (8), for single phase flow the following expression can be written

$$\frac{u_{sl} z}{E_z} = 1.3\,\mathrm{Re}_l^{\,0.1}\left(\frac{z}{d_{eq}}\right) \quad (10)$$

$$E_z = \frac{u_{sl} d_{eq}}{1.3\,\mathrm{Re}_l^{\,0.1}} \quad (11)$$

For two-phase flow Joshi [14] introduced the concept of average liquid circulation velocity and present a general correlation relating dispersion coefficient, column diameter, superficial liquid velocity and average liquid circulation velocity (u_c), which can be written as per concept of single phase as

$$E_z = 0.31 d_{eq}\left(u_{sl} + u_c\right) \quad (12)$$

For two-phase the equation (11) can be written as

$$E_z = \frac{d_{eq}\left(u_c + u_{sl}\right)}{1.3\,\mathrm{Re}_l^{\,0.1}} \quad (13)$$

The Peclet number for two phase flow can be written as

$$Pe = \frac{u_{sl} z}{E_z\left(1 - \varepsilon_g\right)} \quad (14)$$

Now combining equations (13) and (14) one can get

$$Pe = \frac{1.3\,\mathrm{Re}_l^{\,0.1}\,L}{d_{eq}(1-\varepsilon_g)\left[1+u_c/u_{sl}\right]} \quad (15)$$

Now from the above relation one can calculate the experimental value of u_c. Since u_c depends on u_{sl}, u_{sg}. So by dimensional analysis, a relation can be written as

$$\frac{u_c}{u_{sl}} = a\,\mathrm{Re}_l^{\,x}\,Fr_g^{\,y} \quad (16)$$

where *a*, *x* and *y* are constants. $Fr_g = u_{sg}^2/(gd_{eq})$ and $\mathrm{Re}_l = u_{sl}d_{eq}\rho_l/(1-\varepsilon_g)E_z$. By regression analysis the equation (16) can be written as

$$\frac{u_c}{u_{sl}} = 0.199\,\mathrm{Re}_l^{\,0.485}\,Fr_g^{\,0.129} \quad (17)$$

Substituting equation (17) into equation (15) one can get

$$Pe = \frac{1.3\,\mathrm{Re}_l^{\,0.1}\,L}{d_{eq}(1-\varepsilon_g)\left[1+0.199\,\mathrm{Re}_l^{\,0.485}\,Fr_g^{\,0.129}\right]} \quad (18)$$

The model predicts the experimental data for Peclet number within the range of present experimental condition with correlation coefficient and standard error of 0.985 and 0.01 respectively. Once the Peclet number is predicted by the equation (18), using this value one can predict the recovery efficiency of the oil by the following kinetic equation

$$\mathrm{R}\,(\%) = (1 - \frac{4a\exp(Pe/2)}{(1+a^2)\exp(aPe/2)-(1-a^2)\exp(-aPe/2)})\times 100 \quad (19)$$

Where the parameter (a) is defined as

$$a = \sqrt{1+4kt/Pe} \quad (20)$$

The parameter *k* is called flotation rate constant which is defined by

$$k = 3P_cP_au_{sg}/(2d_b) \quad (21)$$

The terms P_c and P_a are probability collision and attachment

$$P_c = 1.5[1+\frac{(3/16)\,\mathrm{Re}_b}{1+0.249\,\mathrm{Re}_b^{\,0.56}}] \quad (22)$$

The Reynolds number Re_b based on the bubble diameterwhich is defined as

$$\mathrm{Re}_b = \frac{u_bd_b\rho}{\mu_l} \quad (23)$$

$$u_b = u_{sg}/\varepsilon_g \quad (24)$$

The gas holdup in the column is defined by

$$\varepsilon_g = 1-\Delta P/(\rho_m gh) \quad (25)$$

where ΔP is the pressure drop, ρ_m is the mixture density and *h* is the mixture height in the column.

$$P_a = \sin^2[2\tan^{-1}\exp(-(\frac{45+8\,\mathrm{Re}_b^{\,0.72}\,u_bt_i}{15d_b(1+d_b/d_p)}))] \quad (26)$$

The bubble diameter d_b can be calculated by

$$d_b = (0.5We_c\sigma)^{3/5}(\rho_l)^{-1/5}(\in)^{-2/5} \quad (27)$$

where $\in$ is the energy dissipation rate per unit mass of fluid for the operation which can be calculated as

$$\in = u_{sg}\,g \quad (28)$$

The drop or bubble size is dependent on the critical weber number. In the present case the critical weber number (We_c) is considered as 1.2.

V. Conclusion

In the present work an attempt has been made to study the mixing characteristics in flotation column and oil recovery from oil in water emulsion. Oil separation depends on residence time of oil droplets and air bubbles. It has been found that the mixing phenomenon and oil separation in the flotation column is affected by many variables such as gas flow rate, liquid/feed flow rate, and column pressure. The gas holdup in flotation column strongly depends on liquid flow rate. The mean residence time of the fluid decreases when the liquid velocity is increased. The mean residence time of the fluid decreases when the gas velocity is increased. The longitudinal dispersion coefficient of liquid increases with increasing either gas velocity or liquid velocity. Size of air bubble droplets increases as gas flow rate increases. Oil separation strongly depends on size of air bubble droplets and oil droplets. Oil separation increases rapidly by addition of surfactants but after certain concentration it decreases. The model developed can be used to scale-up the process to the industrial scale though more studies with different surfactant is required. The flow regime and hydrodynamics of oil and bubble attachment has yet not been studied. Variation of different operating variables can be studied in the future to expand area of research. Effect of emulsifying and demulsifying agents can also be studied more deeply as it is directly related to industrial work. Oil separation process can be further improved by decreasing air droplet size to micro and nano level. Beside this effect of various gas droplets on emulsion can be studied.

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