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# Heat-aided flocculation for flotation harvesting of microalgae

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ABSTRACT

Microalgae biofuels are a promising renewable fuel that can be produced whilst mitigating industrial $CO_2$. However, their production is still not economically comparable to fossil fuels, with microalgae harvesting recognized as an area requiring development. Flotation is emerging as a promising harvesting method, but typically requires addition of flocculating chemicals to allow a foam concentrate to be formed and collected. In addition to adding to process costs, these additives contaminate the biomass and limit applications, such as for animal feed, for the non-biofuel component. An alternative method to achieve efficient harvesting is proposed that could use waste industrial heat to aid the flotation process and avoids the need for chemical additives. The best separation was achieved at 85 °C, when the *Scenedesmus* sp. culture was concentrated to 2.78 g/L from an initial density of 0.13 g/L. This result gives a concentration factor of 25.8 and a recovery of 83%.



## 1. Introduction

Microalgae with high lipid content have the potential to produce biofuel whilst also producing food supplements and other value-added products [1–3]. There are many other advantages to microalgae-based production, including that they do not compete with food crops [4], are capable of year-round production, and do not require pesticides or herbicides [2]. Microalgae production can be also coupled to industrial emissions for carbon capture [5–7] and wastewater treatment [4,8].

However, despite these advantages process development is required to improve production economics [9], including harvesting of microalgae prior to lipid extraction. It has been estimated that 20–30% of total production costs lie in harvesting [10,11] due to difficult separation processes [12,13].

The difficulty in harvesting arises from the nature of microalgae cells, namely; low concentrations [14], small sizes (typically 2–10 μm [10]), a specific gravity similar to water [15], a negative surface charge [11,16], and a requirement for frequent harvesting due to high growth rates [17].

The primary harvesting techniques currently available for microalgae separation are centrifugation, filtration, flocculation, sedimentation and flotation [17,18], electrophoresis [19] and membrane filtration [20].

Flotation has received significant attention due relatively low capital and operating costs, and the possibility for 90–99% recovery [21,22]. However, the process typically requires the addition of a surface-active chemical to make the microalgae cells hydrophobic so that they attach to rising bubbles [20,23]. Bubble surfaces and microalgae cells are typically both negatively charged, so recovery without chemical addition would otherwise be low [24–26]. Ferric chloride, aluminum sulfate, sodium dodecyl sulfate, chitosan, and acetyl trimethyl ammonium bromide are common chemical additives [10]. The addition of these chemicals unfortunately contaminates the biomass, whilst also contributing to the cost of the operation [18,27]. Contamination with metal salts can significantly limit application of the biomass remaining after lipid extraction for uses such as animal feed, fertilizer and in aquaculture [1,28].

In this study, heat-aided flotation without the use of any chemical additives that was originally developed for bacteria recovery [29] is examined as a potential new alternative separation method for dilute microalgae suspensions. As a stand-alone process, the heating of large volumes of microalgae culture is likely to be too expensive, but through utilization of waste heat from industry this approach could be used for on-site separation of process-coupled microalgae. The parameters examined include the influence of temperature and microalgae strain, as well any impacts on recovered lipid quantity and quality.

## 2. Materials and methods

### 2.1. Algae cultivation

*Scenedesmus dimorphus* #1237 (UTEX culture collection, University of Texas at Austin, TX, USA) and wild strains of microalgae were grown in Bold Basal medium [30]. The culture was incubated in a flask, in an Infors HT Multitron Standard (Montreal, QC, Canada) at

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25 °C, continuously agitated at 125 rpm, under photosynthetic light conditions of ~70–80 μmol photon $m^{-2}$ $s^{-1}$ (Sylvania Gro-Lux F15W/Gro T8, Infors) using a 12:12 hour light:dark cycle.

### 2.2. Algae collection

Microalgae strains were collected and screened as described in Eibl et al. [31]. The samples were subject to purification by serial dilution and then transferred to Bold's Basal Medium agar plates [32]. Identification to the genus level was performed using morphological analysis, as described by Bellinger and Sigee [33].

### 2.3. Temperature modification

Prior to separation, the temperature of each sample was raised in a water bath, taking 30 ± 2 min to reach 65 °C and 66 ± 2 min to reach 95 °C. After reaching the run temperature, the samples were immediately transferred to an ice bath and rapidly brought back to room temperature (21 ± 2 °C).

### 2.4. Flotation column design

A laboratory scale dispersed air flotation column (Fig. 1) was made using a clear acrylic tube with a porous stone sparger at the bottom (mean pore size of 15 μm, Refractron Technologies Corp., NY, USA). A collection chamber was located at the top with a weighted deflection plate to force the bubbles to concentrate. A side port was connected to an external water reservoir to maintain a constant level in the tube. Individual flotation tests were carried out with 500 mL of culture and a headspace of 1.5 cm. Separations were conducted with a run time of 5 min.

### 2.5. Separation analysis

Biomass concentration was measured with an Ahlstrom 151 glass microfiber filter according to Eq. 1:

$$C_{algae} = \frac{m_{final} - m_{filter}}{V_{filter}} \quad (1)$$

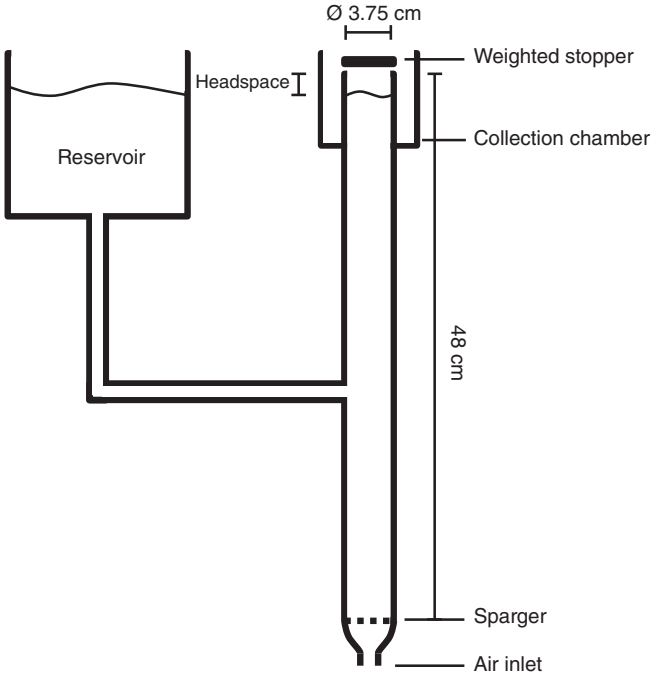


**Fig. 1.** Laboratory flotation column design.

where $C_{algae}$ (g/L) is the biomass of the measured algae. $m_{final}$ (g) and $m_{filter}$ (g) are the weights of dried sample and filter paper, respectively, and $V_{filter}$ (L) the volume of sample filtered.

The volume and the biomass concentration of concentrate was measured, along with the initial biomass of the culture, in order to calculate the concentration factor and collected (%), as shown in Eqs. 2 and 3:

$$\textbf{Concentration factor} = \frac{C_{concentrate}}{C_{initial}} \quad (2)$$

$$\textbf{Collected}\ (\%) = \frac{C_{concentrate} \cdot V_{concentrate}}{C_{initial} \cdot V_{initial}} \cdot 100\% \quad (3)$$

The concentration factor is the ratio of concentrate biomass concentration (g/L) to initial medium biomass concentration (g/L). The recovery (%) is the comparison of mass in the concentrate (g) to the mass initially in the column (g). These masses are calculated as concentration (g/L) multiplied by their respective volumes (L).

### 2.6. Lipid extraction and total lipid analysis

A modification of the lipid extraction described by Folch et al. [34] was performed. Microalgae samples (100 mg) were freeze-dried and mixed with 3 mL of chloroform:methanol (2:1 v/v) and sonicated using a Sonic Dismembrator Model 500 (Fisher Scientific, Ottawa, Ontario, Canada) for approximately 1 min. Samples were then centrifuged using an Allegra X-15R Centrifuge (Beckham, Palo Alto, CA, USA) and the solvent was removed to a weighted vial. Extraction was repeated in triplicate and the resulting extracts were combined. The combined extract was dried using a Savant DNA120 SpeedVac Concentrator (Thermo Electron Corporation, Milford, MA, USA).

### 2.7. Direct transesterification

Direct transesterification of the extracted lipid was performed as described by Velasquez-Orta et al. [35]. Microalgae samples (100 mg) were freeze dried and placed in a glass tube, where they were mixed with 2 mL of methanol:hexane (1:1 v/v). The reaction was initiated with the addition of sodium methoxide (100 μL) as a catalyst. The reaction was allowed to continue for 1 h, whilst being well mixed and maintained at 80 °C. After an hour, 0.5 mL of hydrochloric acid was added to neutralize the catalyst. The samples were centrifuged and the hexane layer containing the fatty acid methyl esters (FAME) extract was transferred to a gas chromatography vial for analysis.

### 2.8. Gas chromatography

The fatty acid methyl ester (FAME) composition was analyzed via gas chromatography using a Thermo Trace 1300 (Thermo Canada, Ottawa, Ontario, Canada) that was equipped with a flame-ionization detector (FID) and a SGE SolGel-Wax capillary column (30 m × 0.25 mm × 0.25 μm, Canadian Life Sciences, Peterborough, Ontario, Canada). Samples (dissolved in hexane) were spiked with C17:0 as an internal standard. Helium was used as the carrier gas at a flow rate of 1.6 mL/min. Standard split/splitless injection was used with a split ratio of 80 and an injector temperature of 250 °C. The column temperature was ramped from 140 °C to 240 °C at a rate of 4 °C/min. The detector temperature was 280 °C. An external standard, using a C4-C24 FAME mix, was used to identify peaks based on retention time, whilst peak area was used to quantify each FAME relative to the internal standard.

## 3. Results and discussion

It has been demonstrated that the capture of industrial waste heat, such as from ore smelters, to support microalgae cultivation in climates otherwise considered too cold for year-round production has significant