Continuous harvesting of microalgae biomass using foam flotation

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Abstract

Biomass harvesting and dewatering are major operational costs that constrain the development and expansion of the industrial use of microalgae; particularly for low value biofuels. Flotation-based technologies show promise as low cost, energy-efficient harvesters, producing a thickened algae slurry ahead of further dewatering steps. In this study we demonstrate, for the first time, a surfactant-aided foam flotation column that is designed and optimised for the continuous harvest of microalgae. The following operational parameters were optimised; surfactant concentration, air flow rate, feed flow rate, column height, liquid pool depth, and sparger type (i.e. bubble size). Additionally, the effects of cell surface characteristics (hydrophobicity, zeta potential, and contact angle) were investigated on *Chlorella vulgaris* flotation performance. Hydrophobicity was enhanced using three surfactants; the cationic cetyltrimethylammonium bromide (CTAB), the anionic sodium dodecyl sulfate (SDS), and the non-ionic TWEEN®20; with CTAB producing the greatest enhancement. Surfactant concentration, column height, and air flow rate had the greatest effect on the algae concentration factor (CF) and recovery efficiency (RE). The optimised design (CTAB = 35 mg L^{-1}, air flow rate = 1 L min^{-1}, feed flow rate= 0.1 L min^{-1}, column height = 146 cm, liquid pool depth = 25 cm, with a fine porous sparger) yielded RE of 95, 93, and 89% with 173, 271, and 143-fold biomass enrichments for freshwater *C. vulgaris* and marine *Isochrysis galbana* and *Tetraselmis suecica* microalgae respectively. Achieving high RE for freshwater and in the case of marine microalgae (irrespective of ionic strength) at moderate surfactant dosages, gives foam flotation the advantage of being a growth media independent harvesting process. The process had a very low power consumption (0.052 KWh m^{-3} of algae culture). Our findings demonstrate the potential for continuous, low cost, scalable flotation harvesting with particular relevance for the biofuels, water and wastewater treatment industries.

Keywords: Absorptive bubble separation; Algae biofuels; Biodiesel; Hydrophobicity; Microalgae harvesting

1. Introduction

Concerns about the sustainable use of fossil fuels, fluctuating oil prices, environmental pollution, and global climate change are driving moves away from conventional fuels to biofuels, including those derived from microalgae [1-3]. Microalgae are fast growing, photosynthetically efficient oleaginous organisms that can be cultivated in freshwater, brackish, and full strength seawater, together with a range of nutrient impacted wastewaters. Microalgae have the potential (as yet unrealised due to lack of cost competitiveness) to play a vital role in the biofuels market [4-10]. Biofuels aside, there are established markets for microalgae biomass and extracts in the cosmetics, nutraceuticals, and

pharmaceuticals industries. Equally, microalgae are both a problem and an opportunity for water utilities and the wastewater industry.

Harvesting and dewatering of the algae biomass represents a substantial process cost, accounting for an estimated 20-30% of the total cost of production [11-13]. Harvesting from dilute algae suspensions is challenging due to the small cell size translating to a low specific gravity, as well as the cell surface being negatively charged, thereby maintaining a stable colloidal suspension. Other impediments stem from the ionic strength of the culture medium due to salinity, hydrophobicity, pH and culture age [11, 14]. Consequently, there are a number of challenges inherent in microalgae harvesting such as a low recovery efficiency and/or high capital and operating costs.

A cost effective and reliable technique for bulk harvesting has yet to be adopted across the microalgae sector [15-17]. A wide range of solid-liquid separation techniques have been trialled, both individually and in combination, such as coagulation and flocculation, followed by sedimentation, flotation, centrifugation, or filtration (Fig. S1). Gravity sedimentation is a very simple solid-liquid separation method and is commonly used to separate microalgae from water; however, it is time-consuming due to long settling times and requires large land areas for settling ponds. Moreover, the total suspended solids from sedimentation is low which increases the cost of further downstream processes [18]. Therefore, sedimentation is rarely used alone to harvest algal biomass and is often paired with coagulation and flocculation. However, flocculation is currently uneconomical as the amount, and hence costs, of flocculent necessary for large scale harvesting is prohibitive [8]. Centrifugation is the most rapid and suitable harvesting technique for a wide range of microalgae species. However, it is energy-intensive (requiring as much as 3000 kWh ton^{-1}; Schenk *et al.* [19]). Filtration is highly dependent on the size of the microalgae, is abrasive to many species and is energy intensive due to pumping. Frequent replacement or backwash of filters are other disadvantages [20]. A successful harvesting system needs to be effective, rapid, low cost, species independent, scalable, and should be able to operate continuously if required. An added benefit would be the potential to partially process the biomass *in situ*, e.g. weakening of the cell wall prior to conversion into biofuel [16, 21].

Due to its simplicity and low capital and operating costs, adsorptive bubble separation is widely used in industrial and domestic wastewater treatment, and in the mining, pharmaceutical, and food industries [22-25]. Foam flotation, which is a subclass of adsorptive bubble separation, shows considerable promise as a microalgae biomass harvesting and enrichment method. Flotation columns have many advantages over conventional flotation cells, including; simple construction, lower capital and operating cost, improved recovery, higher grade products, less wear and tear due to the absence of moving parts, and a smaller footprint [26].

It is energetically unfavourable for hydrophobic particles to remain wholly within the liquid phase, preferring to adsorb onto the surface of bubbles which will transport them to the liquid surface for collection and removal [27]. Most microalgae are weakly hydrophobic, especially those that are algaenan-free like *Chlorella vulgaris* [28, 29]; therefore, surface-active materials (surfactants) are added not only to stabilise the foam in the system but also to enhance microalgae hydrophobicity. The foam flotation process involves generating bubbles by gas flow, either through a porous or jet sparger. Destabilised microalgae and free surfactant will adsorb onto the bubbles and are removed from the column as foam [30]. Foam is an effective medium to adsorb microalgae as it possesses a high specific surface area which results in a high RE whilst only a small volume of interstitial liquid is collected, enabling good biomass enrichment.

The effectiveness of a solid-liquid separation process is determined by the concentration factor (CF) and the recovery efficiency (RE). CF is the ratio of the microalgae concentration in the final product to the microalgae concentration in the culture whereas RE is the ratio of the microalgae cells in the final product to the microalgae cells in the culture. Previous surfactant-aided flotation harvesting research has been performed in batch or semi-batch modes, with RE of up to 97% [31-38]. When combined with electro-flocculation an RE of 98.9% was achieved [39]. In a forerunner to the present study, Coward *et al.* [18] harvested *C. vulgaris* in batch mode, attaining a high CF of almost 230 but at the expense of RE. For most bulk harvesting techniques, especially flotation operating in batch or semi-batch modes, it is challenging to realise an effective combination of a high RE (for greater biomass removal from the growth medium) and CF (to lower downstream dewatering and drying costs). Few reported works on bulk harvesting have focused on the combination of RE and CF due to the trade-off between them. For instance, Garg *et al.* [37] recovered 85% of *Tetraselmis* sp. using mechanical flotation but at the expense of enrichment in which only six-times more concentrated microalgae was obtained. However, this shortcoming may be overcome if a pivotal combination between the factors affecting both the RE and CF may be achieved in a continuous foam flotation column. Continuous mode harvesters are also more suitable for high throughput applications such as biofuels production, whereas batch or semi-batch modes have more downtime and typically have higher resource demands (i.e. space and energy). Furthermore, commercial scale algae production is typically continuous or semi-continuous; there is thus a demand for the capability to harvest continuously.

The present work developed and optimised a foam flotation column to continuously harvest *C. vulgaris*, *Isochrysis galbana* and *Tetraselmis suecica*. This work also aimed to evaluate the effectiveness and economic feasibility of the process. To the best of our knowledge, this is the first

study to demonstrate low cost and continuous microalgae harvesting using foam flotation with a focus on both biomass recovery and enrichment.

2. Materials and methods

2.1 Microalgae culture

Freshwater *Chlorella vulgaris*, and marine *Isochrysis galbana* and *Tetraselmis suecica* were grown using BG-11 and f/2 media in seven polycarbonate carboys (Nalgene 10 L) at 20 ± 2 ºC in a non-sterile environment. Photoperiod was 16L:8D using a combination of cold and warm fluorescent lights with an average illuminance of 2,500 lux. The cultures were agitated by HEPA filtered (0.2 µm) aeration using an aquarium air pump (Blagdon, Koi Air, KA50, 0.032 mPa), and maintained semi-continuously.

2.2 Surfactant types

Three surfactants were used; the synthetic anionic foam stabilizer sodium dodecyl sulphate (SDS, $CH_3(CH_2)_{11}OSO_3Na$; AMRESCO, USA); the non-ionic emulsifier and detergent TWEEN®20 (polysorbate 20, $C_{58}H_{114}O_{26}$; Sigma-Aldrich, UK); and the common quaternary ammonium cationic surfactant cetyltrimethylammonium bromide (C_{16}TAB, $CH_3(CH_2)_{15}N(Br)(CH_3)_3$; G-Biosciences, USA). CTAB has been demonstrated as the most suitable surface-active material to remove algal biomass from wastewater [16, 18]. It has also been used in wastewater treatment and in the extraction of DNA [40, 41].

2.3 Hydrophobicity tests

Hydrophobicity tests on *C. vulgaris* were carried out using a modified microbial adhesion to hydrocarbons method [42, 43], with or without the addition of 20 and 40 mg L^{-1} of CTAB, 20 and 40 mg L^{-1} of SDS, and 2 and 4 mL of TWEEN 20. Hydrophobicity was also measured after addition of 70 and 100 mg L^{-1} of trivalent aluminium chloride salt, $AlCl_3$ (Sigma-Aldrich, UK) in the presence of 40 mg L^{-1} of SDS. In this method, 8 mL of microalgae culture, 0.46 ± 0.13 g L^{-1} concentration (dry weight equivalent to $9.58 \times 10^6 \pm 1.1 \times 10^6$ cells mL^{-1}, which approximates to cell densities within raceway based microalgae production systems [44]) was placed in a test tube, in duplicate. Two millilitres of n-hexane (95% purity, Sigma-Aldrich, UK) was then added to each tube and shaken vigorously for one minute; the resulting suspension was allowed to settle for two minutes. Afterwards, 2 mL was carefully drawn from the aqueous layer at the bottom of each tube, placed in a UV cuvette, and the absorbance read at 620 nm using a spectrophotometer (Jenway, Model 7315, Bibby Scientific Ltd, UK); this allowed the proportion of cells that had moved to the water-hexane interface to be determined. The hydrophobicity (H) of the algal suspension was calculated using equation 1:

$$H = \frac{A_o - A_{aq}}{A_o} \times 100\% \quad \cdots (1)$$

where: A_o is the absorbance of the microalgae suspension before n-hexane addition and A_{aq} is the absorbance of the aqueous phase after n-hexane addition. Based on the hydrophobicity data, only CTAB was carried forward for optimisation and harvesting trials. The data from the hydrophobicity experiment were compared using an ANOVA test with Dunnett comparison procedure with an alpha level of 0.05.

2.4 Adsorption isotherm

The concentration of CTAB adsorbed onto *C. vulgaris* was determined by surface tension. A calibration curve was created for CTAB in the 0-20 mg L^{-1} range versus surface tension measurements using a microtensiometer (Kibron EZPi^{plus}, Finland) when dissolved in 1 L of water separated from algae culture by centrifugation. Culture medium was used rather than deionized water due to the presence of ions in the medium which may alter surface tension readings. Two different concentrations of algae culture were used; 1.2±0.01 and 0.68±0.01 g L^{-1} (equivalent to $24.1 \times 10^6 \pm 2.6 \times 10^4$ cells mL^{-1} and $14.2 \times 10^6 \pm 2.2 \times 10^4$ cells mL^{-1} respectively). The mixture (20 mg of CTAB in 1 L of algae culture) was stirred continuously for 15 min using a magnetic stirrer. Two 10 mL samples were centrifuged for 30 min at 15,000 rpm (25,155 RCF) to separate the algae from the medium. The supernatant was collected and the surface tension measured to determine the concentration of un-adsorbed surfactant that remained in the medium.

2.5 Zeta (ζ) potential experiments

Colloidal systems such as microalgae suspensions consist of highly dispersed particles (discontinuous phase) distributed uniformly throughout a dispersion medium (continuous phase) [45]. The magnitude of the zeta (ζ) potential is a key characteristic in the colloidal system as it gives an indication of the suspension stability. The ζ-potential of *C. vulgaris* was measured herein with or without the addition of CTAB at different pH values (4, 6, 8, and 10) using a Zetasizer Nano ZS ZEN3600 instrument, Malvern Instruments Ltd., UK. To study the effect of ions from the culture medium on microalgae ζ-potential, trials were performed after resuspension of microalgae in freshwater. In a typical experimental trial with surfactant addition, l L of microalgae culture, 0.46 ± 0.13 g L^{-1} concentration dry weight (equivalent to $9.58 \times 10^6 \pm 1.1 \times 10^6$ cells mL^{-1}), was mixed with approximately 35 mg L^{-1} of CTAB and the mixture was stirred continuously for 15 min using a magnetic stirrer. Four 50 ml samples were collected from the mixture and the pH adjusted using NaOH and HCL solutions. To study of effect of culture ions on ζ-potential, 1 L of microalgae culture

was centrifuged for 10 min at 4,000 rpm and re-suspended in freshwater. The ζ-potential measurements were carried out in triplicate.

2.5 Measurement of contact angle

The contact angle of *C. vulgaris* cells, in the form of algal strata on membrane filters, was measured based on the sessile drop technique using a goniometer (model 250, Rame-Hart, USA) with DROPimage advanced software. The contact angle measurements were performed with and without CTAB addition. Algae with a concentration of 0.46±0.13 g L^{-1} dry weight (equivalent to $9.58 \times 10^6 \pm 1.1 \times 10^6$ cells mL^{-1}) were deposited on a filter (cellulose nitrate membrane, 1.25 μm pore size, 25 mm diameter, MFS) using a syringe filter. CTAB (20, 30, and 40 mg L^{-1}) was dissolved in a 1 L algae culture and stirred continuously for 15 min using a magnetic stirrer prior to filtration. The obtained algal mats were placed on an agar plate to prevent them from drying until the measurements were made. Contact angle measurements were performed in triplicate with deionized water as a probe liquid. Deionized water has been successfully employed in contact angle measurements of various microorganisms including yeasts, bacteria, and algae. For the measurements, the filter papers were taken from the agar plate and fixed to glass slides, then dried in air for 50 min. After air-drying, the filter papers were stored in a desiccator over silica gel until use. Readings were recorded after 0.5 sec of the probe liquid deposition (volume of 5 μm), and each sample was tested ten times within 1 sec [46, 47].

2.7 Foam column dimensions

A bench scale flotation column was used as shown in Fig. 1. The column was constructed from poly(methyl methacrylate) with a 5.15 cm internal diameter. Column height could be adjusted between 30-160 cm by attaching different tubular modules of 25, 30 or 50 cm lengths. The inlet mixture consisted of algae culture with added surfactant from a 25 L reservoir. The processed culture was discharged to waste from the outlet stream valve at the base of the column, 1 cm above the sparging media. A magnetic stirrer was used to mix the microalgae culture with the surfactant in the feed tank for 10 mins before and during the harvesting experiments. The feed flow rate was measured and controlled by a valve with an ultrasonic flowmeter (Atrato, Titan, UK). Another valve was placed on the discharge stream to control the liquid depth in the column. The foam was collected at the top of the column using an annular trough of 30 cm in diameter and 15 cm depth. Low-flow air was fixed against foam flow at the outlet of the foam column to enhance foam collapse. Air bubbles (dispersed phase) were generated by introducing compressed air through a sparger. Two different spargers made from ultra-high molecular weight polyethylene were used with a thickness of 6.0 mm, a diameter of 51.5 mm, and mean pore sizes of 30 and 158 μm for fine and coarse porosity respectively. The air

flow rate for each trial was adjusted before the inlet mixture was fed to the column to prevent liquid weeping into the gas line.

2.8 Harvesting effectiveness criteria

The effectiveness of the harvesting process was determined by the concentration factor (CF; equation 2) and the recovery efficiency (RE; equation 3).

$$CF = \frac{Concentration\ of\ algae\ in\ final\ product}{Concentration\ of\ algae\ in\ inlet\ stream} = \frac{\left(\frac{cell}{ml}\right)_{foamate}}{\left(\frac{cells}{ml}\right)_{inlet}} \quad \cdots (2)$$

$$Recovery\ efficiency\ (RE) = \frac{cells\ of\ algae\ in\ final\ product}{cells\ of\ algae\ in\ inlet\ stream} 100\% \cdots (3)$$

A calibration curve was constructed correlating cell density and their corresponding absorbance at 750 nm using a spectrophotometer (Jenway, Model 7315, Bibby scientific Ltd, UK), yielding an R^2 of 100% (data not shown). The wavelength of 750 nm was selected as the absorption by chlorophyll and most other pigments is at a minimum [48]. Cells density was measured using an improved Neubauer hemocytometer, with a Leica DM 500 light microscope.

The algae dry weight was measured by the following procedure: Whatman quantitative filter paper, grade 42, was dried at 103 ˚C for 3 hr then left to cool in a desiccator over silica gel until use. A pre-dried paper was weighed and a known culture volume (v), approximately 10 mL, was filtered after placing the pre-weighed paper in the filter unit then dried at the same conditions as above and stored in the desiccator overnight. The dried paper was weighed and the dry weight concentration determined according to equation 4:

$$DWC = \frac{weight\ of\ dried\ paper\ containing\ algae - weight\ of\ filter\ paper}{Volume\ (v)} \quad \ldots (4)$$

2.9 Design of experiments

Design of experiments (DOE) is a statistical and mathematical tool used to evaluate and optimise the direct and crossed relations between independent variables and system responses. It is an advantageous method for minimising the number of experimental trials needed for process optimisation wherein rigorous modelling is intractable to apply due to the complexity of the system being investigated [49].

2.9.1 Fractional factorial design

A fractional factorial design approach using Minitab software (release 17, Minitab Inc., State College, PA) was applied as a screening tool prior to response surface methodology. The aim of performing the fractional design of experiments was to select the most appropriate sparger for subsequent use in the response surface design. Process variables were; surfactant concentration, airflow rate, column height, feed flow rate, liquid pool depth, and sparger type. Other factors such as pH were not studied, and thus kept constant. The screening trials were conducted on *C. vulgaris* only and the algae concentration in the inlet stream was held at 0.46±0.13 g L^{-1} concentration dry weight (equivalent to $9.58 \times 10^6 \pm 1.1 \times 10^6$ cells mL^{-1}) [44]. A two-level fractional factorial of a resolution IV, ($2^{(6-2)}$), plus two central points was adopted. The lower and higher values of the lower and upper levels for each factor are represented by -1 and +1 in Table 1.

2.9.2 Response surface design

Once the factorial design evaluation had been completed, a five level half-unblocked Central Composite Design (CCD) with six central points was applied to identify the key process variables, their combinations, and to obtain an optimal higher degree model. CCD was adopted as it provides high quality predictions over the entire design space [50]. The factors of interest were surfactant concentration, air flow rate, column height, feed flow rate, and liquid pool depth. CTAB and the fine porous sparger were used in the CCD trials based on results from the previous experiments. Other factors such as pH were kept constant. The harvesting trials were conducted on *C. vulgaris* only and the algae concentration in the inlet stream was held at 0.46±0.13 g L^{-1} concentration dry weight. Thirty-two experiments were generated and randomized with a repetition of factorial experimental runs i.e. 48 experiments. The five coded levels and their corresponding values of the factors are shown in Table 2.

The CF and RE responses as a function of the independent variables above were fitted to polynomial quadratic regression models given in equation 5 [51]:

$$Y = \beta_o + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i,j=1}^{k} \beta_{ij} x_i x_j \quad \cdots (5)$$

Where: Y is the predicted response; β_o is the intercept term; β_i is the linear effect coefficient; β_{ii} is the squared effect coefficient; β_{ij} is the interaction effect coefficient; and x_i and x_j are the independent variables.

The goodness of fit of the obtained models was assessed by the lack-of-fit test and the coefficient of determination R^2 and adjusted R^2. Analysis of variance (ANOVA) was performed to determine the statistical significance of each independent variable, their combinations and to exclude insignificant

variables at an alpha level of 0.05. A backward stepwise elimination regression was used to build up the quadratic model for the CF and RE responses. This technique starts with all candidate factors in the model, i.e. the full model, and then removes the least significant variable for each step based on a Significance Level to Stay (SLS) criterion [52]. Factorial plots were also employed to study the effect of significant variables and their combinations on process responses.

After the analysis of experimental data from the harvesting trials based on CCD design, the flotation process factors were optimized to maximize microalgae recovery at a considerable enrichment. Later, *C. vulgaris, I. galbana,* and *T. suecica* were harvested continuously, in replicates of two, based on optimised conditions. Algae cell concentrations in the inlet stream were held at $9.58 \times 10^6 \pm 1.1 \times 10^6$ cells mL^{-1}, $1.01 \times 10^7 \pm 1.29 \times 10^4$ cells mL^{-1} and $1.43 \times 10^6 \pm 7.97 \times 10^4$ cells mL^{-1} for *C. vulgaris*, *I. galbana* and *T. suecica* respectively.

2.10 Power consumption and harvesting economics

Compression of the gas phase in a flotation column is essential for the sparging process. In other words, the gas should be compressed to overcome the pressure drop across the sparger, hydrostatic pressure of the liquid pool, and pressure drop because of friction due to flowing foam with the column wall. Total power consumption in the flotation column can be directly linked to the required work of the air compressor. The power required W_{comp} for an isentropic compression of an ideal gas was calculated using equation 6 [27]:

$$W_{comp} = \frac{RT_o}{\eta_{is}} \frac{\gamma - 1}{\gamma} \left[\left(\frac{P_1}{P_0} \right)^{\frac{\gamma-1}{\gamma}} - 1 \right] \cdots (6)$$

where: W_{comp} is the compressor work (J mol^{-1}); R is the universal gas constant (= 8.314 J mol^{-1} K^{-1}); T_o is the absolute initial temperature (= 298 ºK); η_{is} is the efficiency of air compressor; γ is the ratio of the isobaric to isochoric heat capacities (= 1.4 for dry air); P_0 is the pressure upstream of the compressor; and P_1 is the pressure of the compressed gas. A pressure gauge connected to the gas line was employed to measure the compressed gas pressure. The overall compressor efficiencies are within the range of 65-90% [53]. In this work, 70% air compressor efficiency was assumed.

The unit of power consumption according to equation 6 is in J mol^{-1} of gas whereas the power consumption of most harvesting techniques in the literature are reported in kWh m^{-3} of algae culture. Therefore, for ease of comparison with other techniques, the calculated work value was converted to kWh m^{-3} of algae culture as elucidated later. The associated chemical cost for foam flotation in US$ m^{-3} of algae culture was also calculated based on the chemical costs and chemical dosage required.

3. Results and discussion

3.1 Hydrophobicity tests

The hydrophobicity assay is a simple and rapid procedure to assess surfactant efficacy prior to foam flotation harvesting. The *Chlorella vulgaris* hydrophobicity data using three surfactant types are shown in Fig. 2. *C. vulgaris* was weakly hydrophobic (24%) but the addition of 20 mg L^{-1} of CTAB increased hydrophobicity to 97% ($p =< 0.001$). Most microalgae species are negatively charged at typical culture pH; the ζ-potential of *C. vulgaris* was -18.02 mV at pH 7. Therefore, CTAB adsorbed onto the algae due to electrostatic interactions between the negatively charged cells and the cationic amphiphilic CTAB with the hydrocarbon tail increasing the alga's hydrophobicity. There was no significant difference in hydrophobicity with 40 mg L^{-1} of CTAB. Insignificant hydrophobicity increases were observed with 20 ($p = 0.771$) and 40 ($p = 0.734$) mg L^{-1} of SDS. This was due to repulsive forces between the cell and the anionic amphiphilic SDS. The small increase was probably due to some algae cells becoming trapped in the foam generated during shaking of the sample, causing some cells to move away from the sample suspension. Similarly, small but insignificant rises in hydrophobicity were found after addition of 2 ($p = 0.255$) and 4 ($p = 0.306$) mL of non-ionic TWEEN 20; likely due to the same reasons as for SDS. The addition of 70 and 100 mg L^{-1} of $AlCl_3$ with 40 mg L^{-1} of SDS increased the hydrophobicity to 50% ($p = 0.001$) and 98% ($p =< 0.001$) respectively (Fig. 2). This was due to the charge neutralisation of the algal cells induced by Al^{3+} after dissociation of $AlCl_3$ in water, thus enabling SDS to be adsorbed onto the cell surface and thereby increasing the hydrophobicity. However, the need for additional chemical treatment increases the harvesting cost. As such, only CTAB was carried forward for harvesting trials.

3.2 Adsorption isotherm

Measuring the quantity of surfactant adsorbed onto the algae cells is essential to qualify the electrochemical surfactant adsorption hypothesis and to quantify surfactant adsorbed for further analysis. In froth flotation two chemicals are added to the feed. The first is called a frother which acts to reduce the surface tension of the gas-liquid interface and consequently stabilises the froth. The second is a collector which adsorbs to the particles' surface, enhancing its hydrophobicity [27]. In the foam flotation column, surfactants are used for both purposes, i.e. as a foaming agent since the surfactants tend to adsorb at gas-liquid interfaces and as a collector because the surfactants adsorb onto algae cells due to the electrostatic forces of attraction. Therefore, calculating surfactant use for enhancing hydrophobicity and foam stabilisation is important. The CTAB concentration-surface tension calibration curve with the fitted polynomial model is given in Fig. 3. It can be seen from Table 3 for the algae culture of 1.2 ± 0.01 g L^{-1} that 32.2 ± 0.2% of the added CTAB was retrieved from the supernatant i.e. adsorbed to the gas-liquid interface. It may therefore be inferred that 67.8 ± 0.2% of

the CTAB was adsorbed onto algae cells. When algae biomass density was reduced to 0.68 ± 0.01 g L^{-1}, the percentage of adsorbed CTAB decreased to 39.9 ± 1.3%, predicting a more stable foam.

It is worth noting that the majority of the remaining free CTAB (non-adsorbed onto algae surfaces) that attached to the air bubbles and generate foam are recovered with the harvested microalgae. Despite the amount of CTAB in the discharge stream not being measured in the current work, this inference was made based on observations from second-stage harvesting trials conducted on samples collected from the discharge stream. A very thin layer of foam was noticed after bubbling air through the samples, indicating that only a small amount of surfactant remained unrecovered in the foamate. The small amount of CTAB in the discharge stream can be easily recovered by a very short-term flotation process, consequently, the surfactant-free water can be used for another cultivation cycle.

3.3 Zeta (ζ) potential experiments

The measurements of ζ-potential for *Chlorella* with and without CTAB addition and after resuspension in freshwater are shown in Fig. 4. The average magnitudes of the ζ-potential were negative and within the range of -13.8 to -18.02 at the tested pH. The highest absolute average ζ-potential was -18.02 at pH ≈ 7. The measurements were in line with those conducted previously by Hao *et al.* [54] in which they reported that the absolute average ζ-potential was -16.88 for *C. vulgaris* at pH 7. CTAB showed an obvious capability to reduce the net charge of the algal cells upon the addition of ≈ 35 mg to the algae culture, thereby it perhaps eliminates their stable suspension. For instance, the average ζ-potential at pH 8 reduced from -17.76 to -8.28 mV. The presence of ions in the microalgae culture had a negative effect on ζ-potential as shown in Fig. 4. The average ζ- potential, absolute value, increased when *C. vulgaris* was re-suspended in freshwater, e.g. at pH 8 ζ-potential changed from -17.76 to -24.12 mV. The centrifugation and re-suspension in freshwater resulted in the removal of most positive ions in the BG11 culture medium. For the foam flotation process to recover microalgae successfully at higher RE, the charge difference between the cell and the surfactant should be high. This increases the capability of microalgae to capture surfactant due to the electrostatic attractive forces between them. This observation was also validated by conducting some batch harvesting trials using the foam column on a microalgae culture that was centrifuged and re-suspended in freshwater (data not shown). Wang *et al.* [55] have reported that the surface structure, in addition to extracellular products, are the main factors affecting the net charge of cell surfaces. These factors are directly related to the growth and metabolic level of the algae cells. Therefore, selection of the most suitable culture age in which medium ions are as low as possible is important for an efficient harvesting of microalgae by foam flotation. However, this may increase ash content in the harvested microalgae and thus reduce the biofuel yields.

3.4 Measurements of contact angle

The measured mean contact angles for *C. vulgaris* with and without CTAB are shown in Fig. 5. Due to difficulties in getting an ideal surface because of the size and shape of microalgae cells, the contact angle was measured over an algal mat according to Ozkan and Berberoglu [46]. As seen from the contact angle measurements (Fig. 5), *Chlorella* without any surfactant addition had hydrophilic surfaces (contact angle = 30.17˚). This hydrophilicity was due to the surface functional groups present on the cell walls. *C. vulgaris* are algaenan-free species and their cell wall contains neutral sugars, proteins and uronic acids which have hydrophilic surface functional groups such as hydroxyl, carboxyl, and amine groups [46, 56]. The contact angle value in this work was lower than that reported by Ozkan and Berberoglu (contact angle = 42.7˚) which might be due to differences in the biochemical composition between the algal samples. However, the stabilization time for the probe liquid on the mats was 0.5 sec, a little longer than that adopted in Ozkan and Berberoglu's work (0.2 to 0.3 sec) which might result in a higher contact angle. The low hydrophobicity of *C .vulgaris* increased after addition of 20 mg L^{-1} CTAB as the contact angle increased from 30.17 to 45˚. The increase was likely due to the attachment of long alkyl hydrophobic groups originating from CTAB after dissociation in water. When the CTAB concentration increased to 30 and 40 mg L^{-1}, the contact angles increased to 49.16 and 53.87˚ respectively, indicating that the hydrophilicities reduced due to the additional attachments of hydrophobic alkyl groups (Fig. 5). In contrast to the hydrophobicity test by the adhesion to hydrocarbons method, the contact angle method had more capability to trace the influence of adding more CTAB on microalgae hydrophobicity while no significant increase was observed between 20 and 40 mg L^{-1} CTAB with the former method.

3.5 Analysis of experimental design

3.5.1 Fractional factorial design of experiments

Factorial design of experiments (DOE) is often used as a screening test to differentiate the most powerful effecting factors from those of lesser importance [49]. From the DOE screening trials, higher RE were gained using the fine porous sparger. When the coarse porous sparger was used the CF increased; however, the RE decreased (data not shown). An estimation of the bubble size in the liquid pool was made based on Kutatelabze and Styrikovich's empirical formula, equation 7 [57]:

$$r_b = \left[\frac{\sigma r_o}{g(\rho_f - \rho_g)}\right]^{1/3} \cdots (7)$$

where r_b is the bubble radius; r_o is the sparger mean pore size (30 μm for fine porous and 154 μm for coarse porous); σ is the fluid surface tension; g is the acceleration due to gravity; and ρ_f and ρ_g are the fluid and gas densities respectively. A bubble diameter of 1.02 mm is produced using the fine

porous sparger at a CTAB concentration of 40 mg L^{-1}, versus 1.76 mm with the coarse porous sparger. Smaller bubbles significantly improved the RE ($F = 25.08$, $p=0.001$) but had no significant effect on the CF. The concentration of algae in the foamate increased using the coarse porous sparger. Smaller bubbles provide a larger interfacial area for cell adsorption. They also have a longer residence time within the liquid pool, which increases contact time and adsorption resulting in a higher RE. However, a drawback of smaller bubbles is the formation of a wetter foam due to a greater volume of interstitial liquid (of low algae concentration) trapped between the foam lamellae, combined with slower liquid drainage in the rising foam. Based on the DOE outcomes, the fine sparger was employed in all subsequent response surface experiments.

3.5.2 Response surface design

The design matrix and results obtained for the CCD are presented in Table S1. The CCD data were evaluated to determine the statistical significance of each independent variable and the interactions among variables. The linear effects of all individual factors were significant ($F = 216.18$, $P = < 0.001$; Table S2). In addition, the square effects of surfactant concentration, air flow rate, and column height were also significant. The surfactant concentration had the largest effect on the CF followed sequentially by air flow rate, column height, feed flow rate, surfactant concentration^2, column height^2, liquid pool depth, and air flow rate^2. There were significant interactions between: feed flow rate and surfactant concentration; feed flow rate and air flow rate; feed flow rate and column height; surfactant concentration and air flow rate; surfactant concentration and column height; air flow rate and column height; air flow rate and liquid pool depth; and column height and liquid pool depth (Table S2). Feed flow rate and surfactant concentration had the greatest effect on the CF.

All individual factors had a significant linear effect on RE (Table S3). In addition, the square effect of the liquid pool depth was also significant. Surfactant concentration, column height, air flow rate, feed flow rate, liquid pool depth, and liquid pool depth^2, in that order, most influenced the recovery of algal biomass. There were significant factor interactions between: surfactant concentration and air flow rate; surfactant concentration and column height; and air flow rate and liquid pool depth (Table S3), with the interaction between surfactant concentration and air flow rate or column height having the greatest effect on RE.

The plots of the linear, square and interaction effects of the factors for CF and RE are shown in Figs 6 and 7 respectively. Lower feed rates resulted in lower CF and higher RE (Figs 6A and 7A). This is due to the longer retention time of algae cells in the effervescent liquid which provides more contact time between bubbles and algae. As the feed flow rate increased, the CF increased and the RE decreased. According to the adsorption isotherm models for surface active materials such as the Langmuir isotherm model, it is clear that the surface excess, i.e. surface concentration, increases when

the surfactant concentration in the bulk liquid increases [58]. Similarly, when the feed flow rate increases, the concentration of algae and free surfactant increases in the liquid pool at the base of the foam column, i.e. the concentration is slowly depleted and the surface concentration is correspondingly high. However, both microalgae and un-adsorbed surfactant concentrations in the liquid pool increase when the feed flow rate is increased. Consequently, the latter has an effect on process responses similar to that of surfactant concentration in the feed stream and leads to a decrease in the influence of feed flow rate.

CTAB concentration affects the CF in a negative way while it affects the RE in a positive way (Figs 6A and 7A). Thus, lower CF and higher RE were obtained at higher CTAB concentrations. The surface tension of the effervescent liquid reduces when the concentration of surface-active materials increases. This causes a reduction in bubble size leading to a wetter foam [18, 27, 59]. Therefore, a wetter foam results in a lower CF and a higher RE.

Air flow rate negatively affected the CF but improved the RE. Thus, at higher air flow rates lower CF and higher RE were observed. The amount of bubble surface available in a flotation column is crucial in collecting microalgae cells. The effect of air flow rate can be investigated by calculating of bubble surface area flux (S_b) rather than gas hold-up. Bubble surface area flux can be evaluated from the bubble flow rate (n_b), the mean or Sauter mean bubble diameter (d_b), and the column cross sectional area (A_c) as shown in equation 8 [60], where J_g is the superficial gas velocity.

$$S_b = \frac{n_b \pi d_b^2}{A_c} = \frac{6.J_g}{d_b} \quad \ldots (8)$$

Increasing the air flow rate will increase the bubble surface area flux resulting in higher RE. Furthermore, Stevenson and Li [61] stated that in a porous medium the generated bubble size decreases with increasing gas flow rate. At lower gas rates, only bigger pores are active and generating mainly big bubbles. When the gas flow rate increases, most of the inactive small pores become active, leading to an increased number of smaller bubbles [62], and thus a wetter foam. Saleh et al. [63] stated that, in a foam fractionation column, increasing the volume of a wet foam with the gas flow rate was due to the short residence time for the rising foam to drain the liquid, resulting in a decrease in enrichment and an increase in RE [64]. This may partially explain the decreasing CF and increasing RE of the harvested algae.

The effect of column height was comparable to that of the feed flow rate. An increasing column height positively influenced the CF but at the expense of the RE (Figs 6A and 7A). The fraction of interstitial liquid trapped between the foam lamellae was negatively related to the column height. This is due to the change in bubble size distribution in the zone beyond that where capillary forces become dominant

[65, 66]. Also, the foam carrying microalgae dries as it rises up the column, consequently, microalgae cells stick on the column wall at the top resulting in a reduction in the RE. This was observed clearly through the harvesting trials especially when low CTAB concentration and air flow rate were used.

An increasing liquid pool depth had a negative effect on the CF but increased the RE. This was due to the longer retention time of algae cells and hence a longer contact time. A deeper liquid pool also increased the gas residence time at the same bubble rise velocity i.e. more time for bubbles to adsorb cells.

The contour plot for significant interactions affecting the CF is shown in Fig. 8 in which any two factors change within the design range while the other three factors are kept constant at their centre values. This reinforces the importance of the interaction between surfactant concentration and the feed flow rate. CF in the range of 250 to 300 can be achieved by combining a high feed flow rate with a low surfactant concentration. Similarly, higher CF were gained due to the interaction between the surfactant concentration with air flow rate and surfactant concentration with column height (Figs 8D and E). CF between 150 and 200 can be achieved by combining a high feed flow rate with a low air flow rate and/or high column height (Figs 8B and C). Thus, increasing feed flow rate can counteract the negative effects of the high surfactant concentration and air flow rate on the CF response.

The quadratic model (equation 9) for CF was significant (p = >0.05; Table S1). The lack-of-fit compares the residual error to the pure error that was obtained from the six replicate runs at the centre points. In addition, high R^2 and R^2_{adj} values were achieved for the fitted model, 98.11 and 97.14% respectively i.e. the model can explain more than 98% of the total variability in the data.

$$CF = 442.1 + 387.3F - 9.83S - 142.4A - 1.07H - 7.83D + 0.13S^2 + 17.55A^2 + 0.02H^2 + 0.08D^2 - 10.03FS - 96.2FA + 2.1FH + 2.45FD + 3.61SA - 0.06SH - 0.41AH - 1.83AD + 0.06HD \quad \cdots (9)$$

Where: F is the feed flow rate; S is the surfactant concentration; A is the air flow rate; H is the column height; and D is the effervescent liquid depth.

The RE interaction plots (Figs 7B and 7B) revealed that RE of over 90% can be achieved by combining high surfactant concentration and high air flow rate, due to smaller bubbles produced when the inlet surfactant concentration increases resulting in a high specific surface area and a longer time for adsorption. On the other hand, increasing column height counteracts the positive effect of the high surfactant concentration (Figs 7B and 7A) due to the increased residence time and corresponding interstitial liquid drainage opportunities that a taller column provides.

The regression model (equation 10) was significant (p = 0.412), explaining up to 90% of the total variability in the data.

$$RE = -50 - 29.07F + 2.21S - 6.2A + 0.47H + 044D + 0.098D^2 + 1.04SA - 0.02SH - 1.52AD \quad \cdots (10)$$

Where: F is the feed flow rate; S is the surfactant concentration; A is the air flow rate; H is the column height; and D is the effervescent liquid depth.

3.6 Harvesting of freshwater and marine microalgae based on the optimised flotation factors

The outcomes from the CCD design demonstrated that CTAB concentration, air flow rate, and column height had the strongest effects on biomass recovery. However, using a high CTAB concentration and a high air flow rate does not favor high CF. Instead, prolonging the contact time for adsorption by increasing liquid pool depth and reducing feed flow rate with a moderate CTAB concentration and air flow rate is more desirable to achieve a good combination between recovery and enrichment of microalgae biomass. The factors from the CCD design were optimized by the response optimizer to achieve the above objective. The optimum values were CTAB = 35 mg L^{-1}, air flow rate = 1 L min^{-1}, feed flow rate = 0.1 L min^{-1}, column height = 146 cm, and liquid pool depth = 25 cm. *C. vulgaris*, *I. galbana,* and *T. suecica* were then harvested continuously based on the above optimum values. Results for RE and the CF are shown in Fig. 10.

The results showed an excellent RE of 95% and a final biomass 173-times more concentrated than the initial *C. vulgaris* culture. For marine microalgae, RE of 93% and 89% at 271 and 143 enrichment factors were obtained for *I. galbana* and *T. suecica* respectively. Even though the CF for all harvested species differed, attaining close RE for both freshwater and marine microalgae increases the potential of foam flotation becoming a growth media independent harvester as opposed to coagulation and flocculation processes where high amounts of coagulants and flocculants are required for harvesting marine microalgae due to the ionic strength of saltwater. More stable foam was also noticed through the harvesting trials of the marine microalgae which is probably due to the ions in the saltwater.

Very close separation efficiencies of *C. vulgaris* were observed in both the current work and that conducted by Kurniawati *et al.* [38]. They were able to achieve a separation efficiency of 93% using a foam flotation column operated in batch mode with a natural saponin surfactant and chitosan flocculants together. Whilst their work has the advantage of using natural biochemicals to harvest microalgae, the need for additional chemical treatment increases the harvesting cost. In comparison to the batch flotation harvesting trials of *C. vulgaris* conducted by Liu *et al.* [67], a lower RE was gained in their work (90%) which was probably due to the lower air flow rate (0.114 L min^{-1}) even though higher CTAB concentration (40 mg L^{-1}) was used. The flotation RE obtained in this work for *Chlorella* and *Tetraselmis* were close to those obtained previously by Garg *et al.* [37] even though the differences between both experimental trials include surfactant types and dosage, the flotation

apparatus type, and the operating mode. They used mechanical flotation cells with the addition of two surfactant types (tetradecyl trimethylammonium bromide, C_{14}TAB and dodecylammonium hydrochloride, DAH). However, the enrichments gained herein for both species were many-folds higher than those obtained by the Garg group. This was probably due to the significant interplay between the process factors, as well as the effect of column height as the foam carrying microalgae dries as it rises up the column. This presents another advantage to column flotation besides the simplicity of construction and low energy consumption. In comparison to other flotation harvesting trials, the percentage recovery obtained in this work for *C. vulgaris* (95%) was similar to that obtained by Henderson *et al.* [68] (94.8%). However, they used dissolved air flotation (DAF) in a batch mode (10 min) with aluminium sulphate as a coagulant to harvest a culture of *C. vulgaris* of cell density of $5 \times 10^5 \pm 5 \times 10^4$ cells ml^{-1}. Prior to their work above, Henderson *et al.* [34] conducted harvesting trials also using DAF working in a batch mode but with different types of cationic and anionic surfactants instead of coagulants. The maximum removal efficiency of *C. vulgaris* obtained in their work (54%) was substantially lower than that obtained by the current work. This reduction in the percentage recovery was probably due to the addition of surfactants to the saturator rather than the microalgae culture which has advantages of reducing the bubble size and altering the bubble charge but it did not enhance the hydrophobicity of microalgae or compensate the absence of the coagulant role on increasing the cell size due to aggregation. With the exception of Garg *et al.'s* work, neither the CF nor the harvesting economics were reported in the other studies since their trials were performed for wastewater treatment rather than producing biomass for biofuel production.

On the other side, CTAB, like ozone, has the ability to disrupt the algae cell wall and promote *in situ* cell lysis. Coward et al. [69] have observed that the presence of CTAB in the harvested microalgae enhanced lipid recovery and profile as well as increased the solubility of some phospholipids in the cell membrane. The disruption of the algal cell wall and the enhancement in lipid recovery and profile due to the existence of the CTAB surfactant with the harvested microalgae offers additional advantages to the flotation technique to drive down the cost of processing and produce biomass which is more advantageous for liquid hydrocarbon biofuels.

3.7 Power consumption and harvesting economics

Selecting the optimal harvesting technique relies on the relationship between the efficiency of algal biomass recovery and the operational energy requirements. The inconsistency between harvesting efficiency and energy consumption is often the major drawback in most harvesting techniques. The power consumption associated with bubble generation was calculated based on the pressure of the compressed air through the sparger plus other operating conditions. The compressor work W_{comp} (J mol^{-1}) was calculated according to equation 6 after measuring the compressed gas pressure (P_1) using

the pressure gauge as shown in Table 4. Other work values were determined after converting joule to kilowatt-hour and calculating the number of moles to volume ratio of the gas using the ideal gas law (equation 11) at the conditions (T_o, P_1) in Table 4. Only one calculated value was reported herein even though all compressor works were calculated for both sparger types, liquid pool depths, and air flow rates.

$$\frac{n}{v} = \frac{p}{RT} \quad \ldots (11)$$

The power consumptions of most harvesting techniques in the literature were reported in units of kWh m^{-3} of algae culture. This can be determined if the calculated work value (kWh m^{-3} of gas) is multiplied by the ratio of the volumetric flow rate of the gas inlet to the volumetric flow rate of the medium inlet (feed) in the flotation process. The optimum values of air flow rate and feed flow rate used to harvest the three microalgae species were 1 L min^{-1} and 0.1 L min^{-1} (0.001 and 0.0001 m^3 min^{-1}) respectively, thereby, the ratio of the volumetric flow rate of gas to the volumetric flow rate of microalgae feed was 10. Energy consumption, RE, and CF for different harvesting techniques used in various operational modes are presented in Table 5. The continuous foam flotation column had a very low power consumption relative to all other techniques except for suspended air flotation. With regards to the CF, Table 5 demonstrates that the CF attained by the optimised foam column outperforms those achieved by other harvesting methods at a high RE.

The calculations of the total cost of the foam flotation column including compressor work and chemicals to harvest 1 m^3 of microalgae culture were also performed as shown in Table 4. The continuous foam flotation (this work) had a low total harvesting cost of US$ 0.179 in comparison to that calculated by Coward *et al.* [70] (US$ 0.915) to harvest the same volume of microalgae by dissolved air flotation using ferric chloride flocculants.

4. Conclusion

In foam flotation, collectors (surfactants) are important to enhance the hydrophobicity of microalgae and create a metastable foam yielding high recovery efficiencies and biomass enrichment (concentration factor). The measurements of the surface characteristics of *C. vulgaris* demonstrated that this species has an electronegative and hydrophilic surface. CTAB was found as the most appropriate surfactant due to the electrostatic interaction between it and the electronegative microalgae. Moreover, CTAB was able to reduce the net charge as well as the hydrophilicity of *C. vulgaris*, resulting in better harvesting performances. This was due to the attachment of the positive long hydrophobic alkyl group originating from CTAB after dissociation in water. The harvesting trials demonstrated that the continuous foam flotation process operated at the optimised factors

yielded RE of 95, 93, and 89% together with 173, 271 and 143-fold biomass enrichments for freshwater *Chlorella vulgaris* and marine *Isochrysis galbana* and *Tetraselmis suecica* microalgae respectively. However, the little reduction in the RE of the marine species was likely due to the salinity of saltwater or to some extent, the surface physicochemical properties of these species. Generally within the flotation process, there is a trade-off between attaining a high RE and a high CF [33]; however, the current continuous process has circumvented that particular compromise, representing a significant advance in foam flotation harvesting of microalgae biomass. What is more, our continuous foam flotation column demonstrated a very low power consumption, 0.052 KWh m^{-3}, with a low total harvesting cost (including the chemical cost) of US$ 0.179 per 1 m^3 of microalgae. Our findings confirm that foam flotation is a very promising approach for the continuous bulk harvesting of microalgae biomass, whether it be for high-value fine chemicals or low-value biofuels. Indeed, the continuous harvesting approach may be especially relevant for the wastewater industry wherein microalgae are used as nutrient scrubbers, or in environmental management and remediation, e.g. the removal of harmful or toxic microalgae blooms from waterways, including municipal water supplies.

Figure legends

Fig 1. Schematic diagram of the continuous foam flotation column. A: Foam collecting cup, B: column tubular module (25, 30 or 50 cm) in height and 5.1 cm in diameter, C: inlet stream, D: inlet flow meter, E: outlet stream valve, F: underflow stream, G: air sparger, H: air input stream.

Fig 2. The hydrophobicity (%) of *Chlorella vulgaris* with and without added surfactants (CTAB, SDS and TWEEN® 20). $AlCl_3$ was added to two further SDS treatments to modify the surface charge of the algae cells. Means ± standard error, n = 2.

Fig. 3. The relationship between CTAB concentration and surface tension, showing the calibration curve with the fitted polynomial model. Means ± standard error.

Fig. 4. Zeta potential (ζ) of *Chlorella vulgaris* at different pH. Means ± standard error.

Fig. 5. Contact angle (˚) of *Chlorella vulgaris* at different CTAB concentrations. Means ± standard error.

Fig. 6. The main effects (A) and interaction plots (B) for the mean of concentration factor (CF) (α = 0.05). Where (a) is the feed flow rate, (b) is the surfactant concentration, (c) is the air flow rate, (d) is the column height, and (e) is the liquid pool depth.

Fig. 7. The main effects (A) and interaction plots (B) for the mean of the recovery efficiency (RE) (α = 0.05). Where (a) is the feed flow rate, (b) is the surfactant concentration, (c) is the air flow rate, (d) is the column height, and (e) is the liquid pool depth.

Fig. 8. Contour plots for the significantly interacting factors in the quadratic model for concentration factor (CF). Hold values: feed flow rate = 0.4 L min^{-1}, surfactant concentration = 40 mg L^{-1}, air flow rate = 1.5 L min^{-1}, column height = 96 cm, liquid pool depth = 13.5 cm.

Fig. 9. Surface plots for the significantly interacting factors in the quadratic model for recovery efficiency (RE). Hold values: feed flow rate = 0.4 L min^{-1}, surfactant concentration = 40 mg L^{-1}, air flow rate = 1.0 L min^{-1}, column height = 110 cm, liquid pool depth = 13.5 cm.

Fig. 10. The recovery efficiency (RE) and the concentration factor (CF) plots for *Chlorella vulgaris*, *Isochrysis galbana,* and *Tetraselmis suecica* based on the optimum conditions for the process factors. Means ± standard error.

Figures

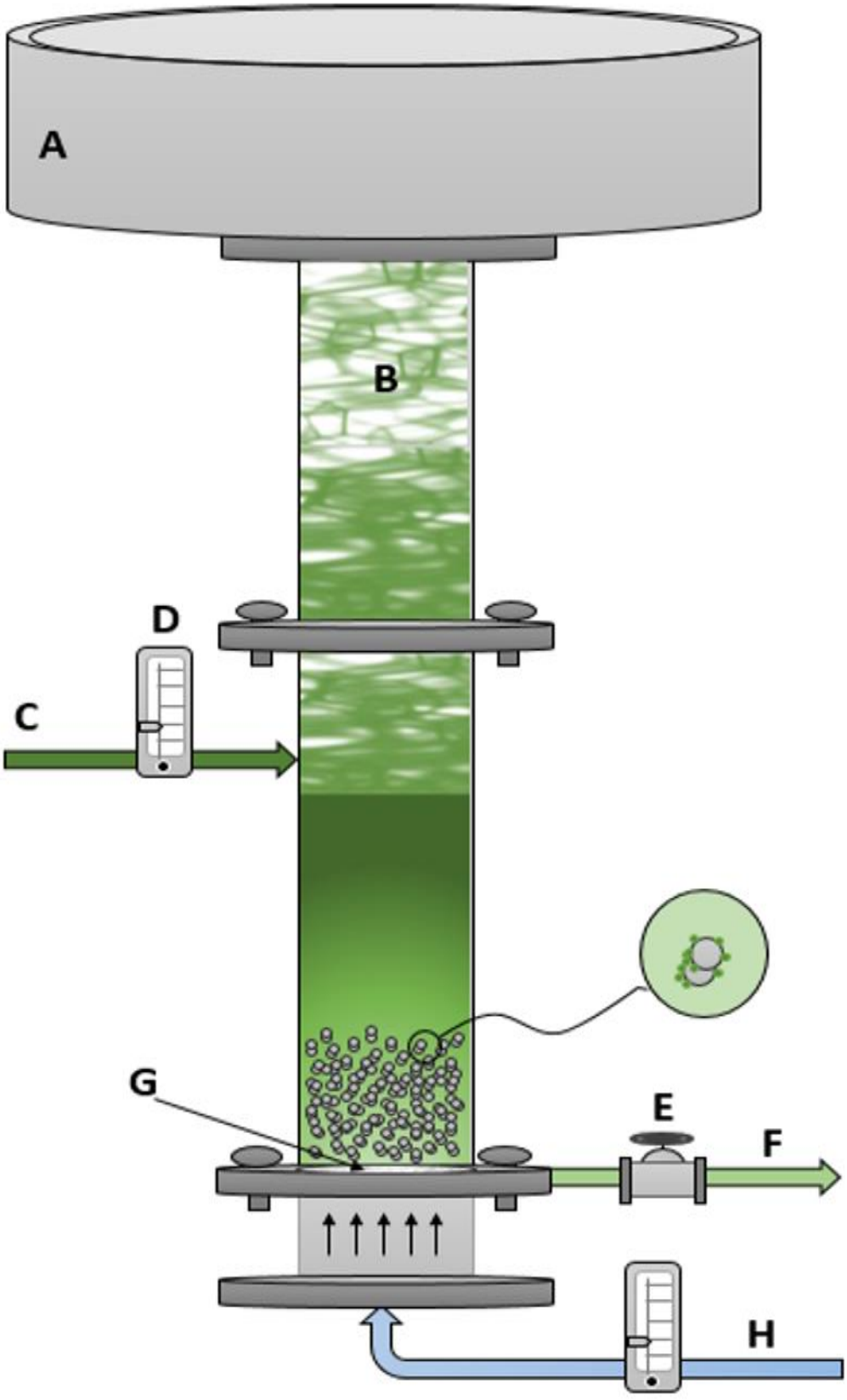

Fig. 1

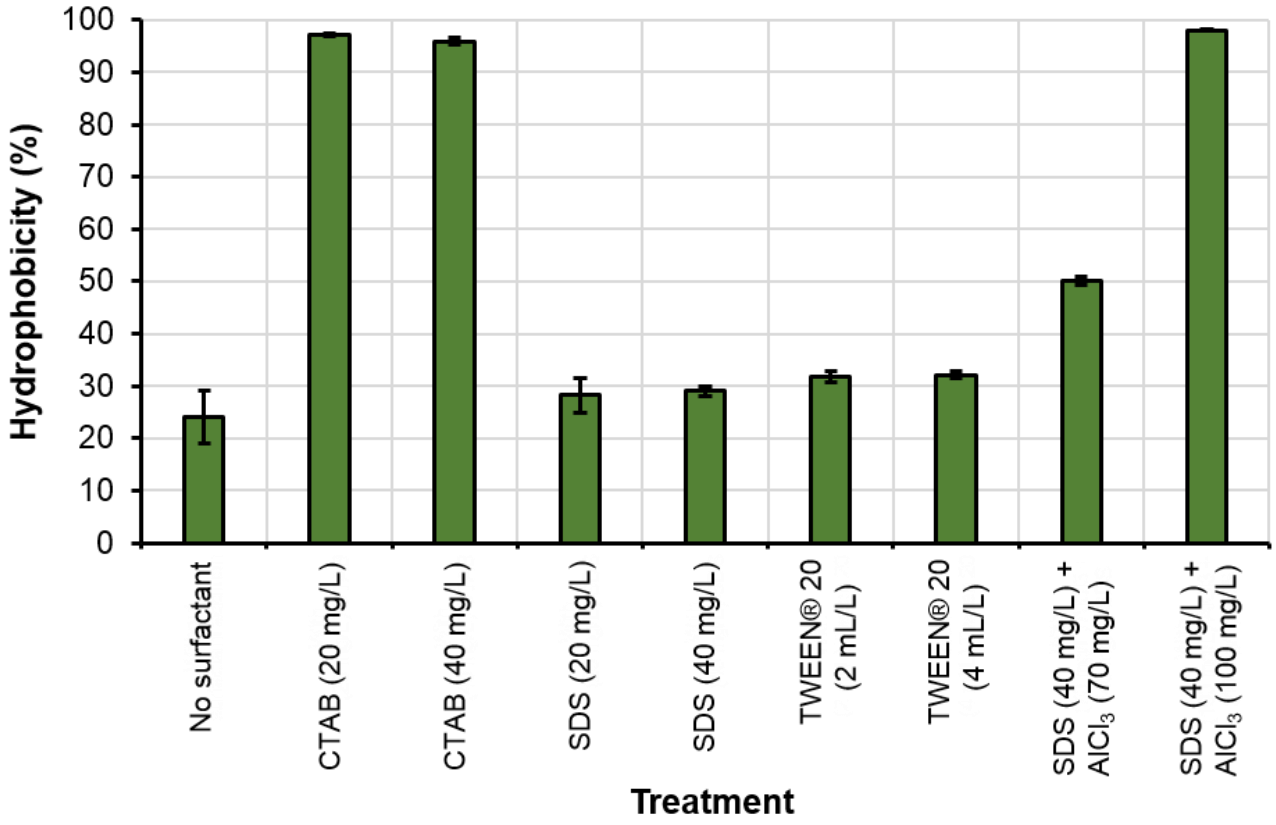
Hydrophobicity (%)
100
90
80
70
60
50
40
30
20
10
0
No surfactant
CTAB (20 mg/L)
CTAB (40 mg/L)
SDS (20 mg/L)
SDS (40 mg/L)
TWEEN® 20 (2 mL/L)
TWEEN® 20 (4 mL/L)
SDS (40 mg/L) + AlCl3 (70 mg/L)
SDS (40 mg/L) + AlCl3 (100 mg/L)
Treatment

Fig. 2

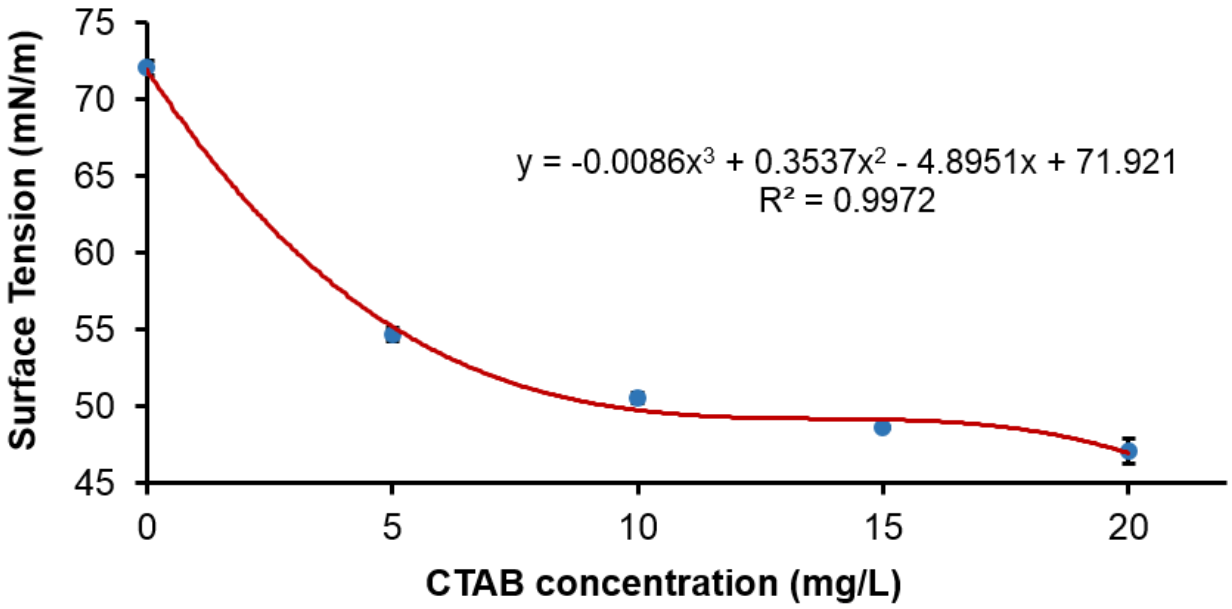
Surface Tension (mN/m)
75
70
65
60
55
50
45
0
5
10
15
20
CTAB concentration (mg/L)
y = -0.0086x³ + 0.3537x² - 4.8951x + 71.921
R² = 0.9972

Fig. 3

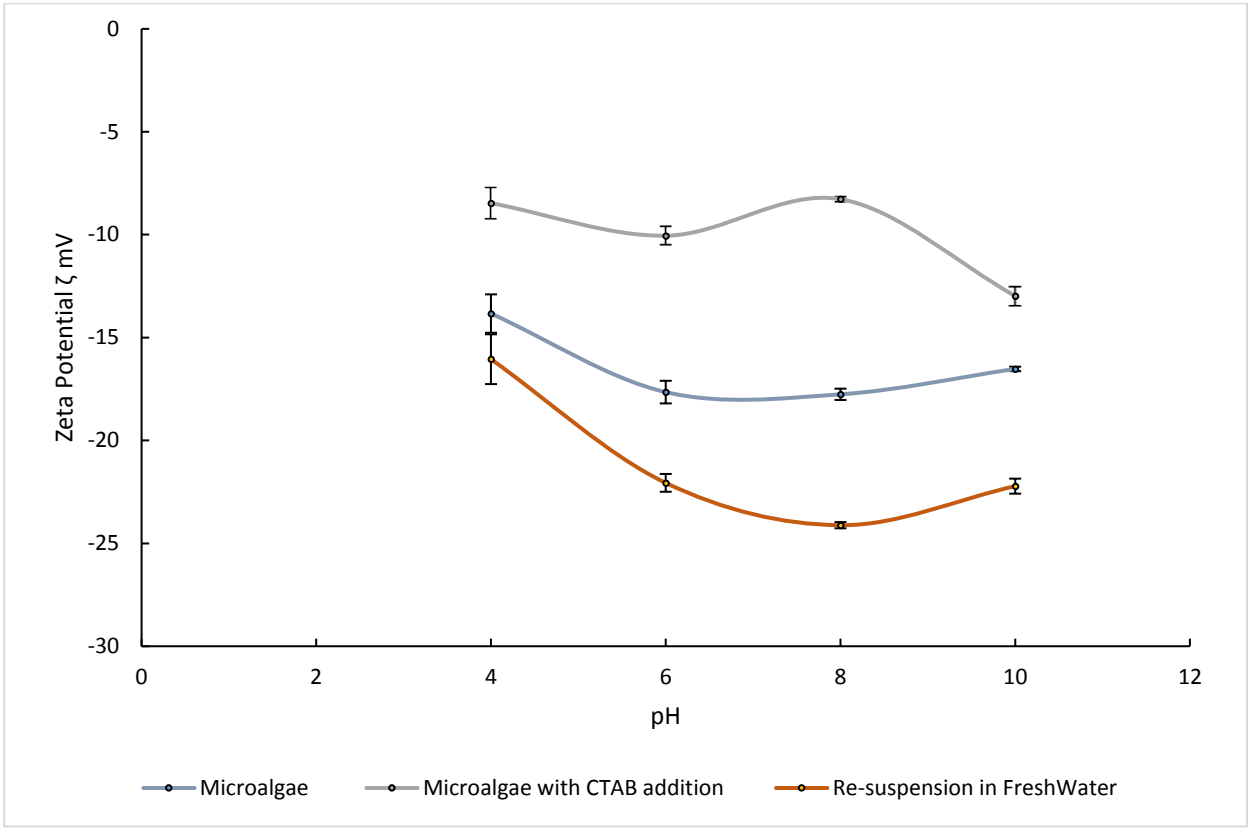
0
-5
-10
-15
-20
-25
-30
Zeta Potential ζ mV
0
2
4
6
8
10
12
pH
Microalgae
Microalgae with CTAB addition
Re-suspension in FreshWater

Fig. 4

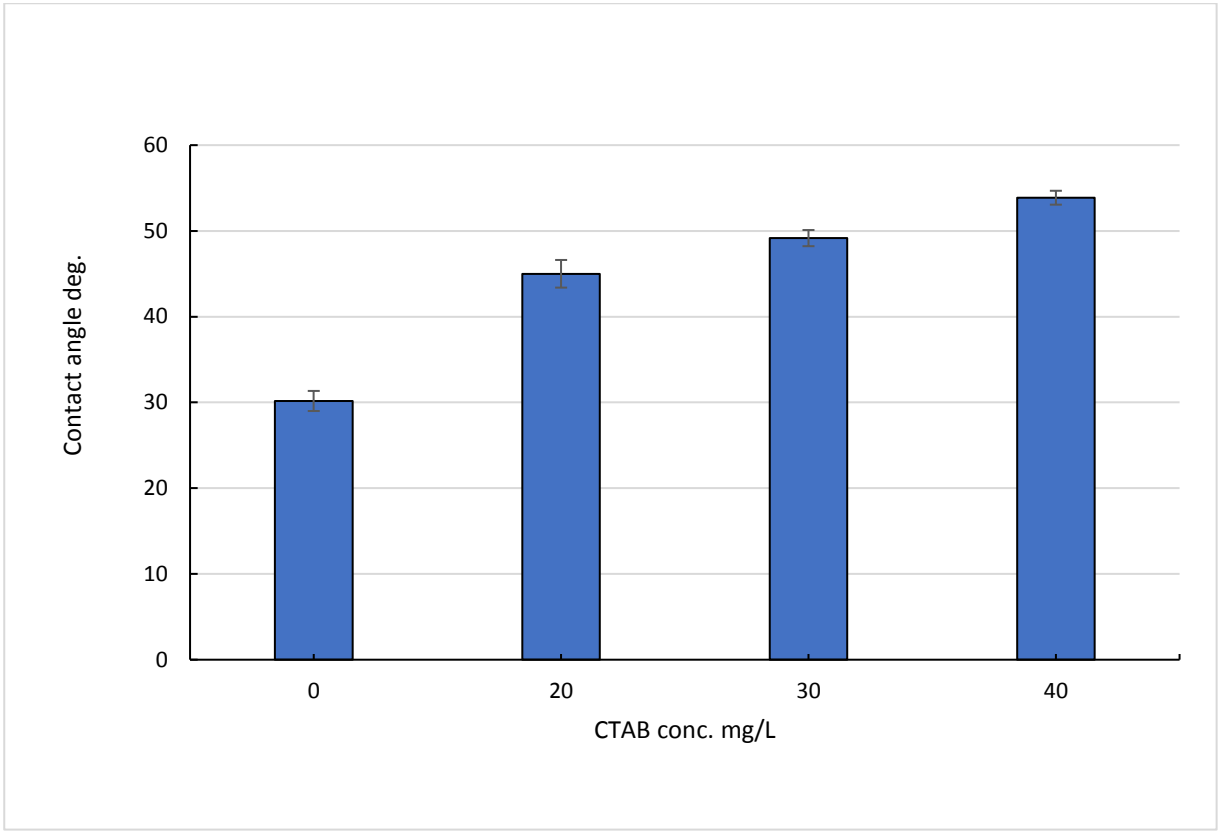

Fig. 5

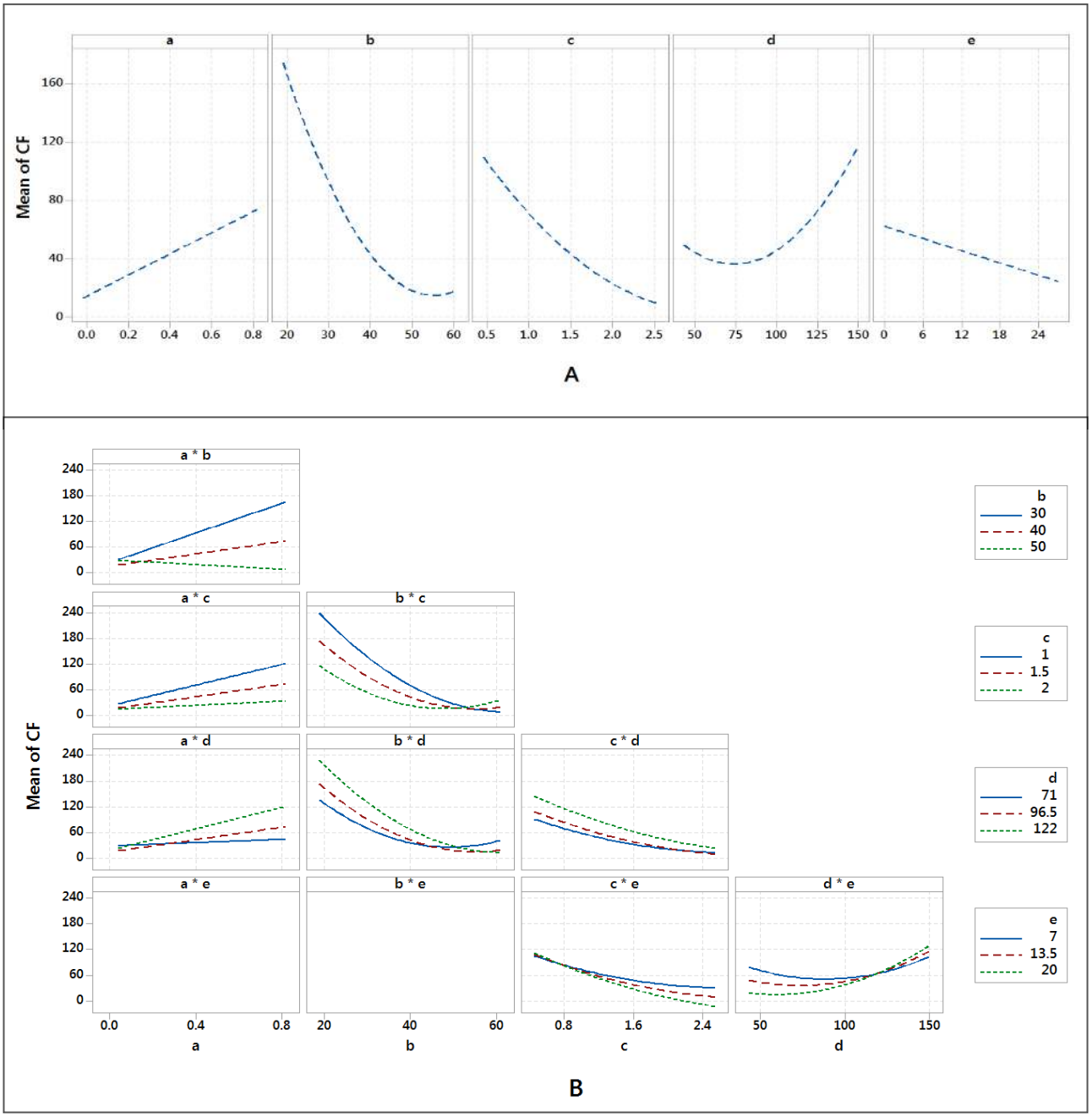
a
b
c
d
e
Mean of CF
160
120
80
40
0
0.0 0.2 0.4 0.6 0.8
20 30 40 50 60
0.5 1.0 1.5 2.0 2.5
50 75 100 125 150
0 6 12 18 24
A
a * b
a * c
b * c
a * d
b * d
c * d
a * e
b * e
c * e
d * e
240
180
120
60
0
b
30
40
50
c
1
1.5
2
d
71
96.5
122
e
7
13.5
20
0.0 0.4 0.8
20 40 60
0.8 1.6 2.4
50 100 150
a
b
c
d
B

Fig. 6

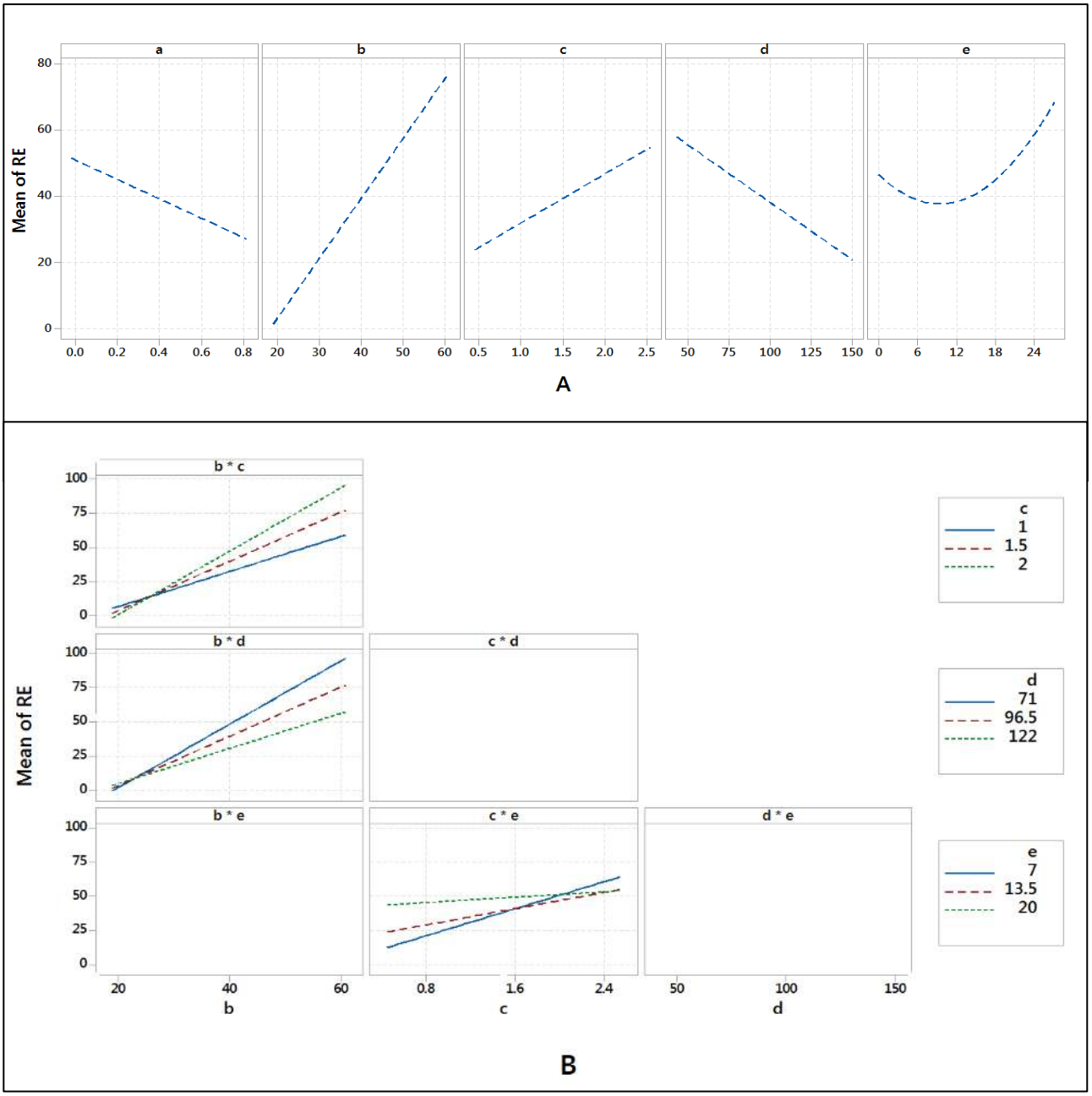
a
b
c
d
e
Mean of RE
A
b * c
b * d
c * d
b * e
c * e
d * e
c
1
1.5
2
d
71
96.5
122
e
7
13.5
20
b
c
d
B

Fig. 7

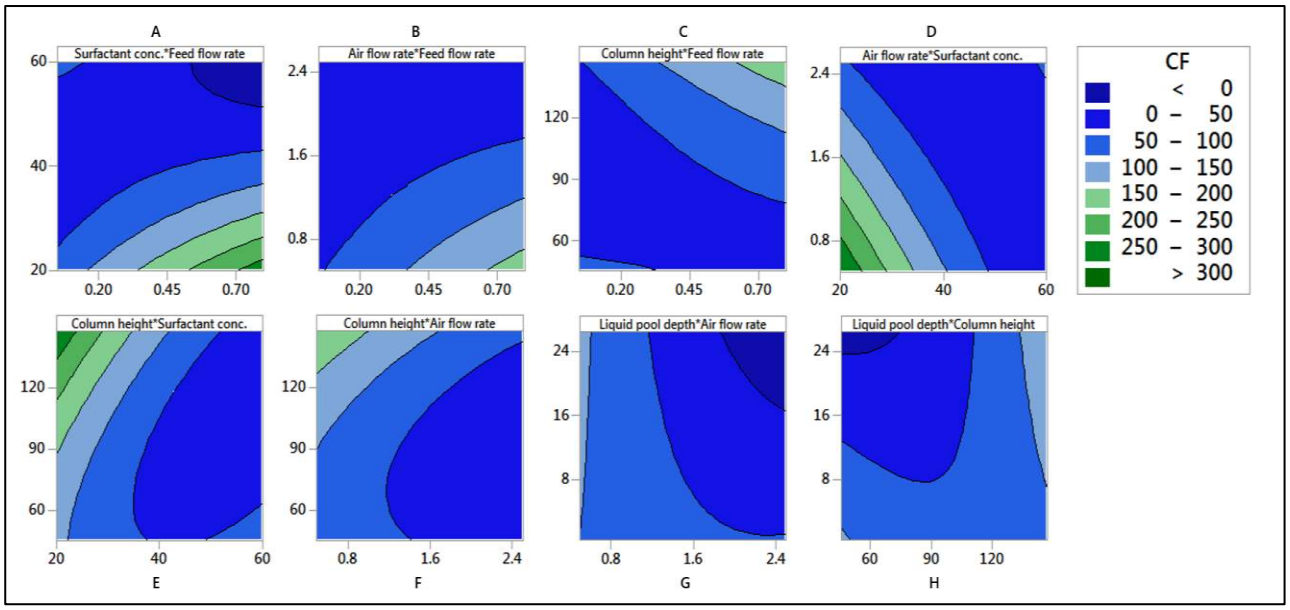
A
Surfactant conc.*Feed flow rate
60
40
20
0.20
0.45
0.70
B
Air flow rate*Feed flow rate
2.4
1.6
0.8
0.20
0.45
0.70
C
Column height*Feed flow rate
120
90
60
0.20
0.45
0.70
D
Air flow rate*Surfactant conc.
2.4
1.6
0.8
20
40
60
CF
< 0
0 – 50
50 – 100
100 – 150
150 – 200
200 – 250
250 – 300
> 300
Column height*Surfactant conc.
120
90
60
20
40
60
E
Column height*Air flow rate
120
90
60
0.8
1.6
2.4
F
Liquid pool depth*Air flow rate
24
16
8
0.8
1.6
2.4
G
Liquid pool depth*Column height
24
16
8
60
90
120
H

Fig. 8

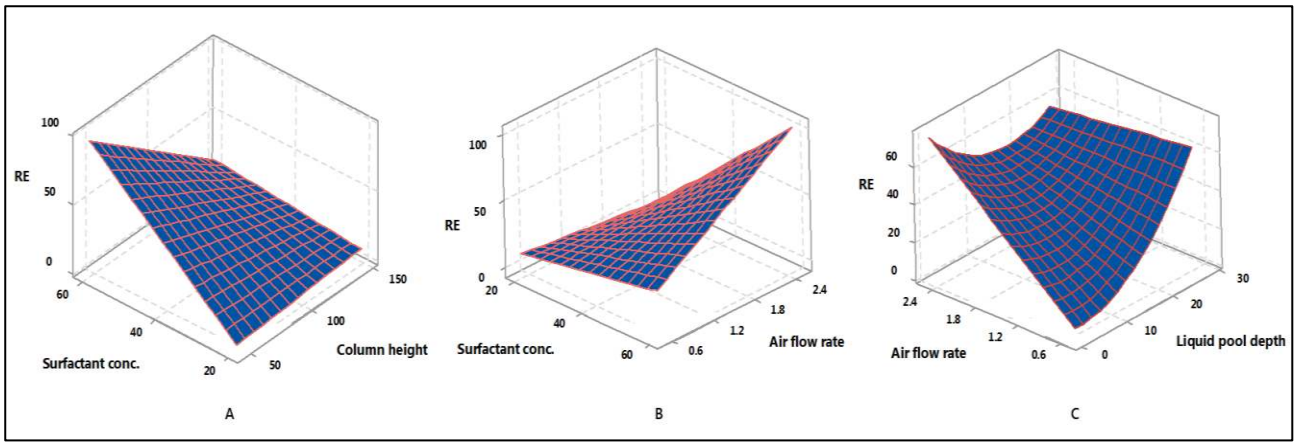
RE
100
50
0
60
40
20
Surfactant conc.
50
100
150
Column height
A
RE
100
50
0
20
40
60
Surfactant conc.
0.6
1.2
1.8
2.4
Air flow rate
B
RE
60
40
20
0
2.4
1.8
1.2
0.6
Air flow rate
0
10
20
30
Liquid pool depth
C

Fig. 9

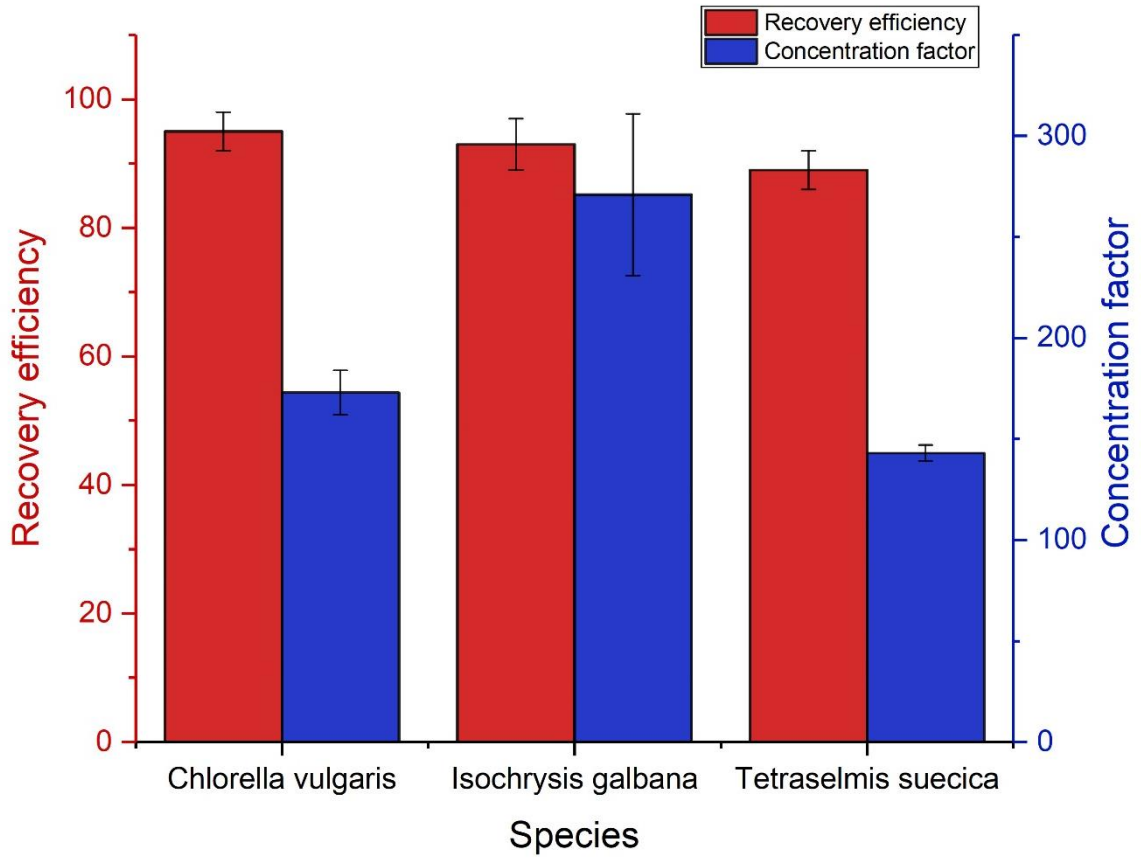
Recovery efficiency
Concentration factor
Recovery efficiency
Concentration factor
100
80
60
40
20
0
300
200
100
0
Chlorella vulgaris
Isochrysis galbana
Tetraselmis suecica
Species

Fig. 10

Table 1. Values of the independent variables for the fractional factorial design.

Independent variables	Levels	
	-1	+1
Surfactant concentration (mg L^{-1})	30	50
Air flow rate (L min^{-1})	1	2
Column height (cm)	71	122
Inlet flow rate (L min^{-1})	0.2	0.6
Liquid pool depth (cm)	7	20
Sparger type	coarse porous	fine porous

Table 2. Values of the independent variables for the central composite design.

Independent variables	Levels				
	-2	-1	0	+1	+2
Surfactant concentration (mg L^{-1})	20	30	40	50	60
Air flow rate (L min^{-1})	0.5	1	1.5	2	2.5
Column height (cm)	46	71	96	122	146
Inlet flow rate (L min^{-1})	0.05	0.2	0.4	0.6	0.8
Liquid pool depth (cm)	0.5	7	13.5	20	26.5

Table 3. Percentage adsorption of CTAB onto algae cells. Means ± standard error

Algae culture	Cells mL^{-1}	g L^{-1}	Sample	Surface tension (mM m^{-1})	Mean surface tension (mN m^{-1})	CTAB % in supernatant	CTAB % adsorbed to algae
1	$24.1 \times 10^6 \pm 2.6 \times 10^4$	1.2 ± 0.01	1 2	52.51 52.44	52.48 ± 0.05	32.2 ± 0.2	67.8 ± 0.2
2	$14.2 \times 10^6 \pm 2.2 \times 10^4$	0.68 ± 0.01	1 2	48.03 47.89	47.96 ± 0.1	60.1 ± 1.3	39.9 ± 1.3

Table 4. The compressor work W_{comp} and the predicated cost of harvesting 1 m^{-3} of algae culture

Condition	R J/mole.K	T_o K	η_{is}	γ (air)	P_1 Kpa	P_0 Kpa	W_{comp} J/mole of gas	W_{comp} kWh/mole of gas	W_{comp} kWh/m^3 of gas
Fine porous sparger, 1 L min^{-1}, air flow rate, and 25 cm liquid pool depth	8.314	293.15	0.7	1.4	113.4	101.3	399.27	$1.11*10^{-4}$	$5.16*10^{-3}$

Condition	W_{comp} kWh/m^3 of algae	Energy cost US$ per kWh	Chemical cost US$ kg^{-1}	Chemical additive g m^{-3}	Chemical cost US$ m^{-3}	Total cost US$ (to harvest 1 m^3 of microalgae)
Fine porous sparger, 1 L min^{-1}, air flow rate, and 25 cm liquid pool depth	0.052[a]	0.004[b]	5[c]	35	0.175	0.179

[a] The value was calculated based on the compressor work kWh per m^3 of gas and the ratio of the inlet gas flow rate and feed flow rate in foam flotation process

[b] Energy cost was calculated from the data prepared by U.S. Deparment of energy based on average price of electricty to the US industrial sector as of November 2017-US$ 0.0679 per kWh [71]

[C] Based on a bulk price of US$ (1-5) per kg with a min. order of 1 metric ton (www.alibaba.com)

Table 5. Energy consumption, concentration factor (CF), and recovery efficiency (RE) of different microalgae harvesting techniques. Where reported, the recovery efficiency (RE %) is given in parentheses.

Harvest method	Operational mode	Microalgae	Energy consumption (KWh m^{-3})	CF and RE
Chamber filter [13]	Discontinuous	*Coelastrum proboscideum*	0.88	245
Vacuum filter; non-pre-coat vacuum drum filter [13]	Continuous	*C. proboscideum*	5.9	180
Vacuum filter; suction filter [13]	Discontinuous	*C. proboscideum*	0.1	80
Tangential flow filtration [72]	Continuous	Multi-strain *Tetraselmis suecica/ Chlorococcum* sp.	0.38	48
Vibrating screens [15]	N/A	N/A	0.4	15-60
Nozzle discharge centrifuge [13]	Continuous	*Scenedesmus*, *C. proboscideum*	0.9	20-150
Decanter bowl centrifuge [13]	Continuous	*Scenedesmus*, *C. proboscideum*	8	11
Hydro-cyclone [13]	Continuous	*C. proboscideum*	0.3	4
Electrolytic flocculation	Batch	Multi-strain algae/ diatoms	0.33	N/A
Electrocoagulation [73]	Batch, 15 min, 10V	*Tetraselmis*	2.75	N/A
Sedimentation Lamella separators [15, 74]	Discontinuous	Multi-strain *Chlorella/ Coelastrum*	0.1	16
Dissolved air flotation [75]	Batch	Multi-strain *Chlorella/ Scenedesmus*	7.6	N/A (85)
Suspended air flotation [75]	Batch		0.003	N/A (77)
Electro-flotation [74]	Batch	Multi-strain *Chlorella/ Coelastrum*	Very high, N/A	N/A
Foam flotation by Jameson cell [76]	N/A	*Tetraselmis* sp. *M8*	N/A	23 (99)
Foam flotation (this study) based on optimised factors	Continuous	*Chlorella vulgaris* *Isochrysis galbana* *Tetraselmis suecica*	0.052	173 (95) 271(93) 143(89)

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