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Successful Removal of Algae through the Control of Zeta Potential

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Abstract: Algae can interfere with treatment processes at a water treatment works. Coagulation control is critical to reduce the impact of algae on downstream processes. This paper investigates the coagulation and flotation of four species of algae – *Asterionella formosa*, *Melosira sp.*, *Microcystis aeruginosa*, and *Chlorella vulgaris*. The zeta potential at optimum removal was measured and it was observed that when the zeta potential was reduced to between −8 mV and +2 mV, removal of algae and associated organic material was optimized, irrespective of the coagulant dose or pH. Process control using zeta potential is therefore a viable tool for algae removal.

Keywords: Algae, coagulation, dissolved air flotation (DAF), zeta potential

INTRODUCTION

Many drinking water source reservoirs are subject to algae blooms which tend to occur on a seasonal basis. During such periods, carry over of algal cells and coagulant from the coagulation/clarification process to downstream filters can occur which can result in either filter blockage or penetration. Key to remedying the problem is better coagulation control such that the likelihood of its failure is minimized. Particles or colloids that enter a water treatment works, including algal cells, are negatively charged (1, 2) either as a result

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of dissociation or ionization of surface functional groups (for organic particles), adsorption of ions originating from organic matter, or lattice imperfections in inorganic particles. Consequently, there is electrostatic repulsion between adjacent particles if they come into contact at close separation distances and thus colloidal stability of the system is maintained (3). Successful removal of influent particles relies on disrupting the stability of the system which is the objective of the coagulation process. Destabilization is usually achieved at a water treatment works by the addition of cationic chemicals, including trivalent metal salts (Al^{3+} or Fe^{3+}) or cationic polymers, which interact with the particle surface to induce neutralization affects, although the mechanism by which this occurs is still disputed (4)–(6). However, there is consensus that in destabilizing the system, the electrostatic barrier to contact between two adjacent components is minimized such that attractive van der Waals forces dominate over repulsive electrostatic forces (3).

Surface charge is therefore an important parameter in coagulation experiments. The zeta potential is a measure of the electric potential at the plane of shear of the electrical double layer. The shear plane forms the boundary between the charged particle surface with adsorbed counter-ions and the diffuse region. Zeta potential therefore gives a measurement of the apparent surface charge. Reduction in the magnitude of the negative zeta potential signifies a reduction in the repulsive electrostatic forces and a critical zeta potential can be reached where the attractive van der Waals forces overcome these electrostatic forces and thus particles agglomerate (3).

Dissolved air flotation is considered to be a more efficient treatment process for algae removal when compared to sedimentation, provided the appropriate operating conditions are implemented (7). When investigating coagulation in combination with dissolved air flotation (DAF) for clarification, the significance of particle charge control is even more pronounced. The DAF process utilizes many microscopic, negatively charged bubbles, generated using pressurized, air saturated, recycled water to float flocs produced by the preceding coagulation process. Successful flotation relies on successful particle-bubble attachment which is subject to the same forces previously described for particle-particle interactions, specifically electrostatic repulsion and attractive van der Waals among others. One study claimed that the electrostatic character of the bubbles and particles was the most important parameter for governing the removal efficiency of a batch DAF reactor (8).

The use of zeta potential for monitoring and controlling the coagulation of natural organic matter (NOM) has been well researched and found to be of great benefit (9, 10). However, there have been fewer studies investigating the use of zeta potential for controlling algae treatment. There is evidence to suggest that it may not be as successful when compared to NOM and kaolin as a result of variable morphology which interferes with the mechanisms involved in coagulation (11). However, if zeta potential is demonstrated

to be a useful control parameter for algae removal, many difficulties associated with algae coagulation control that arise as a result of highly variable population loadings and species diversity could be overcome. Hence, the current paper assesses the applicability of zeta potential for controlling the coagulation of the dynamic and diverse algae cell communities.

MATERIALS AND METHOD

Algae Cultivation Procedure

The following algae species were obtained from the Culture Collection for Algae and Protozoa (CCAP), Oban, Scotland:

1) *Microcystis aeruginosa* (1450/3), a cyanobacteria;
2) *Chlorella vulgaris* (211/11B), a green algae; and
3) *Asterionella formosa* (1005/9), a diatom.

Melosira sp. (JA386) obtained from Sciento, Manchester, UK. *M. aeruginosa*, and *C. vulgaris* were grown under the same cultivating conditions, using sterile Jaworski Media, a growth temperature of 20°C, and constant 24 hour lighting using two Sun-glo 30 W fluorescent tubes. The suspensions were grown in 200 ml volumes and shaken at 75 rpm using a Patterson Scientific Bibby Stuart SO1. The diatoms, *A. formosa* and *Melosira sp.*, favored slightly different conditions for optimum growth as follows: sterile Diatom Media; a growth temperature of 15°C; a lighting cycle of 14 hours light/8 hours dark; and, agitation only once daily by hand. An Environmental Test Chamber (Sanyo Versatile Environmental Test Chamber, MLK 350H), programmed to give a brightness of 1000 lx, was used for diatom growth.

Algae Characterization Procedure

Algae systems comprise two major components: cells and extracellular organic matter (EOM). Algae cells were characterized in terms of concentration, size, and surface area. Cell concentration was observed to increase according to a standard growth curve, as shown in Fig. 1 for *C. vulgaris.* Cell counting was completed using either a haemocytometer, for very concentrated samples, or a Sedgewick Rafter cell, for counting smaller populations, as appropriate. Owing to previous observations suggesting that growth cycle can affect system zeta potential, attributed to varying concentration and character of EOM (12, 13), it was ensured that algae were always harvested at the same stage of growth, selected at the onset of the stationary phase. At this stage, population density was at

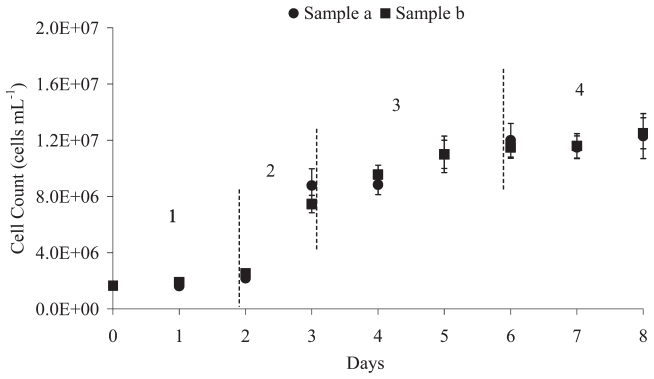


Figure 1. The growth phases of the algae, *C. vulgaris*, where Zone: 1. Lag phase; 2. Unlimited growth; 3. Linear growth; 4. Stationary phase.

its highest and thus dilution to a concentration observed in the natural environment was undertaken prior to flotation experiments (Table 1). Dilution was achieved using deionized water that had been buffered to 0.5 mM using 1.0 M $NaHCO_3$ and made to a final ionic strength of 2.3 mM using 1.0 M NaCl.

The surface area and charge density of *M. aeruginosa*, *C. vulgaris*, and *A. formosa*, and the surface area of *Melosira sp.* are summarized in Table 2 with photographs. Note that the charge density was determined according to an adapted back titration method (14). Algae were specifically chosen to provide variations in terms of morphology and EOM as follows:

1) *M. aeruginosa* and *C. vulgaris* represent similarly shaped cells of simple spherical configuration from different phyla which have very different EOM character;
2) *A. formosa* has a more complex cell structure in comparison to *M. aeruginosa* and *C. vulgaris*;

Table 1. Algae concentration reached in cultivating flasks and concentration used for flotation experiments

Algae species	Stationary phase concentration (Cells ml^{-1})	Experimental concentration (Cells ml^{-1})
Chlorella vulgaris	$1.1 \times 10^7 \pm 1 \times 10^6$	$5.0 \times 10^5 \pm 5 \times 10^4$
Microcystis aeruginosa	$1.3 \times 10^7 \pm 3 \times 10^5$	$6.0 \times 10^5 \pm 1.5 \times 10^4$
Asterionella formosa	$4.5 \times 10^5 \pm 7.6 \times 10^4$	$5.0 \times 10^4 \pm 1.2 \times 10^4$
Melosira sp.	$2.0 \times 10^4 \pm 4.8 \times 10^2$	$2.0 \times 10^3 \pm 2.0 \times 10^2$

Table 2. Summary of algae cell characteristics in terms of cell surface area and charge density previously determined (14) where r = radius, a = width, d = depth and l = length

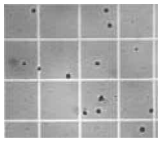
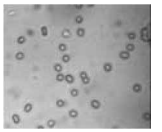
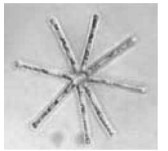
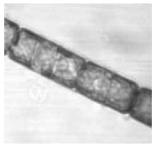
Algae species	Surface area equation	Photos	Average surface area (μm^2 $cell^{-1}$)	Charge density (neq $cell^{-1}$)	Charge density per surface area (μeq m^{-2})
Chlorella vulgaris	Sphere: $4\pi r^2$ where r = 2		55	1.1×10^{-5}	300
Microcystis aeruginosa	Sphere: $4\pi r^2$ where r = 2.75		95	1.9×10^{-6}	40
Asterionella formosa	Cylinder: $2\pi r^2+2\pi rl$ where r = 1.4 and l = 40		370	6.8×10^{-5}	180
Melosira sp.	Cuboid = 2al + 2 ad + 2dl where a = 22; d = 22; and l = 55		6000	1.88×10^{-3}	310

Figure 2. Photograph of the bench scale dissolved air flotation jar tester (EC Engineering Dissolved Air Flotation Batch Tester, Model DBT6).

3) *Melosira sp.* is a large, rigid, filamentous diatom and has been specifically identified by water companies as a "problem" species that has proven difficult to treat.

Flotation Procedure

Batch Dissolved Air Flotation (DAF) experiments were undertaken using an EC Engineering Dissolved Air Flotation Batch Tester, Model DBT6 (Alberta, Canada) (Fig. 2). The coagulation/flocculation/flotation program comprised a 2 minute rapid mix (200 rpm), 15 minute slow stir (30 rpm), and 10 minute flotation time. Aluminium sulphate coagulant was added to 1 liter of algae suspension during the rapid mix stage at which time correction to pH 7 using 0.1 M HCl or 0.1 M NaOH as appropriate was undertaken. A coagulation pH of 5 was used in addition for *C. vulgaris* experiments to determine the impact of pH on zeta potential control. A recycle ratio of 12% and a saturation pressure of 450 kPa were used for flotation. Ionic strength was kept constant throughout the experiments at 2.3 mM using NaCl. It was ensured that the saturated water used in flotation matched buffering, ionic strength and pH conditions set for algae suspensions. At the end of the flotation period, samples were extracted to measure for residual cell count by microscopic analysis using the haemocytometer or Sedgewick Rafter cell as appropriate, where removal was presented as residual cells mL^{-1} divided by the initial cell concentration in cells mL^{-1}, referred to as normalised removal. Finally, all samples were tested for zeta potential as described in a later section.

Flotation experiments at pH 7 were repeated for EOM samples that were prepared by centrifuging the algae for 15 minutes at 10,000 G and filtering through a 0.7 μm filter (Whatman GF/F). Dissolved Organic Carbon (DOC) of the EOM was analyzed using a Shimadzu TOC-5000A analyzer.

Initial DOC was adjusted to 5 mg l^{-1} and the residual DOC and zeta potential of each system was analyzed.

Zeta Potential

Zeta potential measurements were obtained using a Malvern ZetaSizer 2000 (Malvern, UK). The ZetaSizer 2000 measures the electrophoretic mobility (EM) and then converts this to zeta potential (ζ) based on the Smoluchowski Equation (Equation (1)) which is appropriate when $\kappa a \gg 1$, where ε and μ are the permittivity and viscosity of the solution respectively.

$$EM = \varepsilon\zeta/\mu \quad (1)$$

Given the relatively high ionic strength (2.3 mM, equating to a κ value of 0.16 nm^{-1}) and algae cell size (3.2 μm or more) use of the Smoluchowski equation was appropriate for all cell systems. Consideration was also given to zeta potential measurements involving solely EOM, given that the colloid size was much smaller. However, based on molecular weight analysis of EOM (15), it can be assumed that the majority of the EOM had a radius greater than 7 nm, which is that required for $\kappa a \gg 1$.

All zeta potential results were obtained in triplicate. Furthermore, it was ensured that all samples extracted for zeta potential analysis had been exposed to the aluminium coagulant for at least 7 minutes. Previous research has demonstrated that zeta potential can take up to 7 minutes to stabilize after coagulant addition in algae systems depending on the dose administered (16). As anticipated, no difference was observed between zeta potential measurements obtained during the last five minutes of the slow stir period and at the end of the flotation period.

RESULTS AND DISCUSSION

Dose Response Curves

The algae cells under investigation in the current paper had cell surface areas that vary by orders of magnitude and cell charge densities that were similarly variable. For example, the surface areas of *M. aeruginosa* and *C. vulgaris* were 50 and 34 μm^2 $cell^{-1}$ respectively whilst their charge densities were 1.9×10^{-6} and 1.1×10^{-5} neq $cell^{-1}$ (Table 2). In contrast, *Melosira sp.* and *A. formosa* had much larger surface areas of 6000 and 370 μm^2 $cell^{-1}$ and charge densities of 1.88×10^{-3} and 6.8×10^{-5} neq $cell^{-1}$. Coagulant demands for the different algal systems were observed to vary by similar degrees (Fig. 3). For example, *M. aeruginosa* and *C. vulgaris* both had relatively small coagulant demands at optimum removal of 0.0014 and

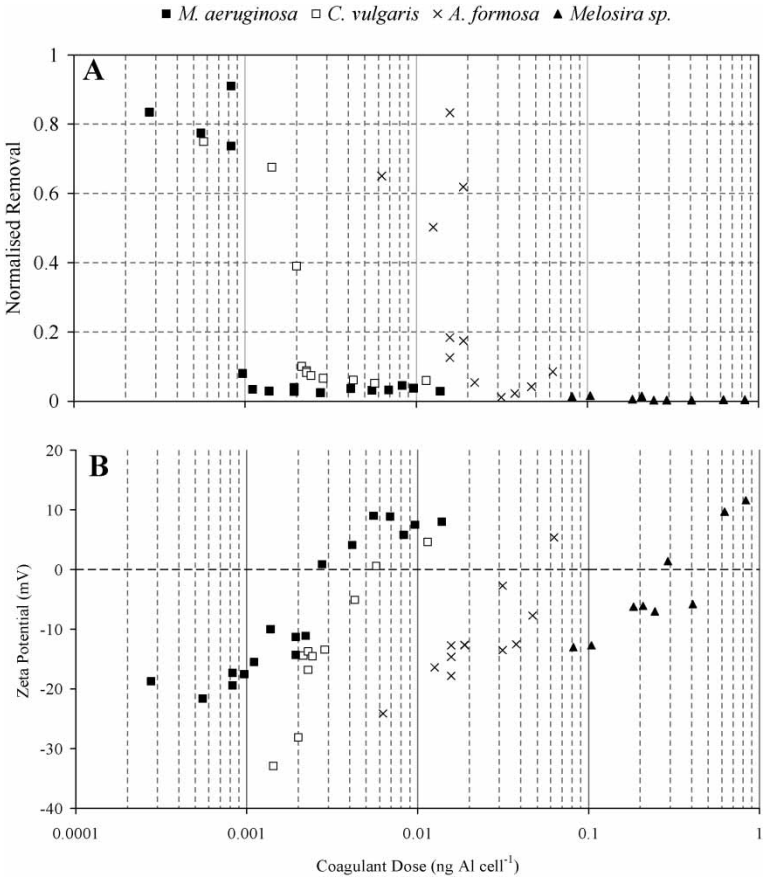


Figure 3. (A). Dose response curves for M. aeruginosa, C. vulgaris, A. formosa, and Melosira sp (17). and (B). corresponding zeta potential values.

0.0057 ng Al cell^{-1} respectively. It is interesting that despite the similarity of surface areas of *M. aeruginosa* and *C. vulgaris* cells, the much higher charge density of *C. vulgaris* resulted in approximately 3 times the coagulant demand. The much larger cells of *A. formosa* and *Melosira sp.* required 0.0314 ng Al cell^{-1} for 98.9% removal and 0.29 ng Al cell^{-1} for 99.7% removal respectively. This represents a respective increase of 22 and 207 times the coagulant required for *M. aeruginosa*. The increases in charge density for *A. formosa* and *Melosira sp*. were of the same order of magnitude at 35 and 990 times greater respectively compared to *M. aeruginosa*, while the surface areas were only 3.9 and 63 times greater. This indicates that increases in coagulant demand per cell are more strongly related to cell charge density rather than surface area. In the UK, the most common way of monitoring algae is by concentration, either by counting total algae cells

microscopically or, even more generically, by analyzing the chlorophyll *a* content in the influent water to give an indication of the overall algal activity. The implication of these results is that monitoring cell concentration will give limited information with respect to controlling the coagulant dose at a water treatment plant as it is the overall charge density that determines coagulant demand.

Charge neutralization was examined in the current study by zeta potential analysis. It was observed that for each species examined there was a decrease in the magnitude of the zeta potential which coincided with coagulant dose. For example, dosages of 0.0028, 0.0057, 0.05, and 0.29 ng Al cell^{-1} were required to neutralize *M. aeruginosa*, *C. vulgaris*, *A. formosa*, and *Melosira sp*. systems respectively (Fig. 3). The onset of optimum removal was observed as the zeta potential approached more neutral values. Hence, charge neutralization is an important mechanism for flocculation and subsequent removal of algae cells at pH 7, and additionally infers that there is a potential for utilizing zeta potential for process control. This has frequently been reported in previous studies. For example, optimum removal of *Cyclotella* and Chlorella using aluminium at pH 6.5 by DAF coincided with the reduction of the electrophoretic mobility (11).

The Zeta Potential "Operational Window"

The zeta potential operational windows for the species tested are varied (Fig. 4). To illustrate, optimum removal for *C. vulgaris* was observed at zeta potential values less negative than −16.8 mV, whilst that of

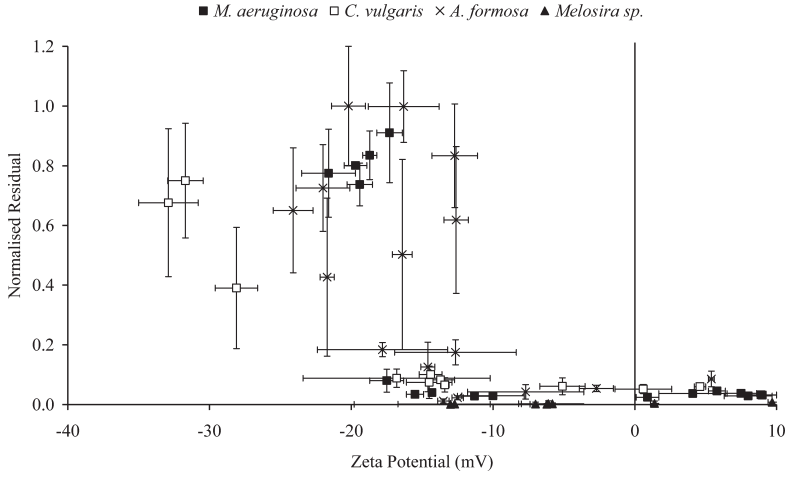


Figure 4. The zeta potential operational window for *M. aeruginosa*, *C. vulgaris*, *A. formosa*, and *Melosira sp*.

M. aeruginosa and *A. formosa* required zeta potential values less negative than −15.5 mV and −12.5 mV respectively. Good removal of *Melosira sp*. was observed at all dosages for which zeta potential values ranged from −13 mV to 11.6 mV. Furthermore, cell removal of up to 95% was achieved for *Melosira sp*. without addition of a coagulant. This good removal can be attributed both to the relatively low zeta potential observed for the system and to the relatively large size of the *Melosira sp*. cells, which were approximately 20 μm width by 55 μm length and linked together to form long filaments. This would mean that even at low coagulant doses good particle-bubble collisions and attachment could occur.

Overall, reduction of the magnitude of the zeta potential to approximately −10 mV and below would ensure removal for all algae species. It was noted that no decrease in removal efficiency was observed at positive zeta potentials. This is typical of coagulation experiments of organic components conducted at pH 7. At the relatively high doses required to instigate charge reversal, removal efficiency is not observed to decrease, generally attributed to a change in dominating the coagulation mechanism from charge neutralisation to sweep flocculation (6). This explains the observations that the system does not restabilize.

Similar operational windows have previously been observed when treating algae using aluminium based coagulants and DAF. For example, treatment of Cyclotella was optimized at zeta potential values less negative than −15 mV compared to −13 mV for Chlorella (12). Similarly, it was demonstrated that reducing the zeta potential to values less negative than −10 mV was required to ensure optimum removal for NOM when using a ferric based coagulant, irrespective of whether the zeta potential is altered using a coagulant dose or pH adjustment (10). Furthermore, the same study showed that if zeta potential values become too positive, +3 mV, then poor removal was observed (10). It has also been observed that zeta potential operational ranges may become smaller when sedimentation as opposed to DAF is employed for clarification. For example, operational windows of −8 mV to 0 mV and −5 mV to 0 mV were observed for a mixed algae sample and for a sample dominated by *Melosira* and *Pediastrum* respectively during sedimentation processes (17). However, Bernhardt and Clasen (11) showed that while zeta potential could be related to coagulant demand for many species, there was no relationship for cells with more complex morphologies, such as the diatoms *Stephanodiscus hantzchii*, which had long spines, and *Fragillaria crotonensis*, a large colony forming algae. This is in contrast to the current study where good correlation was obtained for complex species A. formosa and *Melosira sp*. The previous study utilised direct filtration and optimum coagulant demand was determined based on the run length. Direct filtration is susceptible to filter clogging when large algae of complex morphologies are introduced, which severely limits run times, irrespective of system zeta potential. DAF is not subject to these limitations and hence is a far more robust process for algae removal and therefore the removal of the large complex algae tested in the current paper (*A. formosa* and *Melosira sp*.) could still be controlled using zeta potential.

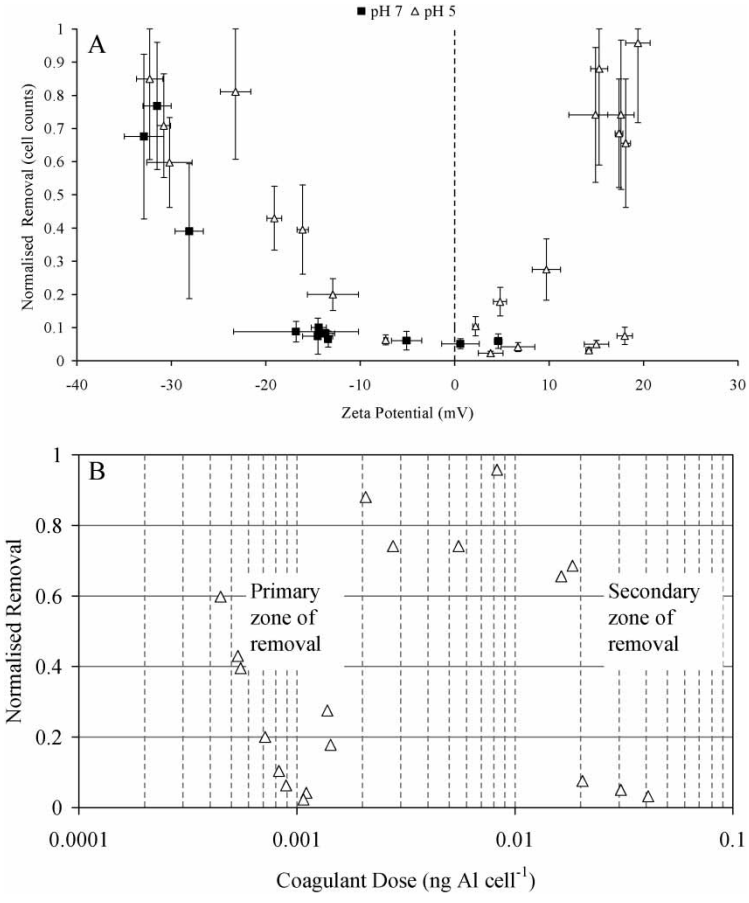


Figure 5. (A). comparison of the zeta potential operational window for C. vulgaris at pH 7 and pH 5 and (B). the corresponding dose response curve at pH 5 demonstrating two zones of removal.

The importance of pH was investigated for *C. vulgaris*, where the zeta potential operational window obtained for pH 7 was compared with that at pH 5 (Fig. 5). The primary observation was that optimum removal was obtained irrespective of pH if the zeta potential was maintained between −10 mV and +2 mV. Secondly, coagulation experiments conducted at pH 5 achieved more positive zeta potential values than those obtained at pH 7 and this coincided with a decrease in cell removal (Fig. 5). This was attributed to restabilization of the system as a result of the extremes of positive charge which cause electrostatic repulsion (6). The data points at extreme positive charge (+14 to +18 mV) that demonstrated good removal (Fig. 5) were obtained during a secondary zone of removal that was observed at high

coagulant doses (0.02 ng Al cell^{-1}) which can be attributed to sweep flocculation mechanisms. This follows a pattern commonly observed for NOM and kaolin systems (6). An additional observation was that the zeta potential operational window was narrower for pH 5 experiments. For example, at pH 7, optimized removal was obtained by −16.8 mV whereas at pH 5, by −6.1 mV only 60% of the algae had been removed. This can be explained by the presence of high concentrations of cationic amorphous aluminium hydroxide precipitates at pH 7 and low concentrations of dissolved cationic hydrolysis species which is in contrast to that which occurs at pH 5 (6). The precipitates can take part in sweep flocculation whilst the dissolved species are more important in charge neutralization. This suggests that at pH 7, the high rates of removal observed at relatively high negative zeta potential values (−16.8 to −13.4 mV) are a consequence of not only charge neutralization but also sweep flocculation mechanisms. It is interesting to note that there was no apparent difference in removal efficiency between pH 5 and pH 7. This supports conclusions made previously for NOM where it was stated that provided zeta potentials within the operational range were obtained, the pH or coagulant dose used to achieve that zeta potential was unimportant (10).

A further benefit of using zeta potential as a control method for removal is that it takes into account removal of both components of the algae system: algae cells and EOM. Overall, optimum EOM removal (measured as DOC) was observed at −10 mV or less (Fig. 6). Coagulant demands for optimum removal were 0.89, 1.25, and 1.56 mg Al/mg C (not illustrated here) for EOM of *C. vulgaris*, *M. aeruginosa*, and *A. formosa* respectively. In the case of *A. formosa*, the optimum EOM removal required the same reduction in zeta potential as was observed for the cells of approximately −12 mV. However, EOM originating from *C. vulgaris* and *M. aeruginosa* required a

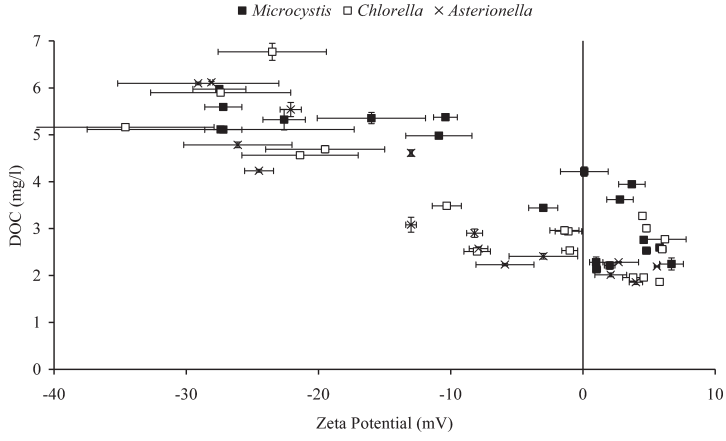


Figure 6. The zeta potential operational window for the EOM of *M. aeruginosa*, *C. vulgaris*, and *A. formosa*.

reduction in the zeta potential to at least −10 mV for optimum removal, whilst optimum cell removal was observed at −16.8 mV and −15.5 mV respectively (Fig. 4). Again, the zeta potential operational range observed for EOM was very similar to that observed for NOM (−10 mV to +5 mV) (10).

Overall, reducing the magnitude of the zeta potential to −10 mV for a pH between 5 and 7 ensured optimum cell removal was achieved. This was accomplished by either adjusting the pH or the coagulant dose, or a combination of these two actions. The removal efficiency did not depend on how the zeta potential was controlled for the two pH conditions examined. Optimum removal of the dissolved organic component of the organic system was also achieved at −10 mV or less, indicating that both components of the algal system are satisfactorily removed within the same operational window.

It is advised that a zeta potential operational window of −5 to 0 mV is targeted if using this technique for process control purposes. This range is within the operational window and additionally has outer margins to aid with process robustness. For example, if the algae population was to increase or decrease rapidly, as is frequently observed, there is leeway in the system for the zeta potential to be raised or decreased allowing time for pH or coagulant adjustment. At present one issue with using zeta potential for process control is the lack of an on-line instrument. Previously, studies have attempted to utilize a streaming current detector to determine coagulant dose using charge neutralization principles; (18) however, practically, these instruments have been unpopular due to difficulty in data interpretation and instrument calibration. The development of an on-line zeta potential meter would allow a relatively straightforward method for controlling coagulant demand.

CONCLUSIONS

It can be concluded that monitoring cell concentration will give limited information with respect to controlling coagulant dose at a water treatment plant. A more informative approach is desired for robust process control. It was determined that provided the zeta potential range was kept between −10 mV and +2 mV, through a combination of coagulant dose and/or pH adjustment as preferred, optimum removal efficiency of both cells and EOM occurred. This operational range is very similar to that required for optimal removal of NOM, suggesting that no matter the influent organic character, optimal particle/colloid removal should be achieved. Overall, the use of zeta potential for process control is a viable tool for algae removal.

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