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6 **Ballasted flotation with glass microspheres for removal of natural**  
7 **organic matter**

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17

## 18 **Abstract**

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

29

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

32

## 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 ( $1\text{-}2\text{ m}^3\text{ m}^{-2}\text{ h}^{-1}$ ). 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  $40\text{-}100\text{ }\mu\text{m}$  [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  $5\text{-}40\text{ m}^3\text{ m}^{-2}\text{ h}^{-1}$  [5, 6] although operational practice with natural organic matter is normally closer to  $4\text{-}8\text{ m}^3\text{ m}^{-2}\text{ h}^{-1}$  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

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

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

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

## 83    **Methods**

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

### 89    ***Jar testing***

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

## ***Water source***

The water tested on was sampled from a water treatment works (WTWs) in the north of the UK that was considered to be characteristic of a NOM-laden water being of a brown colour, as a result of the high humic content of the water, and low turbidity. The water correspondingly had a dissolved organic carbon (DOC) content of 10.6 mg L<sup>-1</sup>; ultraviolet absorbance at 254 nm (UV<sub>254</sub>) of 0.45 cm<sup>-1</sup>; turbidity of 1-4 NTU and alkalinity of <20 mg L<sup>-1</sup> CaCO<sub>3</sub>. Water was coagulated using ferric sulphate (Ferripol XL, EA West) at pH 4.5 (a pre-determined optimum for this water) [5, 14].

## ***Microsphere dosing***

Initial experiments used SID 350Z microspheres (commercially known as Eccospheres<sup>®</sup> from Trelleborg, Emerson and Cuming Inc, USA). The hollow spheres are composed of a thin shell made from sodium borosilicate glass. This type of glass has a lower density and higher chemical and thermal stability when compared with silicate glass. Manufacturer information reported the densities of the microspheres as 0.35 g cm<sup>-3</sup> with a median particle size of 50 µm. The microspheres were used in coagulation tests as provided by the manufacturer. Later tests used the following microspheres (density and median diameter in brackets): SI 100 (0.10 g cm<sup>-3</sup>, 70 µm); SI 160 (0.16 g cm<sup>-3</sup>, 62 µm); SI 200 (0.20 g cm<sup>-3</sup>, 55 µm).

The different experimental dosing parameters investigated were: coagulant concentration (0-20 mg L<sup>-1</sup> Fe), microsphere concentration (0-450 mg/L SID 350), microsphere dosing sequence and different microsphere variants (density and particle 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  $300 \text{ mg L}^{-1}$ . 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  $300 \text{ mg L}^{-1}$  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<sup>-1</sup>. 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<sup>-3</sup>). Residual turbidity for the other microspheres were 0.5 NTU for the SI160 (density = 0.16 g cm<sup>-3</sup>) and the SI 2000 (density = 0.3 g cm<sup>-3</sup>). The highest density microspheres of 0.35 g cm<sup>-3</sup> (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<sub>254</sub> and DOC followed the same

trend with an increase in removal with coagulant dose up until a dose of 8 mg L<sup>-1</sup> Fe. At coagulant concentrations above this, removals did not improve. Residual DOC was 2 mg L<sup>-1</sup>, an 83% reduction. UV<sub>254</sub> was reduced to 0.024 cm<sup>-1</sup>, 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<sup>-1</sup> as Fe.

Alteration of the dose of SID 350Z microspheres decreased the resultant turbidity up to dose limit of 150 mg L<sup>-1</sup> 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<sup>-1</sup> (equivalent to the comparative residual turbidity observed for a sedimentation system) to 5.7 NTU with a dose of 50 mg L<sup>-1</sup> after 15 minutes of clarification. The residual turbidity then decreased to 1.6 NTU at a dose of 150 mg L<sup>-1</sup> before decreasing slightly to 1.3 NTU at a dose of 450 mg L<sup>-1</sup>. 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 difference 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  $4.5 \times 10^{-6} \text{ kg m}^{-1}$  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  $3.7 \times 10^{-6} \text{ kg m}^{-1}$ . At the lowest value of  $\Delta\rho d^2$  at  $9.9 \times 10^{-7} \text{ kg m}^{-1}$  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  $\mu\text{m}$  when no microspheres were added compared with 590  $\mu\text{m}$  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  $\mu\text{m}$  when exposed to a shear rate of 200 rpm ( $G = 127.5 \text{ s}^{-1}$ ) and re-growing to a size of 200  $\mu\text{m}$  when the shear rate was reduced to 30 rpm ( $G=7.4 \text{ s}^{-1}$ ) for a further 15 minutes (Figure 6).

Visual inspection of the aggregates revealed that the microspheres were successfully incorporated into the floc and were 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

275 indicates similar levels of removal for NOM treatment such that use of microspheres  
 276 can be considered as an alternative to bubbles for operation of a low energy, lower  
 277 carbon footprint alternative to traditional DAF due to removing the need for a  
 278 saturation system. Estimates of energy demand associated with saturation range  
 279 between 0.01-0.04 kWh m<sup>-3</sup> [8, 9] with equivalent numbers for hydrocyclones  
 280 suggesting between a 60-80 % energy saving if ballasted flotation was adopted [18].  
 281 Placed into the context of a medium size water treatment works (50 ML d<sup>-1</sup>) this  
 282 equates to a saving of, based on an average value of energy demand, 365,000 kWh  
 283 year<sup>-1</sup> which represents a cost reduction of £25,550 year<sup>-1</sup> (based on electricity costs  
 284 of 7p per kWh) or 198.6 tCO<sub>2</sub>e year<sup>-1</sup> (0.544 kgCO<sub>2</sub>e kWh). Extending this across  
 285 multiple DAF plants represents a significant saving to an individual water company.  
 286 The presented results also offer insight into key design and operating issues. In terms  
 287 of microsphere selection, the combined term  $\Delta\rho d^2$  can be used to grade appropriate  
 288 microspheres. Little difference was observed for longer clarification periods but clear  
 289 advantage when using high  $\Delta\rho d^2$  ( $>4 \times 10^{-6}$  kg m<sup>-1</sup>) was seen when operating on short  
 290 clarification periods. The explanation relates to the microsphere numbers required to  
 291 achieve suitable rise velocities. Typical floc rise velocities of bubble-floc aggregates  
 292 have been measured between 3 and 15 m h<sup>-1</sup> [19, 20]. Based on flocs having an  
 293 average diameter of 500 µm, with a conservative density of the flocculated material of  
 294 1050 kg m<sup>-3</sup> and assuming that the flocs are spherical in nature and obey Stokes' law,  
 295 for a target rise velocity of 6 m h<sup>-1</sup> only 12 of the bigger, lower density SI 100  
 296 microspheres would need to be contained in the floc. For the smaller, lower density  
 297 spheres approximately 150 of the SI 350Z microspheres would need to be contained  
 298 in each floc aggregate to achieve the same rise velocity. This explains why the  
 299 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 an effective technology in the future.

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