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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

33 **Introduction**

34 Enhanced clarification through the use of ballasting agents is common place in the
35 water industry. In recent times the embodiment of this concept has focussed on
36 enhancing sedimentation through incorporation of sand [1], sludge [2] or magnetic
37 particles [3] resulting in hydraulic loading rates (surface overflow rate – SOR) up to
38 40-60 times higher than traditionally experienced with sedimentation ($1-2 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$).
39 However, the earliest ballasting agent to be routinely used was air bubbles in the
40 dissolved air flotation (DAF) process [4]. The process operates by forming micro
41 bubbles of air in the size range 40-100 μm [5] through depressurisation of air
42 saturated water passing through a nozzle. The generated bubble cloud then combines
43 with the pre formed flocs, reducing their effective density causing the combined floc-
44 bubble aggregate to rise to the surface. The requirement to lower the density of the
45 floc to below that of water means that the process is primarily utilised for removal of
46 naturally low density pollutants such as algae and natural organic matter (NOM).
47 A major advantage of all ballasting systems is their inherent flexibility as the
48 clarification rate is linked to the dose of ballasting agent applied. In the case of the
49 DAF process the number of bubbles released is controlled by the combination of the
50 super saturation pressure and the flow rate through the saturation tank. The latter is
51 more commonly used to control the process and is known as the recycle ratio as it
52 uses post clarified water which is then returned to the DAF process. Corresponding
53 surface overflow rates range between $5-40 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ [5, 6] although operational
54 practice with natural organic matter is normally closer to $4-8 \text{ m}^3 \text{ m}^{-2} \text{ h}^{-1}$ due to
55 concerns over floc breakage [7]. As with all ballasting systems, the improved
56 performance and flexibility is balanced against a need for additional infrastructure and
57 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⁻³ [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.

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 (UV₂₅₄) of 0.45 cm⁻¹; turbidity of 1-4 NTU and
112 alkalinity of <20 mg L⁻¹ CaCO₃. 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).

128 ***Sampling and analysis***

129 For each jar test, samples were analysed for turbidity using a Hach 2100 turbidimeter
130 after 1, 5 and 10 minutes of flotation. During the testing of increasing coagulant dose,
131 samples were also analysed for residual DOC and UV₂₅₄ using a Shimadzu 5000A
132 TOC analyser and a Jenway UV/Vis spectrophotometer respectively.

133

134 ***Physical characterisation of microspheres and floc***

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

144 The physical characteristics of the flocs formed with and without microsphere
145 addition were measured using the Mastersizer instrument. Floc formation tests were
146 carried out on a jar tester. After the slow stir at 30 rpm, the effect of increased shear
147 rate was investigated by increasing the rpm on the jar tester to 200 rpm for a further
148 15 minutes. The shear rate was then reduced back to 30 rpm for 15 minutes to
149 determine the floc re-growth potential. Dynamic floc size was measured during
150 growth, breakage and re-growth of the flocs using the Mastersizer. The suspension
151 was monitored by drawing water through the optical unit of the Mastersizer and back
152 into the jar by a peristaltic pump on the return tube using 5 mm internal diameter

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

157

158 **Results**

159 *Microsphere characterisation*

160 The median diameter of the different microspheres (manufacturers rated size in
161 brackets) based on a volume based measurement was 39, 71, 71 and 68 µm for the
162 SAD 350Z (50 µm), SI 100 (70 µm), SI 160 (62 µm) and SI 200 (55 µm) spheres
163 respectively. For most of the microspheres, the PSD was closely distributed around
164 the median values given. The exception was the SI 100 spheres which had a much
165 wider particle size distribution with values ranging between 26-158 µm. Addition of
166 the microspheres into a standard jar testing beaker filled with deionised water
167 revealed that, as expected, the resultant residual turbidity was related to the density of
168 the microspheres. The initial turbidity due to inclusion of the microspheres was 95
169 NTU which decreased to 0.4 NTU for the SI 100 microspheres (density = 0.1 g cm⁻³).
170 Residual turbidity for the other microspheres were 0.5 NTU for the SI160 (density =
171 0.16 g cm⁻³) and the SI 2000 (density = 0.3 g cm⁻³). The highest density microspheres
172 of 0.35 g cm⁻³ (SID 350Z) left a residual turbidity of 0.7 NTU.

173 *Coagulation performance*

174 Comparison of effectiveness of coagulation with and without the presence of SID
175 350Z microspheres revealed no significant difference between the two indicating that
176 the microspheres did not inhibit coagulation or exert an additional demand for
177 coagulant (Figure 1). In both cases, the removal of UV₂₅₄ and DOC followed the same

178 trend with an increase in removal with coagulant dose up until a dose of 8 mg L⁻¹ Fe.
179 At coagulant concentrations above this, removals did not improve. Residual DOC was
180 2 mg L⁻¹, an 83% reduction. UV₂₅₄ was reduced to 0.024 cm⁻¹, a decrease of 95%.
181 These removals were consistent with previous observations when using this water [5,
182 14]. Subsequent coagulation trials were therefore carried out using a ferric sulphate
183 concentration of 8 mg L⁻¹ as Fe.

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

194

195 ***Impact of operational decisions***

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

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

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

230

231 The other major operational decision in relation to ballasted flotation relates to the
232 selection of the most effective microsphere. Comparison of the different microspheres
233 tested revealed no significant difference between the residual turbidity after 10 minutes
234 of clarification at between 1 and 1.3 NTU (Figure 4). In comparison, operation of the
235 jar tester in standard DAF (equivalent recycle ratio of 4 %) or sedimentation mode
236 resulted in a residual turbidity of 0.8 NTU and 1.8 NTU respectively indicating that
237 the ballasted microsphere systems performed effectively. To reflect that both the size
238 and density of the microspheres were changing, the residual turbidity was compared
239 with the lumped parameter $\Delta\rho d^2$ based around Stokes' law where $\Delta\rho$ is the difference
240 in density between the water and the microsphere and d is the diameter of the
241 microsphere (Figure 5). Comparison of the data revealed that microsphere properties
242 had minimal influence on residual turbidity after 10 minutes of clarification. In
243 contrast, microsphere properties were seen to strongly influence performance over
244 short clarification times. For instance, after 1 minute of clarification the highest $\Delta\rho d^2$
245 value of $4.5 \times 10^{-6} \text{ kg m}^{-1}$ generated the lowest the residual turbidity of 2.5 NTU.
246 Residual turbidity reached a maximum of 15.9 NTU as the $\Delta\rho d^2$ decreased to 3.7×10^{-6}
247 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
248 was observed. After 5 minutes flotation time, the same trend in residual turbidity was
249 observed for the same $\Delta\rho d^2$ as for 1 minute flotation, but there was less difference
250 between the highest and lowest turbidity.

251 ***Floc characterisation***

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

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

272 **Discussion**

273 The results of this work have demonstrated the potential to use low density
274 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⁻³ [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⁻¹) this
282 equates to a saving of, based on an average value of energy demand, 365,000 kWh
283 year⁻¹ which represents a cost reduction of £25,550 year⁻¹ (based on electricity costs
284 of 7p per kWh) or 198.6 tCO₂e year⁻¹ (0.544 kgCO₂e 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⁻¹) 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⁻¹ [19, 20]. Based on flocs having an
293 average diameter of 500 μ m, with a conservative density of the flocculated material of
294 1050 kg m⁻³ and assuming that the flocs are spherical in nature and obey Stokes' law,
295 for a target rise velocity of 6 m h⁻¹ 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

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

304 The other major observation concerning application of microspheres is related to
305 dosing sequence where optimum performance was observed when the microspheres
306 were incorporated during the initial floc growth stages. This is in contrast to
307 traditional DAF where the air bubbles combine with the pre formed floc [21] or sand
308 which is combined with partially formed flocs and bound with additional chemicals
309 [1]. In the current case, post floc incorporation is limited as the microspheres have
310 insufficient mass to penetrate the formed flocs due to their momentum [15]. The
311 consequence of which is that dosing arrangements require careful management when
312 using microspheres. The ideal arrangement would be injection of microspheres into
313 the incoming feed line to ensure effective mixing with the water prior to the initiation
314 of flocculation [13].

315

316 **Conclusions**

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

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

328

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