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Ballasted flotation with glass microspheres for removal of natural organic matter

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Abstract

Low density microspheres were used to float flocs formed from the coagulation of natural organic matter (NOM) using ferric sulphate coagulant. Microspheres were visually observed to be incorporated into the floc structure during the coagulation phase. In comparison with conventional flotation with air bubbles, the residual turbidity after flotation using the microspheres was very favourable and did not impact on overall NOM removal. Spheres of the lowest density and largest particle size gave the most rapid floc clearance, but the residual turbidity after 10 minutes flotation was similar for all of the spheres investigated. The results of this work have shown that floating microspheres offer an effective, energy efficient alternative to conventional dissolved air flotation for removal of flocs containing high concentrations of NOM.

Keywords: ballasted flotation, Flocs, low energy, natural organic matter, flotation, dissolved air flotation.

Introduction

Enhanced clarification through the use of ballasting agents is common place in the water industry. In recent times the embodiment of this concept has focussed on enhancing sedimentation through incorporation of sand [1], sludge [2] or magnetic particles [3] resulting in hydraulic loading rates (surface overflow rate – SOR) up to 40-60 times higher than traditionally experienced with sedimentation (1-2 m^3 m^{-2} h^{-1}). However, the earliest ballasting agent to be routinely used was air bubbles in the dissolved air flotation (DAF) process [4]. The process operates by forming micro bubbles of air in the size range 40-100 μm [5] through depressurisation of air saturated water passing through a nozzle. The generated bubble cloud then combines with the pre formed flocs, reducing their effective density causing the combined floc-bubble aggregate to rise to the surface. The requirement to lower the density of the floc to below that of water means that the process is primarily utilised for removal of naturally low density pollutants such as algae and natural organic matter (NOM).

A major advantage of all ballasting systems is their inherent flexibility as the clarification rate is linked to the dose of ballasting agent applied. In the case of the DAF process the number of bubbles released is controlled by the combination of the super saturation pressure and the flow rate through the saturation tank. The latter is more commonly used to control the process and is known as the recycle ratio as it uses post clarified water which is then returned to the DAF process. Corresponding surface overflow rates range between 5-40 m^3 m^{-2} h^{-1} [5, 6] although operational practice with natural organic matter is normally closer to 4-8 m^3 m^{-2} h^{-1} due to concerns over floc breakage [7]. As with all ballasting systems, the improved performance and flexibility is balanced against a need for additional infrastructure and increased operating costs associated with managing the ballasting agent. In the case of

DAF, the key issue is related to the generation of the micro bubbles which requires pumping recycle flow under pressures of between 4-6 bar. Associated energy estimates range between 0.01-0.03 kWh m^{-3} [8] which accounts for between 50-90% of the total energy demand associated with the process [9, 10].

Conceptually, the same benefits can be realised using low density microspheres instead of air bubbles in a technology more akin to the use of sand in ballasted sedimentation [1]. In such cases the microspheres can be used to further enhance the clarification rates when used in combination with air bubbles or reduce the energy of operation when used as the sole ballasting agent. The latter idea has been detailed in two patents: WO/2006/008474 [11] and US Patent 6890431 [12], however published work verifying the concept is limited.

The current paper investigates this idea by assessing the potential to use low density glass microspheres as a flotation ballasting agent for the clarification of NOM flocs. A previous study has demonstrated the potential of the idea for algae flocs where removal efficiencies of between 63-97% were observed when using 100 μm glass microspheres with no additional air [13]. The highest removal was seen for *Microcystis* and the lowest for *Chlorella* indicating that organic matter components were likely to be important as the algae are fairly similar in size. As NOM exists predominantly in dissolved or colloidal states, organic matter interactions are expected to be even more important. Consequently, the paper addresses the current knowledge gap by assessing the potential for use of microspheres in the treatment of a NOM-laden water, a water that is typically treated using DAF technology. In addition to assessing the overall potential of using the micropsheres for NOM removal, the investigation considers the impact of dosing regime and microsphere incorporation into the floc to resolve practical issues associated with developing the technology.

Methods

A series of bench scale tests were carried out to determine the feasibility of using microspheres for treatment of NOM laden waters. This consisted of: 1) jar testing to determine removal of the NOM and 2) microsphere and floc characterisation to determine the physical characteristics of the flocs formed and the microspheres dosed during coagulation.

Jar testing

Jar tests were carried out using a model DBT6 DAF batch jar tester (EC Engineering, Canada). The DAF jar tester operates in a similar way to a standard jar tester during the floc formation stage. One-Litre (1-L) samples of raw water were placed in 1-L square beakers after the addition of Fe coagulant (ferric sulphate - Ferripol XL, EA West). Water was rapid-mixed for 1.5 minutes at 200 rpm followed by a slow stir period at 30 rpm for 15 minutes. Unlike on a standard jar tester, where sedimentation is used to separate the formed floc from the water, the DAF jar tester adds air saturated water pressurised at 450 kPa into the jar. At the lower pressure in the jar, air bubbles precipitate and attach to the flocs in the jar and float to the surface. The amount of air saturated water added into the jar can be varied from 0-20 % of the 1-L sample in the jar (subsequently referred to as the recycle ratio). Water was sampled from the jar at a sampling point a third of the way up the jar at time intervals of 1, 5 and 10 minutes of flotation for conventional DAF jar tests (no microspheres added). For systems where microspheres were used, no air was added. Tests were also carried out on the jar tester without the addition of microspheres and air for a traditional sedimentation test, as the flocs formed were more dense than water.

106 ***Water source***

107 The water tested on was sampled from a water treatment works (WTWs) in the north
108 of the UK that was considered to be characteristic of a NOM-laden water being of a
109 brown colour, as a result of the high humic content of the water, and low turbidity.
110 The water correspondingly had a dissolved organic carbon (DOC) content of 10.6 mg
111 L^{-1}; ultraviolet absorbance at 254 nm (UV_{254}) of 0.45 cm^{-1}; turbidity of 1-4 NTU and
112 alkalinity of <20 mg L^{-1} $CaCO_3$. Water was coagulated using ferric sulphate (Ferripol
113 XL, EA West) at pH 4.5 (a pre-determined optimum for this water) [5, 14].

114 ***Microsphere dosing***

115 Initial experiments used SID 350Z microspheres (commercially known as
116 Eccospheres® from Trelleborg, Emerson and Cuming Inc, USA). The hollow spheres
117 are composed of a thin shell made from sodium borosilicate glass. This type of glass
118 has a lower density and higher chemical and thermal stability when compared with
119 silicate glass. Manufacturer information reported the densities of the microspheres as
120 0.35 g cm^{-3} with a median particle size of 50 μm. The microspheres were used in
121 coagulation tests as provided by the manufacturer. Later tests used the following
122 microspheres (density and median diameter in brackets): SI 100 (0.10 g cm^{-3}, 70 μm);
123 SI 160 (0.16 g cm^{-3}, 62 μm); SI 200 (0.20 g cm^{-3}, 55 μm).
124 The different experimental dosing parameters investigated were: coagulant
125 concentration (0-20 mg L^{-1} Fe), microsphere concentration (0-450 mg/L SID 350),
126 microsphere dosing sequence and different microsphere variants (density and particle
127 size).

Sampling and analysis

For each jar test, samples were analysed for turbidity using a Hach 2100 turbidimeter after 1, 5 and 10 minutes of flotation. During the testing of increasing coagulant dose, samples were also analysed for residual DOC and UV_{254} using a Shimadzu 5000A TOC analyser and a Jenway UV/Vis spectrophotometer respectively.

Physical characterisation of microspheres and floc

The particle size distribution (PSD) of the microspheres used in this work was measured using a laser diffraction instrument: Malvern Mastersizer 2000 (Malvern Instruments). Microspheres were added into 1-L of de-ionised (DI) water in a 1-L square beaker at a concentration of 300 mg L^{-1}. The microspheres were mixed on a jar tester at 200 rpm and pumped through the optical unit of the Mastersizer and back into the jar. An average of three measurements was used to provide the final PSD. The clearance rate of the different microspheres was measured by adding 300 mg L^{-1} of the microspheres into 1-L of DI water in 1-L square beakers and turbidity was measured from the sampling port in the jar after 10 minutes flotation.

The physical characteristics of the flocs formed with and without microsphere addition were measured using the Mastersizer instrument. Floc formation tests were carried out on a jar tester. After the slow stir at 30 rpm, the effect of increased shear rate was investigated by increasing the rpm on the jar tester to 200 rpm for a further 15 minutes. The shear rate was then reduced back to 30 rpm for 15 minutes to determine the floc re-growth potential. Dynamic floc size was measured during growth, breakage and re-growth of the flocs using the Mastersizer. The suspension was monitored by drawing water through the optical unit of the Mastersizer and back into the jar by a peristaltic pump on the return tube using 5 mm internal diameter

peristaltic pump tubing at a flow rate of 1.5 L hr^{-1}. Size measurements were taken every minute for the duration of the jar test and logged onto a PC. Flocs were also characterised by investigation under a light microscope (Olympus) to determine the effectiveness of microsphere incorporation into the floc.

Results

Microsphere characterisation

The median diameter of the different microspheres (manufacturers rated size in brackets) based on a volume based measurement was 39, 71, 71 and 68 μm for the SAD 350Z (50 μm), SI 100 (70 μm), SI 160 (62 μm) and SI 200 (55 μm) spheres respectively. For most of the microspheres, the PSD was closely distributed around the median values given. The exception was the SI 100 spheres which had a much wider particle size distribution with values ranging between 26-158 μm. Addition of the microspheres into a standard jar testing beaker filled with deionised water revealed that, as expected, the resultant residual turbidity was related to the density of the microspheres. The initial turbidity due to inclusion of the microspheres was 95 NTU which decreased to 0.4 NTU for the SI 100 microspheres (density = 0.1 g cm^{-3}). Residual turbidity for the other microspheres were 0.5 NTU for the SI160 (density = 0.16 g cm^{-3}) and the SI 2000 (density = 0.3 g cm^{-3}). The highest density microspheres of 0.35 g cm^{-3} (SID 350Z) left a residual turbidity of 0.7 NTU.

Coagulation performance

Comparison of effectiveness of coagulation with and without the presence of SID 350Z microspheres revealed no significant difference between the two indicating that the microspheres did not inhibit coagulation or exert an additional demand for coagulant (Figure 1). In both cases, the removal of UV_{254} and DOC followed the same

trend with an increase in removal with coagulant dose up until a dose of 8 mg L^{-1} Fe. At coagulant concentrations above this, removals did not improve. Residual DOC was 2 mg L^{-1}, an 83% reduction. UV_{254} was reduced to 0.024 cm^{-1}, a decrease of 95%. These removals were consistent with previous observations when using this water [5, 14]. Subsequent coagulation trials were therefore carried out using a ferric sulphate concentration of 8 mg L^{-1} as Fe.

Alteration of the dose of SID 350Z microspheres decreased the resultant turbidity up to dose limit of 150 mg L^{-1} beyond which increasing the dose further had no impact on the residual turbidity (Figure 2). To illustrate, residual turbidity increased from 2.3 NTU at a microsphere dose of 0 mg L^{-1} (equivalent to the comparative residual turbidity observed for a sedimentation system) to 5.7 NTU with a dose of 50 mg L^{-1} after 15 minutes of clarification. The residual turbidity then decreased to 1.6 NTU at a dose of 150 mg L^{-1} before decreasing slightly to 1.3 NTU at a dose of 450 mg L^{-1}. Visual observation of the trial verified that inclusion of the microspheres resulted in flotation compared with sedimentation without microspheres (Figure 3) as seen by the generation of a float layer that remained stable post clarification.

Impact of operational decisions

A key feature with all ballasting technologies is the sequence of additions with regards to the coagulant and the ballasting agent. A series of trials was conducted to establish the impact of dosing sequence to reflect the different operational practices reported for systems that used other ballasting agents [15, 16]. Optimum removal under each dosing strategy revealed that simultaneous dosing of coagulant and microsphere provided the most effective method of microsphere incorporation and resultant residual turbidity (Figure 3). For instance, dosing the microspheres 1 minute

after the coagulant resulted in a 1 NTU increase in residual turbidity. This was attributed to reduced microsphere incorporation into the floc because the aggregates had already started to form before the microspheres had been added. In contrast, ballasting with sand is conducted post floc formation where the mass of the sand relative to the floc is thought to enable the sand to enter into the floc structure [15]. In such cases a chemical addition occurs at two points: initially to drive floc formation and post sand inclusion to aid binding. In the current case this idea was tested through multi coagulant doses where either the total dose was kept constant (4+4) or the full dose used at each injection point (8+8) (Figure 3). In both cases the residual turbidity improved to 1.9 and 2.3 NTU respectively but remained higher than the base case of simultaneous dosing. It is also common to utilise ballasting agents when coagulating in high pH environments which favour sweep flocculation mechanisms. Residual turbidity under such conditions (pH =8.5) resulted in a deterioration of performance leading to a residual turbidity of 6.7 NTU. Comparison across the different options suggests that the microspheres should be injected prior to, or simultaneously with, the coagulant to ensure maximum incorporation into the floc. This is consistent with the overall understanding of coagulation of NOM laden waters such as the one tested in the current investigation. Previous investigations into coagulation have shown that floc growth is rapid and that floc size reaches a steady state value typically within 5 minutes after coagulant injection with substantial growth being evident within the first two minutes of coagulation [17]. Consequently, post addition of microspheres after coagulation restricts the potential for microsphere incorporation resulting in less even spread of microspheres throughout the aggregates and less total incorporation. Comparison with the mechanism proposed for sand inclusion into flocs indicates that the low density of the microspheres is unlikely to have sufficient momentum to drive

post flocculation incorporation and hence the need to be incorporated as the flocs initially form for maximum inclusion.

The other major operational decision in relation to ballasted flotation relates to the selection of the most effective microsphere. Comparison of the different microspheres tested revealed no significant different between the residual turbidity after 10 minutes of clarification at between 1 and 1.3 NTU (Figure 4). In comparison, operation of the jar tester in standard DAF (equivalent recycle ratio of 4 %) or sedimentation mode resulted in a residual turbidity of 0.8 NTU and 1.8 NTU respectively indicating that the ballasted microsphere systems performed effectively. To reflect that both the size and density of the microspheres were changing, the residual turbidity was compared with the lumped parameter $\Delta\rho d^2$ based around Stokes' law where $\Delta\rho$ is the difference in density between the water and the microsphere and d is the diameter of the microsphere (Figure 5). Comparison of the data revealed that microsphere properties had minimal influence on residual turbidity after 10 minutes of clarification. In contrast, microsphere properties were seen to strongly influence performance over short clarification times. For instance, after 1 minute of clarification the highest $\Delta\rho d^2$ value of 4.5×10^{-6} kg m^{-1} generated the lowest the residual turbidity of 2.5 NTU. Residual turbidity reached a maximum of 15.9 NTU as the $\Delta\rho d^2$ decreased to 3.7×10^{-6} kg m^{-1}. At the lowest value of $\Delta\rho d^2$ at 9.9×10^{-7} kg m^{-1} a residual turbidity of 10.5 NTU was observed. After 5 minutes flotation time, the same trend in residual turbidity was observed for the same $\Delta\rho d^2$ as for 1 minute flotation, but there was less difference between the highest and lowest turbidity.

Floc characterisation

Monitoring of floc growth dynamics with and without microsphere inclusion revealed that the inclusion of microspheres did not influence the initial growth rate but reduced the steady state floc size achieved (Figure 6). To illustrate, flocs reached a maximum median diameter of 750 μm when no microspheres were added compared with 590 μm with microsphere inclusion. In addition, inclusion of microspheres did not result in any difference in terms of the impact of increased shear rate and subsequent re-growth of the floc with both systems decreasing to 160 μm when exposed to a shear rate of 200 rpm ($G = 127.5\ s^{-1}$) and re-growing to a size of 200 μm when the shear rate was reduced to 30 rpm ($G=7.4\ s^{-1}$) for a further 15 minutes (Figure 6).

Visual inspection of the aggregates revealed that the microspheres were successfully incorporated into the floc and where distributed throughout the floc structure (Example provided as an insert in Figure 6). In addition, visual inspection of the jars post clarification suggested that the ballasted system was performing better than the standard DAF version. Closer inspection revealed that the main contribution to the residual turbidity was microspheres that had not been incorporated into the aggregates. This was confirmed by filtering the residual water through a 450 nm filter which collected the unincorporated microspheres. The retained free microsphere appeared to have no organic coating or coagulant associated to them which was interpreted as microsphere that had not been incorporated within the floc during flocculation as opposed to release of beads during floc breakage.

Discussion

The results of this work have demonstrated the potential to use low density microspheres as a ballasting agent for flotation. Comparison with traditional DAF

indicates similar levels of removal for NOM treatment such that use of microspheres can be considered as an alternative to bubbles for operation of a low energy, lower carbon footprint alternative to traditional DAF due to removing the need for a saturation system. Estimates of energy demand associated with saturation range between 0.01-0.04 kWh m^{-3} [8, 9] with equivalent numbers for hydrocyclones suggesting between a 60-80 % energy saving if ballasted flotation was adopted [18]. Placed into the context of a medium size water treatment works (50 ML d^{-1}) this equates to a saving of, based on an average value of energy demand, 365,000 kWh $year^{-1}$ which represents a cost reduction of £25,550 $year^{-1}$ (based on electricity costs of 7p per kWh) or 198.6 tCO_2e $year^{-1}$ (0.544 $kgCO_2e$ kWh). Extending this across multiple DAF plants represents a significant saving to an individual water company.

The presented results also offer insight into key design and operating issues. In terms of microsphere selection, the combined term $\Delta\rho d^2$ can be used to grade appropriate microspheres. Little difference was observed for longer clarification periods but clear advantage when using high $\Delta\rho d^2$ (>$4x10^{-6}$ kg m^{-1}) was seen when operating on short clarification periods. The explanation relates to the microsphere numbers required to achieve suitable rise velocities. Typical floc rise velocities of bubble-floc aggregates have been measured between 3 and 15 m h^{-1} [19, 20]. Based on flocs having an average diameter of 500 μm, with a conservative density of the flocculated material of 1050 kg m^{-3} and assuming that the flocs are spherical in nature and obey Stokes' law, for a target rise velocity of 6 m h^{-1} only 12 of the bigger, lower density SI 100 microspheres would need to be contained in the floc. For the smaller, lower density spheres approximately 150 of the SI 350Z microspheres would need to be contained in each floc aggregate to achieve the same rise velocity. This explains why the increased turbidity was much greater after 1 and 5 minutes for the smaller, higher

density spheres: not enough microspheres were incorporated into the floc to give comparable rise velocities as for the large, low density spheres. After 10 minutes, the floc-microsphere aggregates, and any residual microspheres, had had enough time to clear from the water.

The other major observation concerning application of microspheres is related to dosing sequence where optimum performance was observed when the microspheres were incorporated during the initial floc growth stages. This is in contrast to traditional DAF where the air bubbles combine with the pre formed floc [21] or sand which is combined with partially formed flocs and bound with additional chemicals [1]. In the current case, post floc incorporation is limited as the microspheres have insufficient mass to penetrate the formed flocs due to their momentum [15]. The consequence of which is that dosing arrangements require careful management when using microspheres. The ideal arrangement would be injection of microspheres into the incoming feed line to ensure effective mixing with the water prior to the initiation of flocculation [13].

Conclusions

The overall conclusion from the work is that the application of glass microspheres as a ballasting agent is an effective alternative to DAF. The use of microspheres provides a route to significantly reduce the energy and carbon footprint of flotation whilst retaining the treatment efficiency. Selection of the most appropriate bead appears only crucial when operating at very short residence times such that the main area for optimisation and control relates to microsphere incorporation into the floc. This is best achieved through pre-dosing the microsphere prior to chemical addition which then ensures both good incorporation and even distribution. Whilst the current work

demonstrated the potential for using microspheres, further work is required to resolve the operational issues that will ensure the process becomes and effective technology in the future.

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