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## ABSTRACT

Surfactants stabilise oil droplets in water, forming a dispersed oil-water emulsion. Treatment of oily effluents is a serious challenge owing to the high stability and colloidal nature of the oil droplets. In many applications, microbubbles are employed for separation purposes due to their buoyancy and increased surface area to volume ratio. This property has been exploited in the water treatment industry for separation in a process known as dissolved air flotation (DAF). Though practically efficient, the process is energy intensive operating at >5 bars and consequently consuming ~90% of the total energy required in water purification plants. In this study microbubbles were produced by fluidic oscillation via a nomoving part diverter valve to cut down the energy consumption considerably. Microbubbles are applied for the separation of emulsified oil in a process known as microflotation. The mean bubble size generated by fluidic oscillation from the 50  $\mu$ m pore diffuser was  $\sim$ 100  $\mu$ m, otherwise coarse bubbles were produced under steady flow. The effect of surfactant concentration on oil droplet size was investigated. It was found that oil droplet size varied inversely proportional to surfactant concentration. In addition, it was found that the oil removal efficiency also depends on the surfactant concentration. The maximum oil removal efficiency by Microflotation was found to be 91% under lowest surfactant concentration tested (0.3 wt%) whilst at highest surfactant concentration used (10 wt%); lowest recovery efficiency (19.4%) was recorded.

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# 1. Introduction

Oily effluent treatment is a major environmental challenge experienced by the wastewater and the minerals and metallurgy industries. Effluent water from these industries is widespread and contains various pollutants particularly chemicals, ions, powders, organic (Rubio et al., 2002), etc. which sometimes make recovery of valuable materials difficult (Galvin et al., 1994). Some sources of oil effluents are generated at mills, offshore platforms, tailing ponds, processing plants, mines, etc. (Rubio et al., 2002). Occasionally, given the effluent volume and their complex chemical composition, treatment becomes uneconomical even in instances where potentially recoverable valuable products are involved. In addition, discharge of raw effluent rich in organic material into natural water bodies poses a serious problem during clean-up/separation, especially when emulsification of the oil occurs and moreso when the oil droplets are small ( $<10 \,\mu m$ ) and stabilized by surfactants (Beeby and Nicol, 1993; Rubio et al., 2002).

One of the ways to separate oil emulsion is by the application of microbubbles (Edzwald, 2010; Hosny, 1996; Zouboulis and Avranas, 2000). Microbubbles are used in many operations where mass transport between gas and liquid is important. The surface area to volume ratio available in microbubbles improves the mass transfer rate. Furthermore, a substantial amount of experimental evidence demonstrates that separation efficiency and rate of flotation varies inversely with bubble size (Derjaguin and Dukhin, 1993; Hewitt et al., 1994; Ralston and Dukhin, 1999). To this end, many methods of generating bubbles aiming to reduce bubble size have been developed, e.g. dissolved air flotation, ultrasound techniques, etc. In the last decade however, the former, which relies on releasing saturated liquid to nucleate small bubbles, is the most widely used and developed technique for water treatment (Rubio et al., 2002). DAF is a well-established separation process that employs microbubbles (<150  $\mu$ m) to separate low-density particulates from coagulated raw water within the potable water treatment plant. In principle, this is done by recycling and pressurizing (typically up to 6 bars) a fraction (normally 10%) of the clarified water. Air is injected into the recycled flow within a saturator pressure vessel to an equivalent of 130 mg/l for a typical design saturator pressure of 500 kPa (Edzwald, 2010). The

saturated recycled water then enters the flotation tank via a nozzle system located within the baffle arrangement of the DAF cell. The



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sudden pressure drop across the nozzles causes the recycled flow to become supersaturated, thereby nucleating microbubbles as the dissolved air comes back out of solution. Unfortunately, this process is highly energy intensive (Edzwald, 1995, 2010) as more than 90% of the total operational energy used in DAF is spent on pumping and pressurizing recycled clarified water into the saturator. Another process that has been applied in oily wastewater treatment is Induced Air Flotation (IAF). IAF is the application of mechanically formed air bubbles in flotation. Essentially, the process involves the combination of a high-speed mechanical agitator and an air injection system (Rubio et al., 2002). A continuous gas supply is brought in contact with a liquid by forcing the gas through a nozzle bank, where bubbles are generated. The Jameson cell - where air is entrained into a plunging jet - is the most effective flotation technique in this sector with flotation separation results ~98% reported for algal and phosphorus removal. However, the main challenge facing IAF is the inefficiency to generate sub-150 µm bubbles (Rubio et al., 2002). The additional moving part and energy consumption due to the use of an impeller unit makes IAF more energy expensive relative to dispersed air flotation. Similar to DAF, part of the clarified effluent must be pumped back into the system to combine with the air supply increasing the operational cost. This challenge has encouraged researchers to develop a less energy intensive process for water treatment exploiting energy efficient microbubble generation by fluidic oscillation (Zimmerman et al., 2011a).

The fluidic oscillator (Tesař, 2007; Zimmerman et al., 2008; Tesař and Bandalusena, 2011) is a bistable device featuring one inlet, two mid-ports and two exit ports that controls a continuous fluidic flow, switching between two outlets ports at a regular frequency. According to Tesař et al. (2006), the fluidic oscillator works on the Coanda effect. The fluidic diverter valve alternates the flow path at a frequency, which depends on the gas flow rate and the length of the feedback loop (Tesař, 2007; Zimmerman et al., 2009) with typical frequency ranging from 1 to 100 Hz (Tesař et al., 2006; Zimmerman et al., 2009). Currently, more works have begun to explore the feasibility of the fluidic oscillator driven microbubble generator in many fields concerned with mass transfer. Examples include the application of ozone as a sterilisation agent in the purification of water (Lozano-Parada and Zimmerman, 2010), transfer of oxygen to enhance yeast growth and production as well as rapid and efficient dissolution of CO<sub>2</sub> to promote algal growth for biofuel production (Al-Mashhadani et al., 2011; Zimmerman et al., 2011a). The fluidic oscillator influences bubble sizes by facilitating early break-off just after the bubble grows beyond the hemispherical stage, resulting to mono-dispersed microbubbles (Zimmerman et al., 2008). The fast switching of the flow between two outlets disrupts boundary layer formation within the device; hence lead to less friction and energy savings. Unlike other flotation systems, another advantage of the fluidic oscillator is its robustness. It has no moving parts. It should be noted that only an industrial blower is needed to operate the oscillator system at an offset pressures slightly higher than the head of water. Therefore, such a system does not require the capital cost of a saturator system and large pumps which easily cost an order of magnitude more. Microflotation is used to describe the application of fluidic oscillator generated bubbles in flotation.

The aim of this paper is twofold: First, to explore the feasibility of fluidic oscillator generated bubbles to separate emulsified oil as Microflotation and second, to study the effect of surfactant concentrations on oil removal efficiency. This paper is organised as follows. In Section 2, the materials and methods employed to test the efficiency of the microflotation system for oil removal are presented. The experimental results on the effect of surfactant concentration on oil emulsion separation are presented and discussed in Section 3. Finally, based on the study findings conclusions are drawn in Section 4.

# 2. Materials and methods

## 2.1. Oil separation

#### 2.1.1. Material preparation

Wastewater contains surfactants due to anthropogenic activities and when present, these surfactants stabilize oil in the wastewater, forming an emulsion. However, the degree and stability of the emulsion is a function of surfactant concentration. To replicate this, a test sample of raw water (o/w emulsion) composed of oil, water and an emulsion stabilizer was prepared by adding 10 ml of oil into 1 L of distilled water and surfactant at varying concentrations (0.3, 1, 3, 5 and 10 wt%). The surfactant used was Span 20; a non- ionic surfactant (Sigma Aldrich, UK) with a hydrophile–lipophile balance of 8.6 and density 1050 kg/m<sup>3</sup>. The oil used was Vista Oil 100 (Pennine Lubricants, UK) solvent refined base oil with density 880 kg/m<sup>3</sup> at 20 °C. All the components were emulsified at 18,000 rpm in a blender (Model No.: XB9165; 500 W, Argos, UK) for 5 min to form a stable emulsion.

### 2.1.2. Experimental procedure

After formation of emulsions, coagulation with aluminium sulphate (Sigma Aldrich, UK) and flocculation were followed for 5 min and 7 min respectively. The pH value was adjusted to 8 to achieve the highest possible efficiency for aluminium sulphate as reported by Al-Shamrani et al., 2002a, 2002b). Fig. 1 shows the schematic representation of the experimental set-up. The rig consists of an air supply, fluidic oscillator and a flotation column. A microporous diffuser is placed at the bottom of the flotation column for bubble generation. The fluidic oscillator is supplied with filtered compressed air at 0.8 bars. The frequency of oscillation was measured with an accelerometer.

After flocculation the microbubble generating unit was turned on before the prepared raw water was gradually introduced into the flotation column from the top to a level of 15 cm above diffuser. Samples were collected from sampling port located midway the Microflotation column every 10 min and oil concentration was measured using a turbidimeter 2100Q and a spectrophotometer DR 2800 (HACH Lange, UK) to assay absorbance at 682 nm wavelength. The recovery efficiency (R) was determined using the formulae:



**Fig. 1.** Schematic representation of the experimental rig for oil–water separation. Compressed air is fed into the fluidic oscillator, which then feeds the diffuser. The sample port was positioned mid-way on the flotation column.

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