Desalination and Water Treatment
www.deswater.com
16 (2010) 290–297
April
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doi: 10.5004/dwt.2010.1551

PACl coagulation for the solid–liquid separation of highly concentrated algae suspensions

Wen Po Cheng*, Wei Yi Chen, Ruey Fang Yu

Department of Safety, Health and Environmental Engineering, National United University, Miaoli, Taiwan 360
Tel. +886 (3) 7381764; Fax +886 (3) 7333187; email: cwp@nuu.edu.tw

Received 6 October 2009; Accepted 27 December 2009

ABSTRACT

The ability of algae to absorb CO_2, a greenhouse gas, for photosynthesis has received considerable attention in recent years due to the possibility of growing algae to extract into bio-energy. Therefore, technology that rapidly separates and recovers an algal mass from water is needed. In this study, the coagulant polyaluminum chloride (PACl) with high basicity (B) was applied as a chemical coagulant in a Jar Test for a solid–liquid separation study using two algal species, *Chlorella sp.* and *Spirulina sp.*, suspended in both freshwater and seawater. The coagulation process was monitored using an on-line photo-turbidimeter to examine the effect of chemical dosage and operating conditions on solid–liquid separation efficiency. Experimental results demonstrate that, for the 200 NTU suspension, *Spirulina sp.* required a much higher PACl dosage than *Chlorella sp.* for effective coagulation owing to the higher negative surface charge of the *Spirulina sp.* Additionally, a wide range of dosages achieved effective coagulation for both *Chlorella sp.* and *Spirulina sp.* when suspended in seawater, but not in freshwater. The minimum coagulant dosage for a seawater suspension was the same as that for a freshwater suspension, indicating that high concentrations of ions in seawater do not adversely affect the efficiency of PACl coagulation.

Keywords: Algae harvest; Coagulation; Polyaluminum Chloride (PACl); Turbidimeter; *Chlorella sp.*, *Spirulina sp.*

1. Introduction

In addition to attempting to reduce CO_2 emissions, many investigators have developed methods of disposing of or recovering CO_2 before it is discharged into the environment. Of these methods, the use of algae to fix CO_2 reduces CO_2 emissions and promotes recovery of resources. Therefore, this method is considered cost-effective [1, 2]. Based on molar weights, carbon from 1 kg of CO_2 increased the cyanobacteria mass by 25/44 kg [3]. In addition, microscopic algae are especially rich in oils and can be automatically cultivated in large basins or bioreactors [4, 5]. Hence, using algae to reduce CO_2 emissions and generate bio-fuel has become an important research topic [6, 7]. At the industrial level, bioreactors using microalgae to trap CO_2 and capture the sun's rays for photosynthesis are now under development. The algae must have access to large quantities of CO_2 in the basins or bioreactors where they grow. Notably, algae grow fast and can be harvested in a few days [5, 8]. Additionally, microalgal are light and separating or harvesting algae from liquid is typically difficult. Therefore, effectively separating or harvesting a microalgal biomass from water is considered the single most limiting factor in expanding the range of applications of microalgae for generating bio-fuel and other uses [9, 10]. Benemann and Oswald [11] reviewed available approaches for harvesting and concentrating a microalgal biomass for bio-fuel production and other uses. The main approaches are filtration, centrifugation, and chemical flocculation with either settling or flotation.

*Corresponding author.

Filtration is a very effective approach for harvesting microalgae [12]; however, small screen size with a large filter surface area is prone to clogging. In some cases, an algal mass is separated by centrifugation [13, 14]. The disadvantage of this method is its relatively high energy demand. Coagulation is a conventional and economic process for separating water-borne colloids, and can be also applied to separate algae. Since seawater must be the growth medium for mass production of algae, high concentrations of various ions in seawater may limit the use of some coagulants [15]. Therefore, using an appropriate amount of coagulants for algae harvested from seawater is a key to cost-effective recovery of the algal mass. Determining the optimal coagulation dosage that minimizes the amount of inorganic salts on a dry mass basis is also necessary [11].

Polyaluminum chloride (PACl) is commonly used to treat drinking water during coagulation to minimize turbidity and remove organic precursors of disinfection by-products [16–19]. The PACl consists of metal hydroxyl complexes of various charges. The degree of hydrolysis of polymeric salts can be controlled during PACl manufacture; therefore, the complicated reactions caused by the hydrolysis of the metal salt in coagulation can be minimized. Consequently, the use of the PACl provides a simpler and more precise way to control the reactions in coagulation [20–21]. Using PACl can markedly reduce the coagulant dosage and increase the range of optimal conditions for effective coagulation. Notably, PACl is particularly effective in treating highly turbidity raw water. Coagulation using PACl can be effectively controlled reaction mechanism to yield large flocs with rapid settling, thereby improving treatment efficiency [22, 23]. Therefore, PACl is a potential coagulant for use in solid–liquid separation to recover a biomass from highly concentrated algal suspensions. In this study, PACl with high basicity (B; B = $[OH^-]/[Al^{3+}]$), which was synthesized in the author's laboratory, was adopted for coagulation study and algal separation. *Chlorella sp.* and *Spirulina sp.*, dominant species in lakes and major sources of bio-fuel when suspended in freshwater and seawater, were used in coagulation studies [7, 24–30]. Coagulation was continuously monitored using an online turbidimeter designed in author's laboratory [31] to study the effect of various parameters on coagulation efficiency in freshwater and seawater.

2. Experimental equipment and methods

2.1. Preparation of algal suspensions

The algae used to prepare suspensions were cultivated artificially in the laboratory.

2.1.1. Chlorella sp.

Chlorella sp., a green alga that causes eutrophication in lakes and is presenting in bodies of freshwater worldwide, is a single-celled organism with an average cell diameter of 4 μm. The seed algae were obtained from the Tungkang Biotechnology Research Center. The seed algae were incubated aseptically in a 1 L glass container until high concentrations were generated. The suspension was then transferred to a 5 L cylindrical glass container. The initial ratio of algae to cultivation solution was maintained at 4:6; the contents were incubated with 1 L/min aseptic air under sideways fluorescent illumination at 3000–4000 Lux for 16 h/d.

2.1.2. Spirulina sp.

Spirulina sp., a species commonly found in health foods and used as a source of bio-energy, grows well in sub-tropical Taiwan. Thus, *Spirulina sp.* was selected for this coagulation study. The seed algae were also obtained from the Tungkang Biotechnology Research Center. *Spirulina sp.* is a multiple-cell helical organism with a cell diameter of 6–8 μm. After incubation in a 1 L glass container, the algal suspension was transferred to a 5 L cylindrical glass container for continual cultivation with sideways illumination at 4000–10000 Lux for 16 h/day.

2.1.3. Preparation of algal suspensions for coagulation studies

This study, confirms the optimal culture duration using particle counter (HIAC ROYCO 8000A) analysis. Experimental results indicate that *Chlorella sp.* and *Spirulina sp.* could obtain maximum concentration of algal culture after about 8 and 14 days. The cultivated algal suspensions were diluted with pure water until turbidity was 200 NTU (9.7×10^7 cell/mL for *Chlorella sp.* and 3.3×10^7 cell/mL for *Spirulina sp.*; both counted with a HIAC ROYCO 8000A Particle Counter. Initial pH was 7.8 prior the coagulation study. To simulate natural seawater, 34.2 g/L sea salt (Coral Marine, USA) was added to the cultivated algal suspensions. The gravity of the final solution was checked using a hydrometer to ensure that it was similar to the specific weight of seawater (1.022–1.023); pH and turbidity were maintained at 7.8 and 200 NTU, respectively. The algal solution was then used in the Jar Test.

2.2. Coagulant preparation

The synthesized PACl used in the coagulation study was prepared in the laboratory by adding 50 mL 0.46 M NaOH to 50 mL 0.2 M $AlCl_3$ solution at 0.05 mL/min

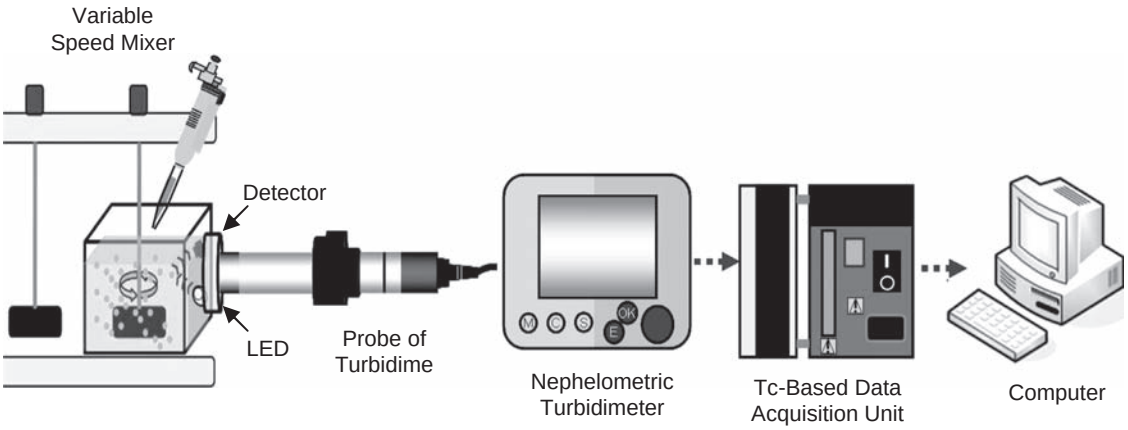

Fig. 1. Laboratory setup for conducting Jar test and continuously monitoring turbidity of coagulated algal solution.

using a peristaltic pump. After adding the NaOH, 100 mL final solution contained 0.1 M Al concentration and the synthesized PACl coagulant had a B value of 2.3. The prepared PACl solution was aged at room temperature for 7 d before use in the subsequent coagulation study.

2.3. Jar tests

Fig. 1 schematically depicts the setup for the Jar Test [31]. One liter of the prepared 200 NTU synthetic algae sample was placed in an acrylic flask. After the coagulant was added, contents were mixed rapidly (100–300 rpm) for 1 min and then mixed slowly (10–30 rpm) for 10 min before a settling period of 60 min. The final solution pH values after rapid mixing indicated that solution pH values exceeded 6.8 in the entire Jar Test.

2.4. Turbidity and zeta-potential measurements

A nephelometric turbidimeter was utilized for continuous online monitoring of suspension turbidity [31]. The turbidimeter probe (WTW model MIQ/C184) was placed 3 cm below the liquid surface (Fig. 1). All turbidity data were obtained using a data acquisition unit (YOKOGAWA model FX-106-0-2) and then sent to a computer. Data were collected at 1 s intervals and residual turbidity at different settling times was calculated using Microsoft Excel. To determine the surface charge of *Chlorella sp.* and *Spirulina sp.*, their zeta potentials were measured using a Zeta-Meter System 3.0 (Zeta-Meter, Inc., USA) over a range of pH values (4, 5, 6, 7, 8, 9 and 10) in 10 mM NaCl solutions. Samples with volumes of roughly 0.5 mL were extracted from the stirred vessel using a 2 mL syringe and transferred directly to a cuvette. Typically, 10 electrophoretic mobility measurements were made for each sample; measurements were made on at least two independent samples at each pH, and the mean electrophoretic mobility was computed directly from every mobility readings. The zeta potentials were calculated from mean electrophoretic mobility.

3. Results and discussion

3.1. Effect of coagulant dosage on removal of Chlorella sp. suspended in freshwater

Since an extremely large amount of algal suspension must be cultivated as a bio-fuel source, coagulation efficiency cannot be determined simply based on residual turbidity of the supernatant unlike in the general Jar Test. The settling property of the resulting floc is relatively important to algal solid/liquid separation in a full-scale plant designed for collecting algal masses. Fig. 2 plots the variation of residual turbidity with settling times of 300 s, 600 s and 1 h for the freshwater *Chlorella sp.*

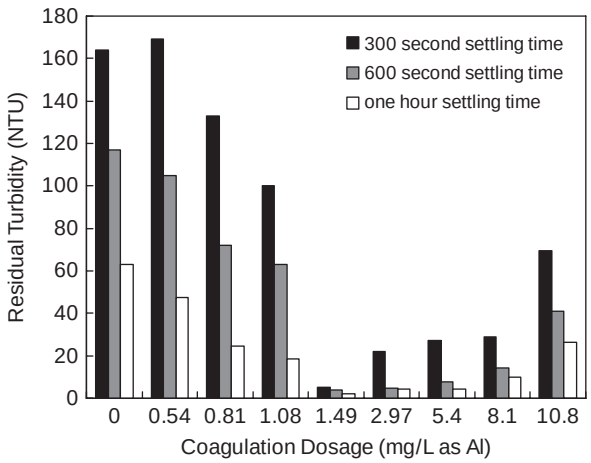

Fig. 2. Residual turbidity versus settling time for freshwater *Chlorella sp.* suspension coagulated with various coagulant dosages.

suspension coagulated using various PACl dosages mixed rapidly for 1 min at 100 rpm, and then mixed slowly for 10 min at 20 rpm. Experimental results at 1 h of settling time indicate that turbidity values with no PACl added (0 mg/L as Al) decreased to 63 NTU from 200 NTU. At a PACl dosage of 0.5–1.49 mg/L (as Al), the resulting flocs settled more rapidly than when no PACl was added. When PACl dosages were 1.49–8.1 mg/L, (as Al) residual turbidity did not vary significantly over the 1 h settling period, indicating that floc settling over a long period was satisfactory for this dosage range. However, when more than 1.49 mg/L (as Al) PACl was added over a short settling time (300 s or 600 s), residual turbidity increased as coagulant dosage increased, indicating that floc settling ability decreased as coagulant dose increased (Fig. 2). At a PACl dosage of 10.8 mg/L (as Al), both floc settling ability and turbidity removal efficiency were unsatisfactory, indicating that excessively high coagulant dosages reverse algal surface charges, thereby inhibiting floc formation. These experimental results demonstrate that 1.49 mg/L PACl (as Al) yielded the best coagulation and reduced residual supernatant turbidity to 3 NTU from 200 NTU, with only 300 s required for settling (Fig. 2).

3.2. Effect of coagulant dosage on removal of Chlorella sp. suspended in seawater

Fig. 3 plots residual turbidity results obtained using *Chlorella sp.* suspended in simulated seawater with an initial turbidity adjusted to 200 NTU. After adding various PACl dosages, samples were subjected to 1 min of rapid mixing at 100 rpm and 10 min slow mixing at 20 rpm. The simulated seawater contained relatively high

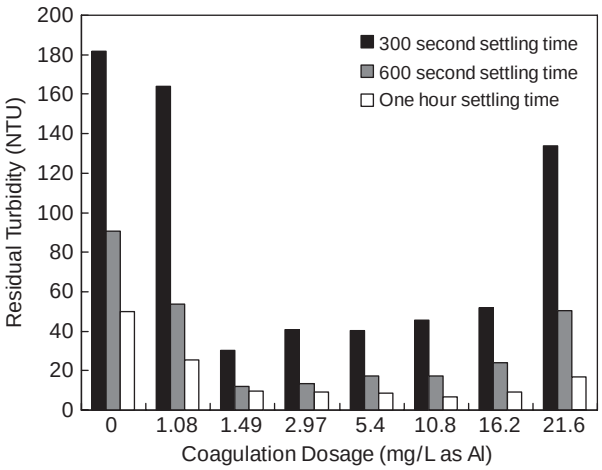

Fig. 3. Residual turbidity versus settling time for seawater *Chlorella sp.* suspension coagulated with various coagulant dosages.

background concentrations of ions, which theoretically reduce PACl coagulant activity; thus, a higher coagulant dosage was needed compared with that for freshwater [32]. However, required coagulant dosages differed only slightly; the required dosage was roughly 1.49 mg/L (as Al) in both seawater and freshwater (Figs. 2 and 3). Above observations imply that PACl used in this experiment had a relatively high basicity (B) and, therefore, was a long-chain polymeric compound [20, 21, 33]. Accordingly, its efficiency was likely unaffected by high concentrations of background ions, which effectively neutralize the algal surface charge. However, as the PACl dose was increased to 10.8 mg/L from 0 mg/L (as Al), residual turbidity during a 1 h settling period decreased. When the PACl dosage exceeded 21.6 mg/L (as Al), residual turbidities exceeded those obtained with 10.8 mg/L (as Al) PACl, indicating algal surface charge reversal. In the simulated seawater, the minimum PACl dosage associated with onset of surface reversal exceeded that for freshwater, and the coagulation range was wider. Overall, the lowest residual turbidity of *Chlorella sp.* suspended in seawater was 7 NTU (down from 200 NTU after a 1 h settling period). This process was slightly less efficient than removal of algae suspended in freshwater. However, increased buoyancy prolonged settling time for coagulated algae cells suspended in simulated seawater. The flocs at a dosage of 1.49 mg/L (as Al) required a settling time of 600 s to reach a similar residual turbidity as that after 1 h of settling (Fig. 3). The required 600 s settling time for seawater coagulation was longer than the required 300 s settling time for freshwater coagulation (Fig. 2) at same dosage 1.49 mg/L (as Al). Therefore, a higher buoyancy in seawater implies a longer settling time, which must be considered when designing sedimentation tanks for full-plant applications. These laboratory results indicate that PACl coagulation was effective in both freshwater and seawater, but the resulting flocs in simulated seawater required a longer settling time because of either their loose floc structure or the high buoyancy of seawater.

3.3. Effect of operating conditions on coagulation efficiency

Fig. 4 presents the coagulation results for *Chlorella sp.* at various high mixing speeds with a 21.6 mg/L (as Al) PACl dosage in simulated seawater. The variations in residual turbidity (Fig. 4) show that increasing mixing speed to 200 rpm from 100 rpm reduced residual turbidity after either 600 s and 1 h of settling, primarily because increasing mixing speed increased the uniformity of the coagulant distribution. However, when mixing speed exceeded 200 rpm, residual turbidity increased as mixing speed increased, indicating that an excessive mixing speed broke up the flocs, thereby increasing residual

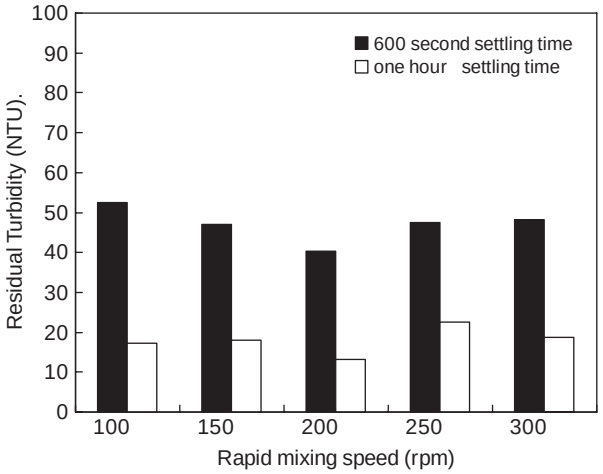

Fig.4. Effect of rapid mixing on removal of *Chlorella sp.* suspended in simulated seawater.

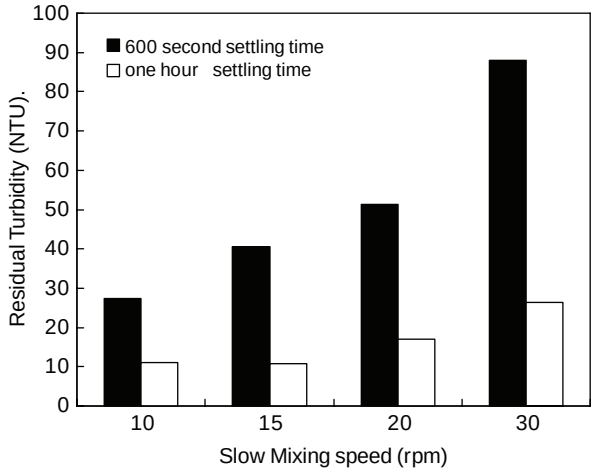

Fig. 5. Effect of slow mixing on removal of *Chlorella sp.* suspended in simulated seawater.

turbidity. However, floc formation at 100–300 rpm was not seriously affected (Fig. 5). When coagulation was tested with a PACl dosage of 21.6 mg/L (as Al) and a rapid mixing speed fixed at 200 rpm, and then at slow mixing speeds of 10–30 rpm, residual turbidity decreased as slow mixing speed increased, suggesting that a sufficiently slow mixing promoted agglomeration of destabilized flocs, thereby improving removal efficiency. Notably, slow mixing affected floc formation more than did rapid mixing (Figs. 4 and 5).

3.4. Effect of coagulant dosage on removal of Spirulina sp. suspended in freshwater and simulated seawater

Fig. 6 plots the variation in residual turbidity for coagulation of the 200 NTU *Spirulina sp.* suspension in freshwater with various PACl dosages, rapid mixing for

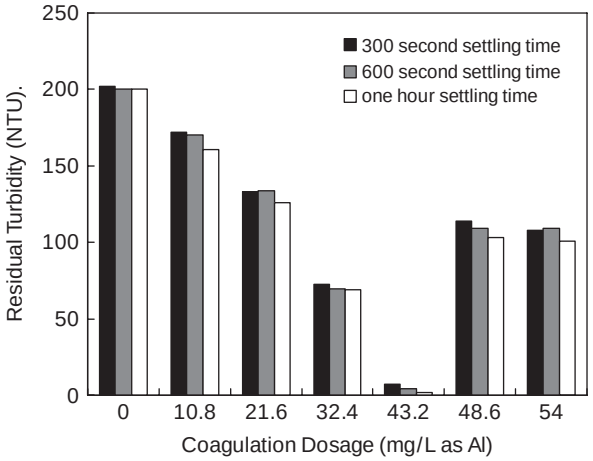

Fig. 6. Effect of coagulant dosage on removal of *Spirulina sp.* suspended in fresh water.

1 min at 200 rpm and slow mixing for 10 min at 10 rpm, with settling times of 300 s, 600 s and 1 h. Without PACl, algal cells exhibited no obvious sedimentation, even after a long sedimentation period. When PACl dosages were 0–43.2 mg/L (as Al), residual turbidity decreased as PACl dosage increased. Increasing the PACl dosages to > 43.2 mg/L (as Al) did not further reduce residual turbidity, indicating that adding excessive PACl reverses the surface charge on *Spirulina sp.* cells. Therefore, the optimal PACl dosage that maximized algal removal was 43.2 mg/L (as Al), and the corresponding residual turbidity values after settling for 300 s, 600 s and 1 h were only 7.4, 4.3 and 1.6 NTU, respectively. These experimental results show that *Spirulina sp.* flocs settled very rapidly, as rapidly as *Chlorella sp.* flocs. However, a comparing Figs. 2 and 6 reveals that when suspended in freshwater, more PACl is required to remove *Spirulina sp.* than that needed to remove *Chlorella sp.*. Accordingly, different algae species may require different coagulant dosages to achieve the same removal efficiency because of differences in cell size, shape and surface structure [34]. Coagulation does not occur as easily for algal cells that have smaller volumes than those in this study or those with different/peculiar shapes. Figs. 7 and 8 present the shapes of the *Chlorella sp.* and *Spirulina sp.* cells, respectively. Although the *Chlorella sp.* cells had a small volume (Fig. 7), they also had smaller surface curvature. In freshwater, they formed larger flocs with smaller voids and therefore settled faster than *Spirulina sp.* cells. In simulated seawater, the large flocs formed from *Chlorella sp.* cells had greater buoyancy than those in freshwater; that is, they settled slower than those in freshwater (Fig. 3). *Spirulina sp.* cells, which are multi-celled organisms, can form large flocs and settle rapidly. Hence, experimental results (Fig. 6) show that residual turbidity

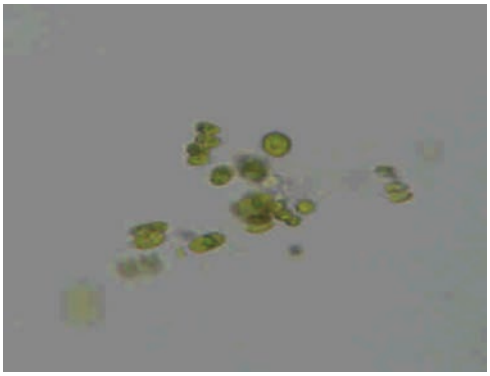

Fig. 7. Photomicrographs of *Chlorella sp.* cells (×400)

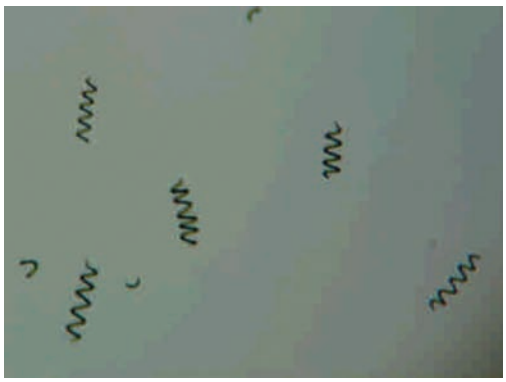

Fig. 8. Photomicrographs of *Spirulina sp.* cells (×400).

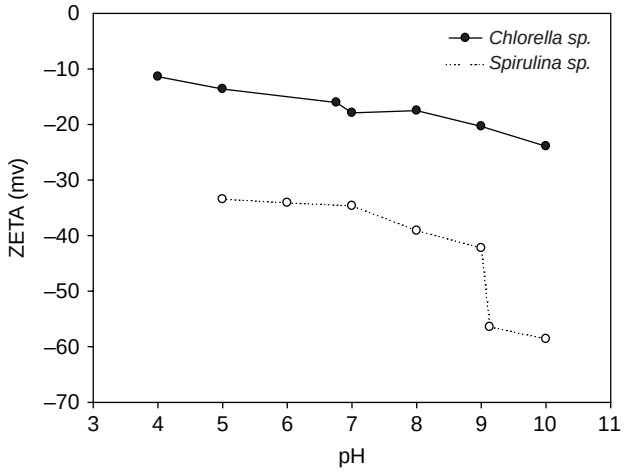

Fig. 9. Changes in surface zeta-potential at various solution pH values for *Chlorella sp.* and *Spirulina sp.*

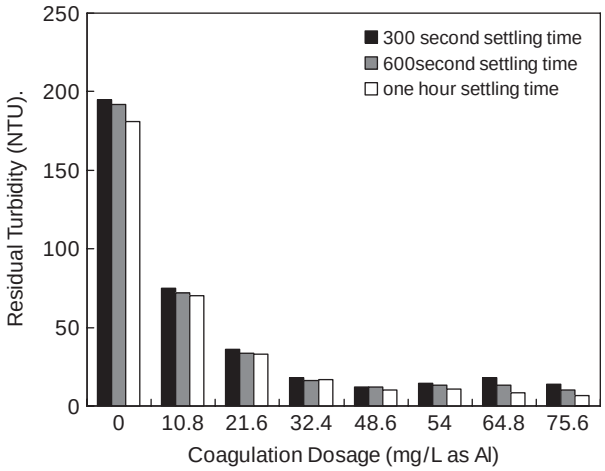

Fig. 10. Effect of coagulant dosage on removal of *Chlorella sp.* suspended in simulated seawater.

did not vary significantly for settling times of 300 s, 600 s and 1 h at any coagulation dosage. Like the cell structure, the surface charge on algae affected the coagulant dosage required. The measured zeta potentials of *Chlorella sp.* and *Spirulina sp.* at various pH values (Fig. 9) were −11.4 to −23.9 mv and −33.45 to −58.60 mv, respectively. Without adding a coagulant, but with sufficiently slow mixing speed, the natural coagulation of *Chlorella sp.* yielded flocs that led to partial removal of suspended cells (Fig. 2). However, for the *Spirulina sp.* suspension, natural coagulation in the absence of PACl was insignificant (Fig. 7) because the surface charge was high. These observations may explain why, in freshwater, the PACl dosage for effective removal of *Chlorella sp.* cells differed from that for *Spirulina sp.* cells.

Fig. 10 plots the residual turbidity of 200 NTU *Spirulina sp.* suspended in simulated seawater, coagulated with various PACl dosages and subjected to 1 min of rapid mixing at 200 rpm and 10 min of slow mixing at 10 rpm with 300 s, 600 s and 1 h of quiescent settling. When PACl dosage was <48.6 mg/L (as Al), residual turbidity in the coagulated sample decreased as the coagulant dosage increased, and residual turbidity remained constant when the PACl dosage was increased further to 75.6 mg/L (as Al), indicating that an excessive dosage did not reverse surface charges. Therefore, in the *Chlorella sp.* suspension, simulated seawater had a wider range of PACl dosages for effective coagulation than did the *Spirulina sp.* suspension. Moreover, the buoyancy of seawater decreased residual turbidity of the *Spirulina sp.* suspension to about 10 NTU, worse than the lowest residual turbidity of freshwater (1.6 NTU). However, the flocs that formed from elongated *Spirulina sp.* cells were less affected by the buoyancy of simulated seawater and had similar settling velocities in both freshwater and seawater.

4. Conclusions

This study demonstrates that, in both simulated seawater and freshwater, the use of highly basic PACl to separate *Chlorella sp.* and *Spirulina sp.* was feasible. In simulated seawater, the range of effective PACl dosages was wider than that for freshwater. However, the minimum effective PACl dosage in simulated seawater was the same as that in freshwater. The flocs in simulated seawater were subjected to a higher buoyancy than in freshwater, such that the coagulated seawater samples had higher residual turbidity. Additionally, the algal structure and surface electrical potential strongly affected the required dosage. *Spirulina sp.* cells have a helical structure and high surface charges requiring much more PACl than *Chlorella sp.* in both freshwater and simulated seawater. For mixing speed, although rapid mixing did not influence turbidity removal, slow mixing promoted algal removal.

Acknowledgement

The authors would like to thank the National Science Council of the Republic of China, Taiwan, for financially supporting this research under Contract No. NSC -96-2628-E-239-001-MY2.

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