Separation of oil/water emulsions by microbubble air flotation

Sumaya L Al-dulaimi1,2, Atheer M Al-yaqoobi1

1 Chemical Engineering Department, College of Engineering, University of Baghdad, Baghdad, Iraq
2 E.mail: Sumayaliwa44@gmail.com

Abstract. Oil/water emulsions are one of the major threats to environment nowadays, occurs at many stages in the production and treatment of crude oil. The oil recovery process adopted will depend on how the oil is present in the water stream. Oil can be found as free oil, as an unstable oil/water emulsion and also as a highly stable oil/water emulsion. The current study was dedicated to the application of microbubble air flotation process for the removal of such oily emulsions for its characters of cost-effective, simple structure, high efficiency and no secondary pollution. The influence of several key parameters on the process removal efficiency was examined, namely, initial oil concentration, pH value of the emulsion, and the effect of adding sodium chloride. The effect of bubble size on the performance of the separation process and its impact on removal efficiency was also investigated. The results demonstrated that removal efficiency obtained by using microbubbles flotation was higher by factor of 1.72 in comparison with that achieved with fine bubbles. The removal efficiency of oil droplets was increased with the increasing of flotation time and initial oil concentration. The removal efficiency reached up 60.68% under alkaline conditions (pH≈9), and it increased to around 75% by decreasing the emulsion acidity to around (pH≈3). The addition of sodium chloride has a significant influence to the efficiency of the flotation process. The efficiency could be reached to about 84% by adding 1 gL⁻¹ of NaCl to the emulsion. While increasing the NaCl concentration to 9 gL⁻¹ resulted in reduction in removal efficiency to around 80%.

Key words: Flotation, Microbubbles, Bubble diffuser, Oily wastewater, Emulsion, Oil removal efficiency, Zeta potential.

1. Introduction
Oil wastewater refers to wastewater that has been mixed with oil at a wide variety of concentrations [1]. Pollutants in oily wastewater are classified into two categories, organic and inorganic, as tabulated in Table 1. A significant quantity of oily wastewater is generated daily from a number of sources including oil refineries, petrochemical industry, crude oil production, oil and natural gas production plants, metal processing, machining, automotive shops, oil-drilling, and many other processes [2–4]. Such waste is mostly in the form of an oil-in-water (O/W) emulsion or a water-in-oil (W/O) emulsion or a water-in-oil-in-water (W/O/W) emulsion [5]. The different forms of the emulsion are shown in Figure 1. The United States Environmental Protection Agency (USEPA) has listed emulsions of oil-in-water among the most hazardous sewers owing to excessive levels of oil and grease, aromatic compounds, heavy metals, salts and recalcitrant substances which are hard to handle naturally in the environment [6]. Huge volumes of oil/water emulsion or oily wastewater effluents are dumped into the river from industries. Once they reach the sea, they have an adverse effect on the environment and...
living aquatics. Pollution of oily wastewater impacts drinking water, groundwater and sea water as a result of the percolation of pollutants into the water supplies below the surface. This process also puts human health in risk, as it tends to contaminate the atmosphere, particularly when oil burners are included. In addition to being toxic, oily wastewater includes petroleum hydrocarbons, polyaromatic hydrocarbons, and phenols, which can limit animals and plants growth. This pollutant increases the risk of mutations and cancer for humans [7–10].

Many countries have imposed strict regulations on oily wastewater disposal as they inflict serious and long-term environmental and human health harm [11]. For example, in Canada, the approved discharge limit for dispersed oil and grease for treated produced water is 30 mg/L in daily average [12]. According to the U.S. EPA regulations, the daily maximum and monthly average limit for oil and grease are 42 and 29 mg/L, respectively [13]. The oil phase in the wastewater usually consists of at least four types depending on the size and stability of the oil droplets. These types are free floating oil with a droplet diameter larger than 150 μm, dispersed oil with droplet sizes ranging from 20 to 150 μm, emulsified oil usually has a droplet size below 20 μm, and dissolved oil that has droplets smaller than 5 μm in size [3, 12, 14]. Free oil is not a major concern, as oil can easily be separated by gravity settling; however, the emulsified oil presents a particular challenge due to presence of steric interaction or structural barriers and electrostatic repulsion between oil droplets [9, 15].

So far, several researchers from around the world have suggested a large number of methods for treating such forms of oil in water, including gravity separation [16], centrifugation [17], the use of skimmers [18], adsorption [19], coagulation-flocculation [20], membrane processes [21], ultrafiltration [22], Microfiltration [23], reverse osmosis [24], Chemical demulsification [25], biological media [26], activated sludge [27], hydrocyclone [28], Coalescing media [29] and combinations of the above. Between them, air flotation technology was pretty widely used to remove oil from oily rejections in the whole world and still presents remarkably high potential related to its high separation efficiency and throughput, energy-efficient, simple process from an operational viewpoint, cost-effective, applicable to a wide range of oily wastewater and no by-product [30–32].

Air flotation is a method of gravity separation [14, 33]. It can be used to obtain higher separation efficiency (up to 80%) even at high loading rates with low retention times [34]. Air flotation is practiced in many fields such as wastewater treatment, mineral beneficiation, fermentation, ink removal, plastic recycling, protein separation, odor removal, harvesting or removal of algae, separation or harvesting of micro-organisms [35, 36]. In this method, components such as small particulate matter, oil droplets and contaminants are removed from the mixture depending on their hydrophobic or hydrophilic surface properties [33, 36]. Air bubbles are used to pick up hydrophobic molecules and transport them to the liquid surface, thereby creating a foaming layer where it can be removed, while hydrophilic particles are released from the bottom section like waste [37].

Generally, the technique of air flotation is divided into dissolved air flotation (DAF) and induced air flotation (IAF). Both techniques vary according to the way air bubbles are produced and the resulting bubble sizes [38]. In DAF units, air is pumped into the flotation chamber which is filled with a fully saturated solution. The air is expelled inside the chamber by applying a vacuum, or by attempting to create a rapid pressure drop. IAF technology uses mechanical shear or propellers to produce bubbles that are presented to the lower part of the flotation chamber [39, 40].

Numerous researches have been done on the treatment of oily wastewater by using flotation process. [41] studied the use of air flotation technique to separate crude oil from polymer-produced water. Approximately 99% of the oil was effectively removed at optimum experimental condition and in the presence of an anionic polymer (GLP-100). [42] used a bubble column of 15.6 cm diameter and 120 cm height to separate emulsified kerosene in water and found that the removal rate improved at a higher air velocity and a lower water height. [43] investigated the ability of separation of pollutants from oil-containing restaurant wastewater by applying Novel microbubble air flotation (MAF), comparing the results with traditional dissolved air flotation (DAF) systems. The maximum oil removal efficiency was achieved in the MAF system when the microbubbles and oil-droplets were similar sizes. [44] studied the separation of oil-in-water emulsions by the flotation method under three
cases: MB treatment only, MB treatment with polyaluminium chloride (PAC) as a coagulant (MB–PAC), and MB treatment with cetyltrimethylammonium chloride (CTAC) as a cationic surfactant (MB–CTAC). MB treatment with PAC and MB treatment with CTAC were seen to be more effective in the separation of emulsified oil than the MB treatment alone.

The essential goal of the current work was to investigate the possibility and efficiency of microbubble air flotation method for oil/water emulsion removal. This study investigated influence of some of key parameters which include initial oil concentration, pH, and effect of adding sodium chloride on the process removal efficiency. Also, the effect of bubble size on the performance of the separation process and its impact on removal efficiency was studied.

Table 1. Classification of oily wastewater pollutants [45].

| Properties               | Organic Pollutants                          | Inorganic Pollutants                      |
|--------------------------|---------------------------------------------|-------------------------------------------|
| Major component          | Petroleum hydrocarbons                      | Inorganic oils                            |
| Types                    | Aliphatic, aromatic, asphaltenes            | Hydraulic, turbine, lubricating, cutting, motor oil |
| Major element            | Oxygen, nitrogen, Sulfur                    | -                                         |
| Accompanying element     | Nickel, cadmium, lead, vanadium organometallic complexes | Gasoline, heavy metals, oily sludge, solvents, particulate matters |
| Distribution in oily wastewater | Dispersed, emulsified, or dissolved  | Floatable or settleable                   |

Figure 1. Forms of emulsions [46].

(a) Oil-in-water (O/W)   (b) Water-in-oil (W/O)   (c) Double emulsion of water-in-oil-in-water (W/O/W)
2. Materials and methods

2.1. Crude oil
In this study, a sample of Kirkuk crude oil was used in the preparation of emulsion provided by a local oil refinery (Al-Dura Refinery, Iraq) and some of its physical properties are summarized in Table 2.

2.2. Chemicals
Surfactant type span 85 supplied by Fluka AG (USA), surfactant type tween 80 supplied by Alpha Chemika (India), Hydrochloric acid (HCl, 38%) from Central Drug House (P) Ltd. (India), Sodium hydroxide (NaOH, 99%) from Applichem GmbH Olloweg D-64291 Darmastdt (Germany), Sodium chloride (NaCl, 99%) from HiMedia Laboratories Private Limited (India), n-Hexane (C₆H₁₄, 95 % ) from Alpha Chemika (India), Xylene (C₈H₁₀, 99.8 %) from Sisco Research Laboratories Pvt. Ltd. (India), Ethanol (C₂H₅OH) from AAG (India) and tap water were used in this work.

2.3. Preparation of Oil/Water Emulsion
Oil/water emulsion was prepared from a petroleum crude oil, 1%wt of surfactants and tap water. Non-ionic surfactants span 85 and tween 80, were used (60% span and 40% tween) to obtain hydrophilic lipophilic balance (HLB) value about 7, that made the emulsion stable for several days [47, 48]. Firstly, adding a measured amount of untreated crude oil and mixed it with surfactants and tap water in a mixing tank. Thereafter this mixture was agitated for ten minutes using 10,000 rpm homogenizer. After agitating, the pH of prepared emulsion was adjusted by adding (0.1 M) HCl or (0.1 M) NaOH and measured by using HM digital pH-200 Waterproof Professional Series pH/Temp Meter. The oil content in oil/water emulsion was determined by TD-500D Oil in Water Meter from Turner Designs Hydrocarbon Instruments (USA).

2.4. Equipment and procedure
The experiments were performed using the laboratory setup shown in Figure 2, which consisted of a cylindrical flotation column made from transparent acrylic resin with a capacity of 50 L and with dimensions (160 cm in height, 20 cm in diameter). The column equipped with five sampling ports placed equally spaced along the column. An air compressor was used to supply a constant air stream to the flotation column. The air introduced to the column through submerged sparger consists of a ceramic micrombubble diffuser (MBD) (type Point Four) with dimensions (16 cm in length, 6 cm in width and 1 cm in height) with an average pores size less than 20µm. This type of diffuser is capable of producing micro scale bubbles. The sparger set at the lower part of the flotation column used for producing the air microbubbles. In case of studying different bubble size, the diffuser was replaced by a perforated Teflon plate with pores size equal to 0.5 mm.

The oil/water emulsion was introduced to the flotation column from the top after the emulsion was adjusted for the desired concentration, volume, and acidity according to the experimental conditions. An initial sample was taken from the emulsion before the air is pumped to the system. Air was introduced from the bottom of the flotation column through the submerged diffuser at the required flow rate. The flotation time was 4 h for all experiment. Samples were taken through the sampling point every 15 minutes and they were immediately tested using the TD-500D UV-fluorescence analyzer for determining the final oil concentration. Then the oil removal efficiency is calculated according to equation (1)

\[
\text{Oil Removal Efficiency} = \frac{c_i - c_f}{c_i} \times 100 \%
\]

Where \(c_i\) is the initial oil concentration before the treatment (ppm) and \(c_f\) is the final oil concentration after the treatment (ppm). At the end of flotation process, the remained liquid was drained out from the system. The top oily layer which is composed of oil, bubble, oil-bubble complex was skimmed off and removed. Column, mixer, and diffuser were cleaned carefully to remove any oil contaminations.
Figure 2. Schematic diagram of microbubble air flotation system. (1) Flotation column, (2) Sampling ports, (3) Air compressor, (4) MB diffuser, (5) Pressure gauge, (6) Air rotameter, (7) Emulsion tank, (8) Feed pump, (9) Discharge pump, (10) Pressure control valve, (11) Regulating valve, and (12) Drain valve.
Table 2. Physical properties of Kirkuk crude oil.

| Parameters       | Value                                      |
|------------------|--------------------------------------------|
| API              | 31                                         |
| Density          | 0.8504 g/cm$^3$ (at 15.6 °C)               |
| Sp. Gr           | 0.8509 (at 15.6 °C)                        |
| Salt content (wt%)| 0.0009                                     |
| Asphaltene (wt%) | 1.29                                       |
| Kin. Viscosity   | 11.6 at (15°C)                             |
| Sulfur content (wt%) | 2.33                              |
| Nickel content, ppm | 10.25                                      |
| Vanadium content, ppm | 30.8                                       |

3. Result and Discussion

3.1. Effect of Bubble size

Effect of bubble size on oil removal efficiency was conducted by using two bubble diffusers of different pore size, microbubble ($< 100 \mu m$) and fine bubble ($< 1 mm$) sizes. Compared the results of these two diffusers with gravitational separation (no bubbles). Experimental conditions were kept the same (initial oil concentration = 200 ppm, pH = 7.32, air flow rate 0.5 L/min, air pressure = 0.2 bar and liquid height = 55 cm).

Figure 3 shows the oil removal efficiency with time for the case of microbubble, fine bubbles, and with no bubbles. After 240 minutes of flotation time, the removal efficiency of microbubble flotation was 68.89% which is significantly higher compared with that obtained with fine bubbles which was 40.9 % and no bubbles (5.14%).

Microbubbles are needed for an efficient separation process due to their high collision rates with oil droplets [49]. Microbubbles have several remarkable features which make them superior to fine bubbles. For example, microbubbles have lower buoyancies, meaning that they slowly rise to the surface of the liquid, offering longer residence periods in the liquid. Microbubbles can either have negative or positive zeta potentials, which is a key factor in preventing bubble agglomeration or coalescence, maintaining a relatively monodisperse size distributions of microbubbles. In addition, the smaller the bubble, the greater the specific interfacial area, which promotes the effective physical adsorption of impurities dissolved in the solution, on the surface of the bubble. Moreover, the smaller the bubble is, the greater its inner pressure. Consequently, the driving force of mass transfer from gas phase to surrounding liquid increases with the decreased bubble size [34, 50, 51]. While fine bubbles have lower surface to volume ratios, lower residence time and large buoyant force. These properties of fine bubbles make them ineffective in the separation of colloidal substances from aqueous solutions [39, 52].
3.2. Effect of initial oil concentration

The effect of initial oil concentration on the oil removal efficiency was studied by preparing an emulsion with two oil concentrations (200, and 300) ppm while maintaining the other parameters constant (pH=7.32, air flow rate 0.5 L/min, air pressure=0.2 bar and liquid height= 55 cm).

The results demonstrate in Figure 4 show the impact of oil concentrations on oil removal efficiency. It can be observed that by increasing the oil concentrations, the removal efficiency of oil was improved. The oil removal efficiency after 165 min, were 46.29% and 73.12% for initial oil concentrations 200 and 300 ppm respectively. The enhancement in oil removal may be due to an increase in the opportunity of air bubbles to attach to floating oil drops in the emulsion. Furthermore, the results show that for both initial oil concentrations, the trend of removal efficiency curve starts to level off after specific time of approximately 210 min. That could be attributed to the distribution of
oil drops size inside the emulsion, once the largest drops are removed; the efficiency of the process slows down, this result is in agreement with the results obtained with other researches [53, 54].

3.3. Effect of pH value of emulsion

The effect of pH on the oil removal efficiency was studied by preparing an emulsion with different pH values (3.32, 5.26, 7.22, and 9) while keeping the other operation parameters unchanged (initial oil concentration= 200 ppm, air flow rate 0.5 L/min, air pressure=0.2 bar and liquid height= 55 cm). The results plotted in Figure 5 showing the oil the removal efficiency (%) with different pH values. From Figure 5, it is evident that lowering the pH from alkaline to neutral and acidic medium significantly increases the efficiency of the oil. The lowest removal efficiency of 60.68% was achieved at pH= 9 after flotation time of 240 minutes. The removal efficiency was obviously higher when the pH become 7.22, where the removal efficiency become 69%. The highest removal efficiency of 75.19% was achieved at pH= 3.32, that results comes in line with the results obtained by [55]. Another crucial factor in determining oil removal is the pH. This is due to changes in pH impact zeta potential of microbubbles and oil droplets. Its indicated that oil droplets have a negative zeta potential under a wide range of pH conditions and the negative value increased as pH rises. The negative potential is thought to result from adsorption of hydroxyl ions at the oil–water interface. As the pH becomes lower, the zeta potential of droplets increases and approaches zero. The zeta potential of microbubbles also increases with decreasing pH and can be potential changes from negative to positive at acidic conditions [56–58]. Since the zeta potential of droplets and microbubbles have negative and positive values, respectively, the oil removal efficiency rapidly increases with increasing positive zeta potential of microbubbles.

![Figure 5](image)

**Figure 5.** Effect of pH value on the oil removal efficiency.

3.4. Effect of adding Sodium chloride

The effect of sodium chloride addition on the oil removal efficiency was studied with different concentrations of NaCl (0, 0.05, 1, 3, and 9) gL−1 while keeping the other parameters constant (initial oil concentration= 200 ppm, pH= 7.32, air flow rate 0.5 L/min, air pressure=0.2 bar and liquid height= 55 cm). This effect is shown in Figure 6 by plotting oil removal efficiency (%) against flotation time at different NaCl concentrations. From this figure, it can be noticed that the addition of NaCl leads to a significant increase in the removal efficiency of oil. Approximately 84% of oil was removed at a
concentration of 1 gL⁻¹ NaCl. The explanation for this increase is that the addition of sodium chloride modified the surface charge of the air bubbles and oil droplets. NaCl caused a reduction in the zeta potential, depending on its concentration. The Cl⁻ anion tended to remain longer at the gas-water interface than the Na⁺ cation [59]. In addition, adding NaCl reduced the air bubbles size and raised the bubbles density [60, 61]. Since small bubbles have less buoyancy than large bubbles, they rise slowly to the surface with high chances of colliding with oil droplets. This improves the method of oil removal [62]. However, adding more NaCl (9 gL⁻¹) reduced the efficiency of oil removal due to collapsed double layers (high ion concentration) around the bubble and oil droplets [63].

![Figure 6. Effect of sodium chloride on the oil removal efficiency.](image)

4. Conclusion
In this work, separation of o/w emulsions with air flotation method was investigated. Based on the results, the following can be concluded:

- The O/W emulsion were successfully prepared using crude oil samples of Al-Dura Refinery in the presence of span 85 and tween 80 surfactants.
- O/W emulsion with oil concentrations (200-300 ppm) could be effectively removed by microbubble air flotation method; High percentage of oil removal (84.03%) was achieved after 4 hours of treatment with oil concentrations of 300 ppm.
- Oil removal efficiency appears to be increased in the acidic to neutral pH range and deceased under high pH condition. More than 75% oil could be removed at pH= 3.32.
- The removal efficiency was found to be improved significantly by adding NaCl, and then decreased with further increased in NaCl concentration due to collapsed double layers of ions.
- Microbubbles showed high removal efficiencies compared with fine bubbles and no-bubbles.

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