



Harvesting of algae in municipal wastewater treatment by calcium phosphate precipitation mediated by photosynthesis, sodium hydroxide and lime



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ABSTRACT

The cultivation of microalgae within municipal and agricultural wastewater offers the opportunity to treat the water and partition nutrients into valuable biomass. Harvesting of those algae is a challenge, as conventional coagulants (iron and aluminum salts) contaminate algae and lock up phosphorus preventing beneficial reuse. We show that flocculation by calcium phosphate precipitation is capable of harvesting algae and phosphate from municipal wastewater in a form suitable for reuse. However, the initiation of flocculation by natural pH increase was infeasible. We harvested 93% of algal solids with an initial concentration of 113 mg VSS/L and 96% of total phosphate from a waste stabilization pond effluent by initiating flocculation with 129 mg-P/L of soluble phosphate and addition of 364 mg/L quicklime to achieve a pH of 10.0, at a calculated cost of \$0.05 USD per kL for alkali addition, which is similar to the chemical addition costs of conventional coagulation.

1. Introduction

Microalgal biomass can have an areal productivity orders of magnitude above that of conventional agricultural crops [17,27], and this has increased the global interest to cultivate microalgae for the production of e.g. biofuels, biogas, high-protein animal feed [7]. However, cost-effective harvesting of microalgal biomass is still a challenge [1,17,30]. Secondly, the sustainable supply of nutrients for large-scale microalgal cultivation is a problem, as it will compete with existing fertilizer utilization in agriculture [19]. Global phosphorus reserves are finite, and improving the sustainable use and reuse of this element is critical [8].

Cultivation of microalgae using municipal and agricultural wastewater in high rate algal ponds (HRAPs) partitions nutrients into algal biomass, which can be recovered and reused [3,9,20,21]. Unfortunately, the iron and aluminum salts regularly used by the water industry, as coagulant to remove algae and excess phosphorus from wastewaters, are toxic in concentrated form, contaminating the algae and preventing its use as feed, while aluminum in particular binds so strongly to phosphate as to be practically irreversible, preventing its use as fertilizer [2,16]. Some studies have explored the use of non-toxic flocculants such as cationic starch and chitosan on laboratory cultures,

however they are currently either too expensive or require further development to become commercially viable in the framework of wastewater treatment [23,28]. In addition, very little work has been performed aimed at recovering wastewater derived algae biomass using such flocculants at the pilot scale using real wastewater.

One potential method to separate algae and phosphorus from phosphate-rich wastewaters in a form suitable for reuse is flocculation by calcium phosphate precipitation. This phenomenon occurs when water containing dissolved calcium and orthophosphate ions experiences a rise in pH, e.g. by the photosynthetic activity of microalgae, resulting in the precipitation of amorphous calcium phosphate. These precipitates are thought to have a positive surface charge, and are electrostatically attracted to negatively charged algae cells [25]. Electrostatic attraction leads to enmeshment of microalgal cells within the charged precipitates, increasing their density and causing them to settle, which has been described by Sukenik and Shelef [24] as “auto-flocculation”.

Flocculation by calcium phosphate precipitation has already been demonstrated in laboratory cultures of *Chlorella vulgaris* by Beuckels et al. [4], and the presence of algogenic organic matter had a substantial inhibitory effect upon flocculation performance. This finding is important because e.g. municipal wastewater contains dissolved

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organic carbon (DOC), which is known to interfere with flocculation [24,29]. However, charge density measurements may be an interesting control tool for water characterization with respect to coagulant dosage [15].

This study aims to evaluate whether microalgae flocculation grown on municipal wastewater by calcium phosphate precipitation is achievable either naturally by algal photosynthetic pH increase, or using alkali such as sodium hydroxide or slaked lime. The conditions (dose, pH, calcium to phosphorus ratio) and the costs necessary to achieve effective flocculation followed by sedimentation were determined. Charge density measurements were conducted to explain the observed differences in required coagulant dosages between municipal wastewater and laboratory microalgal cultures.

2. Materials and methods

2.1. Water sample collection and characterization

Waste stabilization pond (WSP) effluent was collected from the final outlet weir of the Leanyer/Sanderson WwTP, which treats the municipal wastewater from the northern suburbs of Darwin, Northern Territory, Australia; a population of 48,000 equivalent persons. The pond system has two identical treatment trains, consisting of a facultative pond and four maturation ponds. Sample collection always occurred in the morning prior to performing experiments. The WSP effluent was analyzed for a range of physical and chemical parameters (Table 1) by the Northern Territory Government Department of Primary Industries and Fisheries Water Chemistry Laboratory in Darwin NT, Australia (National Association of Testing Authorities, Australia - Accreditation number 15104). Following parameters were determined based on the standardized methodology provided by the American Public Health Association (APHA) as described in [10]: biochemical oxygen demand (BOD₅ - APHA 5210B), chemical oxygen demand (COD - APHA 5220B), suspended solids (SS - APHA 2540D), volatile suspended solids (VSS - APHA 2540E), Free ammonia as N (NH₃-N - APHA 4500-NH₃ G), organic nitrogen as N (org-N - APHA 4500N-org D), nitrate and nitrite as N (NO_x-N - APHA 4500-NO₃⁻ E), filterable reactive orthophosphate as P (FR PO₄-P - APHA 4500P F), total phosphorus as P (TPO₄-P - APHA 4500P C), electrical conductivity (EC - APHA 2510B), pH (APHA 4500 H + -B), total alkalinity (APHA 2320B), bicarbonate alkalinity (HCO₃⁻ - APHA 2320B), carbonate alkalinity (CO₃²⁻ - APHA 2320B), hydroxide alkalinity (OH⁻ - APHA 2320B), total hardness

Table 1

Chemical characterization of WSP effluent effluents (i) Day 1 – pH increase by photosynthesis without addition of alkali, and pH increased by NaOH addition; (ii) Day 36 – used for investigation of flocculation induced by slaked lime; (iii) Day 49 – used for charge density measurements.

WSP sample date	(Day 1)	(Day 36)	(Day 49)
BOD ₅ (mg/L)	62	57	74
COD (mg/L)	250	200	300
SS (mg/L)	110	125	175
VSS (mg/L)	102	113	150
Free NH ₃ -N (mg/L)	15	1.3	1
Org -N (mg/L)	17	17	18
NO _x -N (mg/L)	< 0.1	< 0.1	< 0.1
FR PO ₄ -P (mg/L)	2.8	3.3	3.2
TPO ₄ -P (mg/L)	6.4	6.9	6.6
EC (μS/cm)	630	602	600
pH	7.74	7.59	8.01
Alkalinity (mg CaCO ₃ /L)	150	120	120
HCO ₃ (mg/L)	190	140	150
CO ₃ (mg/L)	< 10	< 10	< 10
OH (mg/L)	< 1	< 1	< 1
Total hardness (mg CaCO ₃ /L)	42.2	53.8	51.3
Ca (mg/L)	7	10	9
Mg (mg/L)	6	7	7

(APHA 3120B), calcium (Ca - APHA 3120B) and magnesium (Mg - APHA 3120B).

2.2. Flocculation by calcium phosphate precipitation using photosynthesis, sodium hydroxide or slaked lime

Flocculation-sedimentation of algae in municipal wastewater effluent by calcium phosphate precipitation was evaluated based on flocculation jar testing (Phipps and Bird PB-900 jar tester). Various concentrations of calcium phosphate were tested through addition of pre-prepared calcium phosphate stock solutions to the native dissolved calcium and phosphate present in the WSP effluent (Table 1). The calcium phosphate stock solutions were prepared by mixing disodium hydrogen phosphate (Na₂HPO₄) and calcium chloride (CaCl₂) in MilliQ water at various calcium to phosphorus (Ca:P) molar ratios (1.33:1, 1.5:1 and 1.67:1). These stock solutions were acidified using concentrated HCl (37%) until all formed precipitates were dissolved. After addition of calcium phosphate, pH was increase using three different methodologies: [1] pH increase by photosynthesis without addition of alkali (Table 1, day 1); [2] addition of 0.1 M sodium hydroxide (Table 1, day 1); and [3] addition of 1.8 M slaked lime (Table 1, day 36). For the addition of slaked lime, 100 g of commercially available lime (CaO, 88% w/w) was 'slaked' with 1 L MilliQ water. pH was adjusted for the flocculation experiments to four different levels (pH 7.0–8.0–9.0–10.0).

Flocculation was assessed based on a standardized jar test protocol [6]. 1 L of municipal wastewater sample was first mixed rapidly at 100 rpm during chemical addition, followed additionally by 5 more minutes of rapid mixing. This was followed by 30 min of slow mixing at 15 rpm to allow floc growth during flocculation, followed by 30 min of settling time. After settling, a sample was extracted, consistently 2 cm below the surface from each jar test to determine the separation efficiency based on turbidity analysis.

The separation efficiency was defined as the percentage reduction in turbidity before and after flocculation-sedimentation using Eq. (1) [13,14,29]:

$$E_f = ((T_i - T_f)/T_i) \times 100 \quad (1)$$

where, E_f = Separation efficiency (%), T_i = initial turbidity (NTU) and T_f = final turbidity (NTU). Prior to chemical addition, T_i of each jar was determined using a Hach H2100Q handheld turbidity meter.

The flocculated supernatant was analyzed for a range of physical and chemical parameters by the Northern Territory Government Department of Primary Industries and Fisheries Water Chemistry Laboratory in Darwin NT, Australia: BOD₅, NH₃-N, org-N FR PO₄-P, TPO₄-P and SS as described above. Due to limited sample volume, the clarified effluent from the triplicate jar tests was decanted from the settled floc and combined for water quality characterization.

2.3. Charge density analysis

The method of charge density measurement was adapted from the methods of Henderson et al. [15] and Kam and Gregory [18] using a Mutek PCD-05 (BTG Pty Ltd. Melbourne) streaming current detector with auto titrator and 0.001 N polydiallyldimethylammonium chloride (Polydadmac). The Polydadmac was purchased in standardized solution from BTG Pty Ltd. (Melbourne) and standardized with 0.001 N polyethylene sulfonate (PES; BTG Pty Ltd. Melbourne). Throughout the study, both Polydadmac and PES polyelectrolytes were regularly tested for degradation of charge equivalences, no degradation was detected. The required pH of the effluent was adjusted using either 0.1 M NaOH or 0.1 M HCl and was buffered using 0.1 M Tris(hydroxymethyl)aminomethane (TRIS) buffer for pH 7.0 and pH 8.0, and 0.1 M NH₄Cl for pH 9.0 and 10.0. The charge density of the effluent at each pH was determined through three colloid titrations, using 40, 50 and 60 mL of effluent respectively. Linear regression was performed, with the slope of

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