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Implementation of flocculation and sand filtration in medium recirculation in a closed microalgae production system

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ABSTRACT

Recycling growth medium is a necessity to reduce production cost and ecological foot print of large scale microalgae production systems. To prevent contamination and/or enrichment with particulate matter, medium recycling requires pre-treatment of the centrifuge supernatant (centrate), prior to medium replenishment and re-use. In this study, we investigated the applicability of high pH induced flocculation and/or sand filtration to interface with an existing microfiltration setup in order to prepare recycled growth medium for the mass cultivation of marine microalgae. Sand filtration partly alleviated the burden on subsequent microfiltration, but proved to be particularly useful to remove high pH induced flocs from the centrate. Combination of both techniques resulted in a removal of 78 ± 18% of particles, resulting in an improvement of 'modified fouling indices' by 75 ± 19%. Despite a partial to complete removal of remaining nutrients such as phosphate, calcium and magnesium during treatment, a cost saving of 72% compared to a scenario with fresh medium can be achieved.

1. Introduction

In order to tap into the enormous potential of microalgae as resource for bulk biofuel, feed/food and chemical applications, substantial reduction in production cost is needed [1]. Recycling culture broth is key to further decrease these costs [2], especially in cases where final applications call for the use of more expensive feed or food grade ingredients. At present, centrifugation is the most commonly used method for harvesting microalgae. Due to the limited separation efficiency inherent to the technique, a fraction of particles present in culture broth, including intact and broken algae cells, bacteria, and other debris are retained in the centrifuge supernatant (centrate). While in principal, this centrate could be reused as growth medium after nutrient replenishment [3–5], applying non-treated centrate as culture medium will contaminate and/or eventually enrich the production system with unwanted algae, protozoan grazers, cell debris and bacteria under normal field operating conditions. Therefore a recycling scheme for culture medium calls for post-treatment of the centrate prior to medium replenishment and re-use. At present, no practical post-treatment process is available that can feed into a microfiltration unit such as the one that is currently operated by us to provide sterile media to Proviron's ProviAPT™ production system. De Baerdemaeker et al. [6] reported that during microfiltration of centrate heavy fouling occurs which results in a severe loss of permeability. Dissolved algogenic organic matter (AOM) including polysaccharides, proteins and humic acid-like organics [7], are reported to cause substantial membrane fouling during microfiltration (MF) [8], ultrafiltration (UF) [9,10] and nanofiltration (NF) [11]. Pre-treatments prior to membrane filtration which decrease the concentration of both suspended solids and dissolved AOM of the centrate are therefore needed to reduce the load on the membrane and thus prolong its lifespan.

Coagulation pre-treatment using metal salts, such as aluminium and iron is commonly applied to control fouling in MF or UF systems [12,13] by removing both particulate and dissolved materials [14]. For our purpose, high pH induced flocculation is an attractive alternative because it is low-cost and is low in energy consumption. Furthermore, it is non-toxic [15], a prerequisite for further use of the cleared medium. The technique was found effective for harvesting microalgae [16,17]. Both NaOH and hydrated lime, $Ca(OH)_2$, are suitable to increase pH in a controlled manner [18]. Presence of AOM, however, interferes with the process [19], resulting in the need of a higher pH to induce flocculation [20]. In this study the applicability of high pH induced flocculation of centrate was investigated.

Sand filtration is frequently used as a cheap, easy and robust technique to remove suspended solids from water. The method has a broad application in the water treatment field, where it has proved its efficacy [21]. A few studies indicated the applicability of sand filtration to separate algae from the liquid broth [22,23]. However, the use of

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sand filtration as a pre-treatment technique to polish centrate has not been investigated previously. In this study the efficiency of sand filtration to eliminate suspended solids from the centrate prior to membrane filtration was researched.

Rapid sand filtration is usually preceded by coagulation, flocculation and/or sedimentation to increase its removal efficiency. Therefore, implementation of both techniques at optimum settings in a linked setup was also studied in order to assess which (combination of) approaches can be used in a large scale installation to reduce fouling and increase MF performance (shorter filtration times, longer filter lifespan).

2. Materials and methods

2.1. Algae cultivation – centrate collection

The centrate used in this study was obtained from a *Nannochloropsis* sp. culture cultivated semi-continuously in a 12 m^3 module of an outdoor production system of the ProviAPT™ photobioreactor [24,25] (Fig. 1).

The growth medium consisted of artificial seawater supplemented with nutrients in concentration ratios derived from the *f* formulation [26]. The seawater was prepared using a refined sea salt mixture (Zoutman Industries, Belgium) to obtain a salinity of 26 g L^{-1}. After addition of nutrients, the medium was filtered (0.2 μm, KrosFlo®, Spectrum®, USA) prior to pumping to the reactors. Algae were harvested daily and dewatered using an automated desludging disc centrifuge (SSD 18-06-007, GEA Westfalia, Germany), yielding an algae paste and a supernatant, the centrate. The density of the harvests which provided the used media, ranged from 1.2 to 3.1 g L^{-1}. These centrates were, in turn, characterised by dry weight and optical density. In addition, macronutrient concentrations of the centrates were determined. Magnesium, calcium and trace elements were only measured of selected centrates (2.5 Analysis).

2.2. General setup of flocculation experiments

High pH induced flocculation was investigated using conventional jar test experiments [27]. Centrate was divided in 200 mL portions in 1 L beakers and mixed using a magnetic stirrer. The pH was adjusted to five different levels ranging from 9.5 up to 11.5 with a 0.5 interval by addition of 1 M NaOH or $Ca(OH)_2$. The amount of alkali needed to obtain the desired pH was recorded. Samples were stirred at 300 rpm for 10 min following pH adjustment. Next, solutions were allowed to settle for 60 min after which a 1 mL sample was collected from the centre of the clarified zone. The flocculation efficiency η_a was determined based on the decrease in optical density of the cleared centrate as suggested by Vandamme et al. [27]:

$$\eta_a = \frac{OD_i - OD_f}{OD_i} \quad (1)$$

where OD_i is the optical density of the solution after sedimentation without pH adjustment (control), and OD_f is the optical density of the suspension after flocculation and settlement. Next, a 15 mL sample was taken from the cleared upper part of the solutions to investigate nitrate and phosphate removal. Remaining quantities were then stirred again and equivalent samples were taken from the blended mixtures. Both types of samples were neutralised with 2 M HCl and the amounts of acid required to reach their initial pH values were recorded. To assess improvement of filterability, the modified fouling index (MFI) was determined of selected samples from the upper part of flocculated solutions and of blended mixtures. At the end of the flocculation experiment, the morphology of settled flocs was documented using a BX51 Olympus microscope with differential interference contrast (DIC) fitted with an E410 Olympus camera.

Furthermore, calcium and magnesium removal from representative samples was calculated. The removal of trace elements due to flocculation was also investigated to determine the need for replenishment prior to reusing the medium. To this end, samples were taken from the top layer of NaOH flocculated centrate and, after neutralisation, elemental composition was determined. A chemical equilibrium model (Visual MINTEQ 3.0 [28]) was used to predict the theoretical manifestation and speciation of precipitates in standard medium with pH increase.

2.3. General setup of sand filtration experiments

The sand filter was constructed of a polyvinyl chloride column of 1 m with inner diameter of 45.2 mm. At the top and the bottom of the column a 3-way ball valve was installed to allow performing a backflush rinsing. After the top and prior to the bottom valve, a gauge was fixed to observe pressure increase (Fig. 2).

Sand with two different grain sizes was used to fill the column. 'Coarse' sand had a diameter between 250 and 500 μm, while 'fine' sand had a median particle size (Dv50) of 170 μm. Sabiri et al. [22] suggested a bed height of 40 cm to obtain a stable removal efficiency. In this study, the bed was packed with 35 cm of coarse sand and 5 cm of fine sand on top to improve separation efficiency. Preliminary experiments indicated that a higher sand bed of fine sand resulted in rapid blockage of the filter, decreasing the flow drastically.

Before filtration, the bed was completely fluidized by backwashing with water to remove air bubbles from the system and to allow sand

Fig. 1. View of a ProviAPT™ microalgae production plant of 48 m^3 culture volume.