

FREE AND EMULSIFIED OIL REMOVAL BY BUBBLE ACCELERATED FLOTATION (BAF)

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ABSTRACT

Operational data from full-scale field applications and pilot tests using the Bubble Accelerated Flotation (BAF) system for the treatment of wastewater are presented. The compact BAF system based on the air-sparged hydrocyclone technology was designed to remove pollutants from industrial waste and process water.

Crude oil and vegetable oil levels in water were reduced non-chemically by 61-66% and with pH control and flocculants - above The system was effective at low starting concentration of oil (<10 PPM) and effective in the removal of oil at initial concentrations >30%. Similar reductions were recorded for TSS and microbiological agents in a wide variety of industrial process and wastewater. Due to high air/water ratio, small bubble size, and rapid removal of air from the system, VOCs were also effectively removed.

Keywords: *flotation, oil, O/W emulsion, air-sparged hydrocyclone, flocculants, VOC*

INTRODUCTION

Frequently oil-in-water emulsions contain droplets which are very small (<10 microns) and stabilized with powerful emulsifying agents. Demulsification and oil extraction from such systems present particular problems, the solutions to which have to be economically feasible. In general, flotation systems for water treatment are used to remove as many contaminants from the water, as possible. Design features and operating conditions of such equipment are geared toward the effective separation of contaminants from water by attaching, or immobilizing, contaminants on bubbles. These bubble-contaminant aggregates float to the surface of the treated water where they are skimmed off, thereby removing the contaminants with the froth. Differences between the predominant flotation methods generally have been determined by control of bubble size. In early flotation devices coarse bubbles (2 to 5 mm) were introduced into the water to be treated by blowing air through canvas or other porous material. Froth flotation or modifications like Induced Air Flotation (IAF) produced relatively large bubbles (1 mm) and Dissolved Air Flotation (DAF) produced bubbles in the 20-100 μm range. Recent improvements of such techniques were discussed by Jameson [1] and an improved version of IAF were described which showed promise for the removal of fats oil and grease from wastewater. These systems required large low-turbulence tanks to permit the bubbles to traverse the water in order to come into contact with the contaminants. Smaller bubbles require more rise time in the bulk fluid, thereby increasing the tank volume. Coalescing plates, baffles and various mechanical means have been used to control the turbulence inside these tanks, so that bubble-contaminant aggregates are not re-entrained into the bulk fluid as a result of the water flowing through it, but instead, can rise to the surface.

The benefits of such wastewater flotation systems include the ability to remove Total Suspended Solids (TSS), Fats, Oils & Grease (FOG) as well as particulate bound Chemical

Oxygen Demand (COD) from water. To neutralize electrostatic surface charges on particles and emulsions, pH controlling agents, coagulants and poly-electrolytes often are added to prevent electrostatic repulsion of the particles from bubbles and each other. These chemical agents dramatically enhance the performance of flotation systems by creating large insoluble contaminant aggregates that are amenable to flotation. In DAF and IAF systems elaborate accommodations are made to permit adequate contact time and mixing of these chemical agents with the water being treated before it enters the flotation tanks.

Initially, Air-Sparged Hydrocyclones (ASH) were developed for applications in mineral flotation [2]. One of the major advantages of the ASH is that the air flow is not limited to such a great extent and therefore the number density of bubbles far exceeds that of any other flotation system. Air-to-water ratios of 2/1 to 100/1 are routinely achieved with the ASH, while DAF systems can not achieve a ratio exceeding 0.15/1. Because the bubbles are sheared from the inside wall of the porous tube in the ASH system, they are broken into significantly smaller sizes than bubbles generated in traditional sparged or mechanical systems. The ASH bubbles are comparable in size to those encountered in DAFs. In ASH and BAF systems, the bubble-size distribution is mostly dependent on the velocity of the water traveling along the inside wall of the porous sparge tube. This is a crucial operational parameter, permitting control of the bubble-size distribution. The water flow rate and surfactant concentration have a greater impact on the bubble-size distribution than the tube pore size. In a recent study of the ASH [3] the mean radius of the bubbles, exiting the bottom of the sparge tube, was 50-150 μm . The minimum bubble radius recorded was 10 μm , which represented the detection limit of the analytical procedure, however, the bubble-size distribution clearly extended below this limit.

BAF technology evolved from ASH systems to address operational limitations resulting from the traditional stream-splitting approach of hydrocyclones. The BAF system no longer includes an underflow restriction to force the froth and contaminants through the vortex finder into the overflow. In the ASH, the removal of the froth through the overflow, resulted in a contaminated stream with relatively low concentration of solids. Removing the underflow restriction in the BAF improved the operational ease and consistency [4]. The froth together with the cleaned water are introduced into a receiving tank with the purpose of rapidly isolating bubble-particle aggregates from the cleaned water and providing a very low turbulence environment for froth and bubble-particle networks to accumulate. The froth and bubble-particle aggregates accumulate at the surface of the receiving tank and water is drained out of the froth, resulting in a high solids concentration (12-25 %) in the froth. Furthermore, the compact tank permits smaller

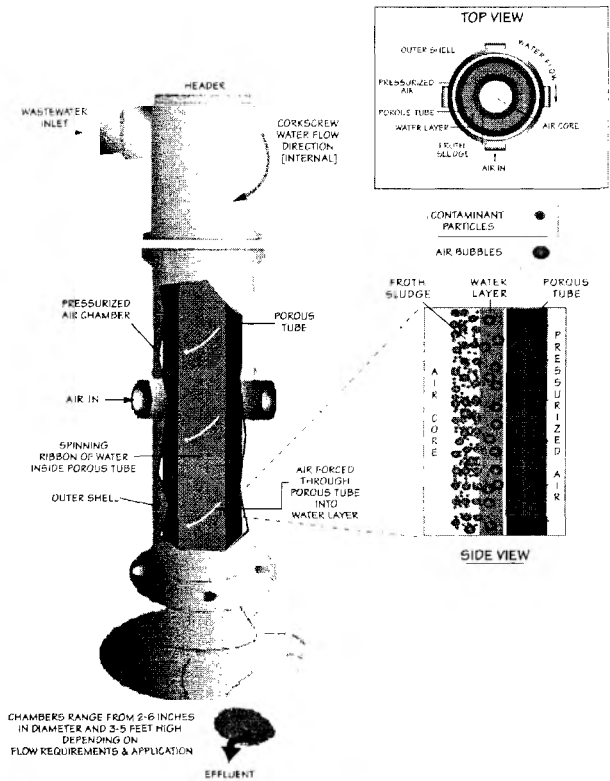


Fig. 1. Cut-away view of a bubble chamber (modified air-sparged hydrocyclone)

particles to float to the surface and provides flow control for the system. The hydraulic flow rate through this receiving tank is more than 10 fold that through any DAF system. This was only possible because coagulation, flocculation and attachment of bubbles to microscopic particles had already occurred inside the ASH bubble chamber and all bubble-particle aggregates were already formed before introduction into the tank. See Fig. 1. Unlike any other flotation system, the receiving tank's purpose was to remove bubble-particle aggregates from the cleaned water stream and not to achieve bubble-particle contact or to float microscopic bubbles. Further details of the system have been described in Owen et al.[4]. See Fig. 2.

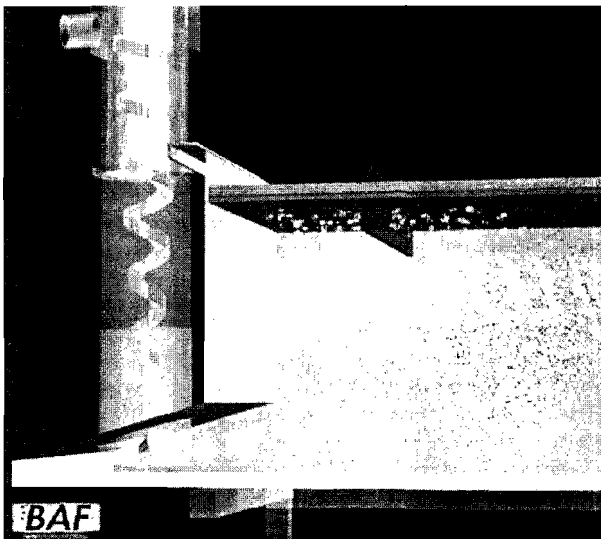


Fig. 2. Cross-section through typical BAF system (bubble chamber and flotation tank) tank

Another fundamental advantage of the BAF system is the high mixing energy in the sparge-tube. For the tests described in the paper, when chemicals were used, low molecular weight surfactants or coagulant such as acids, inorganic salts or organic coagulants are added ahead of the feed pump to permit adequate residence time. However, flocculants and polyelectrolyte polymers are added directly in the sparge tube where the polymers were instantly activated and through the action of the high energy sparge environment brought into intimate contact with the contaminants. This results in the highly efficient use of polymeric flocculants without flocc-tubes or any other means to condition the water with the flotation chemicals. It is important to note that several investigations are conducted entirely without the use of coagulants or flocculants and demonstrated the performance with regard to the mechanical oil-in water emulsion breaking capacity of the BAF mechanism. The efficiency of the BAF system in cleanup of wastewater contaminants will be discussed in this paper. The system has repeatedly demonstrated removal efficiencies of oily contaminants above and beyond those attributable to flocculation or mechanical emulsion breaking alone.

MATERIALS AND METHODS

Standard jar tests were performed to optimize chemical dosages needed to obtain optimum clarification. Visual and turbidity observations were made to evaluate efficiency of the chemical emulsion breaks in the jar tests. Zeta potential measurements (Zeta Meter 3.0, New York) were performed to measure particulate charge before and after the addition of chemicals. Turbidity (NTU, Orbeco-Hellige, Digital Direct-Reading Turbidimeter; FAU, Hach DR 2010) was measured and correlated to the values of total suspended solids (TSS). Chemical oxygen demand (COD) [Hach, DR2010] was determined with standard methods. Spectrophotometric assays for crude oil were performed by creating standard curves with defined quantities of the appropriate crude in Xylene and measuring absorption at 460 and 550 nm. Oil was then separated from samples into the organic phase and absorption values were correlated to the standard curves. Microbial tests (Aerobic Plate Counts, APC) were conducted by standard methods.

Produced Water Tests

In pilot experiments of crude oil/produced water separation, samples were collected at Chevron's platform Gail (Santa Barbara). Sour oil rich in carbon dioxide and hydrogen sulfide gases and

sweet oil were separated from produced water which was recovered together with the crude. The effort was to design an efficient system which would separate oils in the produced water to the point where it could be reinjected into the oilfield without clogging the strata. Since some oil separated during transport from the sweet and sour produced water samples, in both cases, the samples were supplemented with the respective crude oils and homogenized in a blender.

In order to create a finely suspended emulsion, heated water and crude oil were blended with a target of 500 ppm oil in water (weight by volume). Because some oil adhered to container walls and the blender, the final oil in water concentration was lower than the intended 500 ppm. These produced water models were heated to 50°C and 55°C for the sweet and sour stream, respectively. In the sweet water experiments optimum breaks were achieved with SDS, aluminum chlorohydrate (ACH) and high molecular weight anionic polyacrylamide. In the sour water successful break was achieved with highly charged cationic high molecular weight polyacrylamide. It should be noted that these waters exhibited extremely high conductivities and contained minerals in concentrations significantly above that of seawater (42-45 parts/thousand). At these high mineral loads, most charges in electrostatically charged emulsion systems are satisfied by counter-ions and polymeric polyelectrolytes become very inefficient.

The crude oil water was processed in a laboratory bench BAF system. Typically, in these experiments tests were run with the 2.5 cm (ID) hydrocyclone, 2300 dm³/h water flow rate and a 5/1 to 7/1 air/water ratio. At these operational conditions, the centripetal acceleration of the water-ribbon inside the sparge tube was 390 G. The lab-bench system was internally recycled and test runs were timed in order to determine the number of cycles the water was processed.

OLIVE CANNERY TESTS

Olive oil contaminated water from a cannery in Madera, California was used to test the BAF efficiency in separating oil/water emulsions from food processing plants. In this installation, the objective was to remove as much oil as possible before a membrane filtration system (UF system) in a zero-discharge olive cannery. Reprocessing the wastewater generated all the water used in this factory. Several full-scale installations were aimed at polymerically pretreating the water before it accumulated in a central storage tank. A non-chemical system was designed to remove residual oils (introduced by untreated side streams) as well as reduce microbes before the UF-unit. The cannery and flume wastewater exhibited low conductivities (800-2,000 μ S) and moderate pH values (7.8-9.2). Whereas the Vat Room's conductivity frequently exceeded 40,000 μ S and a pH of 12. The combined waters in the storage tank before the non-chemical polisher treatment were in general between 8,000 to 12,000 μ S and maintained at a pH of 10 to retard microbial growth. FOG was typically between 700 and 1200 PPM and TSS was between 800 and 1500 PPM for the cannery. When streams were chemically treated, sulfuric acid was used to adjust pH to 4.9-5.2. A significant exception was the effluent from the olive curing process with pH 12-14 and conductivities as high as 40,000 μ S, (see Table 1). High molecular weight cationic polyacrylamide and high molecular weight anionic polyacrylamide were used to induce bridging-flocculation.

Olive wastewater was treated in full-scale industrial BAF installations. See Fig. 3. Three six inch modified air-sparged hydrocyclones were used in series (cannery, vat room), or in parallel (flume, polisher). Flow rates of 40 - 55 m³/h (cannery), 37 m³/h (vat room), 125 - 193 m³/h (flume) and 450-1,200 GPM (polisher) were used. Receiving tank configurations were described in Owen et al. [4]. An air/water ratio in general was between 4/1 - 6/1 in these applications.

For the flume 1.2-2.4 PPM of a low molecular weight quarternary polyamine (Cytec) was added ahead of the BAF feed pumps as a process aid without pH adjustment (Table 1). Polisher treatments were performed without pH control or any chemical additions. Both these treatments were designed to recirculate the water through the system for several mixed treatment passes. This was accomplished by pumping several times the volume of water through the sparge tubes than was being fed to the system. For example, if the flume polisher was fed at a flow through rate of 300 GPM but the system was operated at 900 GPM, the additional 600 were drawn from the clean water sump on the discharge side of the receiving tanks and mixed with the 300 GPM of untreated water fed to the system. This ensured at least three treatment passes for each volume

of water. This was possible, because the system was drained through a weir, and any water fed into the system would passively drain at that rate from the system.

In general for the cannery, the Zeta Potential measurements indicated that the isoelectric point of the cannery emulsion was at pH 4.5. Sulfuric acid was used to adjust the pH of waste stream to 4.9-5.2, where the emulsion still was somewhat negatively charged (Zeta Potential -5 to -10 mV). 12 PPM of a high molecular weight ($6-8 \times 10^6$ Daltons), 40 % charge density cationic polyacrylamide (C-496 HMW, Cytec) was used to flocculate the emulsion. After passing through the first sparge tube, the water was reprocessed in an integral second system, where an anionic high molecular weight flocculant was added (A 130 HMW, Cytec) to remove any free, excess cationic flocculant. This was a safeguard to prevent any cationic polymers from interfering with the UF and RO membranes. Finally, the water was passed through a third non-chemical sparge to remove any residual polymer or fine flocculated particles that had not floated out in the first two passes. The percent removals and performance data listed in Table 1 were the cumulative difference between the feed entering the first sparge and the discharge from the third sparge.

Other applications (Table 2) were run with similar equipment as described above. Several of these treatment systems were run with 5 cm ID sparge tubes (poultry, meat processor, general laundry) or with larger systems. Polymeric treatment was as noted and formulations similar to those referenced above were used. The non-chemical systems were listed to demonstrate the performance of the mechanical components alone.

RESULTS

Produced Water Tests

The lab-bench system was internally recycled and test runs were timed in order to determine the number of cycles the water was processed. Due to the mixing of the air sparged water with untreated water or water from previous cycles, this system did not provide an optimal evaluation of the number of cycles needed to fully process wastewater. Rather, it provided an indication if flocc and oil emulsions withstood repeated pumping and shear forces. Even though the internally recycled system represented a fractional removal of waste products from the water (instead of a geometric removal in full scale systems with independent stages), it did provide a good reference if multiple stages were necessary for treatment.

Sweet water separation was tested without any flocculants. The system removed 66 % of oil from a sample with 169 PPM of crude oil without addition of chemicals in two recirculated passes. The sweet water conductivity was 31 mS/cm^2 and the zeta potential (ZP) of the emulsion particulates was -10 mV. Passing the water through a BAF system without any chemicals increased the turbidity from 123 NTU to 146 NTU, which suggested some emulsification of the

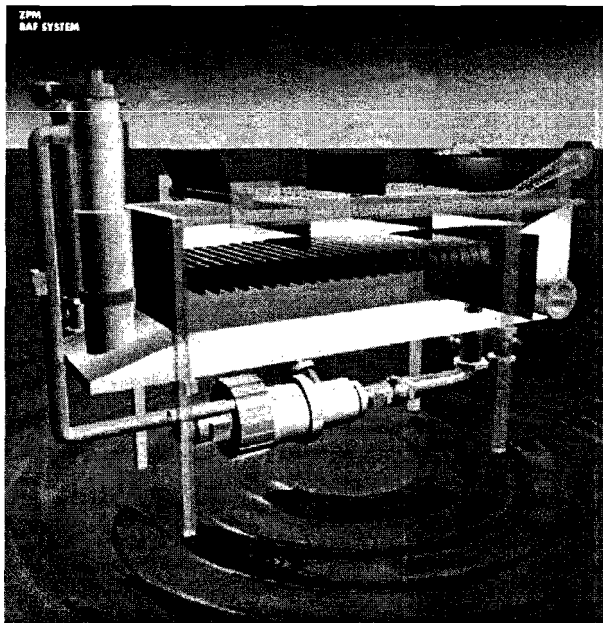


Fig. 3. Schematic presentation of the BAF system applied for olive oil wastewater treatment

oil. After the addition of 40 PPM of SDS and two further passes through the BAF system, 92 % of sweet oil was removed from the produced water, even after emulsification.

For sour produced water, optimal "breaks" were achieved with 40 PPM SDS, 25 PPM aluminum chlorohydrate (ACH) and 30 PPM of a high molecular weight anionic flocculant. A sturdy floc was formed which was easily floated. With this water, some separation (35 %) was achieved even without any chemicals added, however the non-chemical froth was high in water content. Conductivity was measured at 50 mS/cm², and the zeta potential at -5 mV. However, after the addition of cationic high charge flocculant, sturdy flocs were formed, with the production of a skimable phase on top of the tank. After five passes through the BAF system, 92 % of the oil was removed. It is interesting to note that the cationic flocculant was successfully used despite the increase of emulsion charge (-4 mV before addition and +9 mV after addition of flocculant). Sour water could only be treated with highly charged cationic flocculant. In the sour water a slow increase of pH values with increasing cycles of BAF treatment was attributed to the air stripping of CO₂ from the water. A notable decrease in the H₂S odor was noticed.

Olive Oil Extraction Plant Waste Water Treatment

In the flume treatment we managed to nonchemically remove up to 89 % of emulsified and free olive oil from a stream which otherwise accumulated 2,100 PPM of oil. With the use of a low molecular weight coagulant (quaternary polyamine) up to 99.3 % of olive oil was removed from the system. The flume was a closed loop, meaning that the same body of water was retreated approximately every eight minutes. With each treatment pass a smaller fraction of contaminants was removed from the stream (32 % FOG), than the overall load reduction shown above. However, this FOG removal exceeded or balanced the introduction of FOG, rinsed off the olives, travelling through the flume in every treatment cycle. This meant that the load could not be lowered below the level of FOG associated with the olives in the stream (285-380 PPM, FOG).

This reduction in the contaminant load permitted the factory to discontinue the use of 70 GPM makeup water, which was used to dilute the stream in an effort to control the microbial load. Without the ZPM system Aerobic Plate Counts (APC) exceeded 540,000. Even without the 70 GPM makeup water, APC after the treatment averaged 80,000.

The cannery treatment results are described in Table 1. The floc formed exceeded 22-24 % solids. The Vat room treatment is also described in Table 1. It should be noted that the high pH hydrolyzes the cationic polymer in approximately 10 sec after mixing in the stream. Due to the brief residence time (< 1 sec) in the sparge tube, where the floc formation is completed, the polymers still functioned appropriately at a sub-optimal pH. Ferrous gluconate was used as an ingredient in the olive curing process increasing the abundance of iron in the stream. The treatment removed >80 % of iron from the stream. Due to the relatively low FOG load in this stream, proteinaceous matter formed excess light airy foam of low solids content. To control this foam, the Vat room water was partially mixed with cannery water, which provided sufficient oils which acted as defoaming agents.

A similar wet foam was formed in the polisher unit. It was controlled by spraying waste olive oil collected at other processes. The use of olive oil as a foam depressant eliminated the need to use silicone based defoamers. These silicone agents are incompatible with the UF membranes.

Other Oil and Grease Containing Applications

Table 2 lists the performance for other oil-containing applications that have been treated with the BAF system. These numbers represent averages over several weeks of operational time. For the laundry applications, the numbers represent averages of several samples over a period of two days. The system is effective in the removal of FOG from highly contaminated poultry chiller streams, where the contact with food for human consumption precludes the use of polyacrylamides. The system has been used at a meat and frozen food processor with a complex stream, that contains several different caustic cleaners and emulsification agents. The laundry examples illustrate the removal of oil found in automotive mechanics uniforms and shop-rags. Note the non-chemical removal of a large portion of the contaminants (as measured by COD reductions). The laundry detergents effectively emulsify oils but they also assist in the froth flotation process and act as surfactants which permit the formation of smaller bubbles in the

sparge tube. The dominant proportion of laundry detergents are also removed with the froth as evidenced by the relatively low CODs in the recycled water of the linen-laundry. After several reuses, the concentration of dissolved organics did not increase perceptibly.

Table 1. Operational Performance for both Chemical and Non-Chemical BAF Flotation Systems at an Olive Processing Plant

Olive Cannery System Components	Flow GPM	Parameter	% Reduction	Final Discharge Value (ppm)
Cannery	180-240	TSS	97.6	20
		COD	62	1,577
	150	FOG	98.3	15
		APC	76	223,000
Vat Room	150	TSS	76	50
		COD	28	6400
	550-850	FOG	72	20 (with Lye, pH 12)
		APC	75	2,360,000 (no pH adjust)
Flume	550-850	TSS	95	59
		COD	60	2,360
	450-1,200	FOG	85	285 (70 gpm savings)
		APC	84	80,000 (1.4 ppm Poly)
Polisher	450-1,200	TSS	10	29
		COD	10	5,202
	1,200	FOG	5	14
		APC	35	21,000,000

Table 2. Long Term Operational Performance of Completed Installations or On-Site Pilot Projects. Both Chemical and Non-Chemical Applications are Included

Application	Flow GPM	Parameter	% Reduction	Final Discharge Value (ppm)
Poultry Byproducts (Non-chem)	20	TSS	70	150
		COD	27	4800
		FOG	94	83
Rendering (w/ polymer)	100	TSS	90	275
		BOD	55	2400
		FOG	91	54
Meat & Frozen Food Processor (w/ polymer)	70	TSS	98	89
		COD	81	450
		FOG	99.6	13
General Laundry	260	TSS	97	20
		COD	72	930
General Laundry (Non-chem)	70	TSS	58	554
		COD	42	3530
Linen Laundry (w/ polymer)	318	TSS	93	50
		COD	83	690 (recycle water)

DISCUSSION

The fact that the BAF system can be used to demulsify wastewater up to 70 % in some cases without addition of flocculation/demulsification chemicals requires additional explanation. The stability of oil-in-water emulsion is ultimately determined by electrostatic and steric repulsive forces introduced by the addition of emulsifying agents exceeding the attractive hydrophobic forces between dispersed hydrocarbon oils. It is unlikely that the presence of gas modifies

electrostatic or steric repulsive forces to such an extent that this would result in destabilization of emulsions. Therefore, it is quite probable that addition of gas to the system results in an increase in the attractive hydrophobic forces.

A significant number of experimental results accumulated during the last five years seem to support the idea that dissolved gas indeed increases the extent and range of the attractive hydrophobic forces between hydrophobic particles, molecules or surfaces. In recent years methods have been developed to directly measure forces between colloidal particles and surfaces. Atomic force microscopy has been used to measure forces between a particle and a surface and between two particles [5,6]. These methods have been used by at least five research groups to show from direct surface force measurements that the addition of dissolved gas to the system results in long-range attractive hydrophobic forces [7-13]. The range of such forces (up to 100 nm and even more cannot be explained by van der Waals forces or short range hydrophobic forces. The presence of submicroscopic bubbles near the surface and cavitation of these defects as the surfaces approach each other is a reasonable explanation to account for the observed long-range hydrophobic forces.

In another related research area it was shown that dissolved gas in water might be somewhat aggregated in the form of small submicroscopic bubbles, which researchers named babbstones [13,14]. Other researchers believe that such gas aggregates do not have a well-defined interface and called them blobs. Laser cavitation experiments near hydrophobic surfaces indicated that the presence of dissolved gas significantly enhanced the nucleation rate of microscopic bubbles [15]. This suggested that some small non-visible gas nuclei, such as babbstones or blobs indeed could be present in water and accommodated at hydrophobic interfaces. Miller and coworkers [16] recently used in-situ surface spectroscopic techniques and confirmed the accommodation of dissolved gas at a hydrophobic surface. Such accommodation was not detected at the same surface in a hydrophilic state.

Macroscopic experiments also showed the effects of dissolved gas on hydrophobic forces and stability of emulsions, emulsion polymerization, surfactant aggregation, coal filtration and sedimentation [17-19]. In all experiments, the presence of dissolved gas modified the system behavior and generally decreased the colloidal stability of hydrophobic systems, which would indicate an increase in the range and extent of hydrophobic forces, as suggested by the result of direct force measurements.

Other macroscopic experiments might help explain the high efficiency of the BAF system. Air or gas is introduced into the ASH space of the BAF system through the rough hydrophobic porous surface. We showed that this is a very efficient way of nucleating small bubbles required to collect small particles or emulsion droplets. Nucleation studies indeed showed that in order to nucleate small bubbles efficiently, one needs not only a hydrophobic, but also a rough surface [21]. Jones and coworkers developed a model showing that gas nucleation near rough porous hydrophobic surfaces or particles can occur in a system with virtually no supersaturation or with very low supersaturation [22,23]. High shear and centrifugal forces inside the ASH chamber of the BAF system also help in detachment of such bubbles from the porous tube surface. Jones and coworkers indeed showed that bubble detachment could be the time limiting factor in the nucleation rate at hydrophobic rough surfaces [22]. Of course once bubble/particle attachment is achieved the bubble/particle aggregates must be separated from the water. In this regard the flotation tank of the BAF system is specially designed and baffled to prevent turbulence, which could reemulsify the system.

Ultra-flocculation and micro-flotation under vigorous hydrodynamic conditions, as well as the introduction of microbubbles into emulsions are discussed in the literature [23]. Some of the high energy environments (300-600 G) discussed in this paper are also frequently encountered in the BAF system. These results provide an additional framework for the thermodynamic discussion of these phenomena. Traditionally, flocculation, flotation and aggregation are investigated in low Reynolds number flow environments. However, the data presented above should expand the range of energetic environments in which flocculation and flotation are discussed. Definition of these processes will aid in the design and engineering of future applications.

SUMMARY AND CONCLUSIONS

As discussed in the introduction, the BAF system has advantages over the existing DAF, IAF and classical ASH flotation systems for the treatment of oily wastewater. The compact design, low consumption of flotation and other chemicals, efficient mixing of chemicals with short coagulation residence times, large air to water ratios possible, small bubble size and rapid removal of sparged air from the system are attractive features of this new flotation technology. The BAF system has been successful in many applications that represent challenging environments such as high conductivity, pH complex emulsification chemistries, where traditional flotation techniques were ineffective. These applications were selected to demonstrate removal of petrochemical oils as well as vegetable and animal products which are naturally emulsified or solubilized. For the purpose of environmental remediation, these BAF systems can be scaled to much larger flows which allows for the treatment of large wastewater streams for the removal of TSS, COD, FOG and VOC.

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