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# Vibrating membrane filtration as improved technology for microalgae dewatering

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## HIGHLIGHTS

- Microalgae membrane filtration is highly improved with vibratory technology.
- Fouling is notably reduced by using shear enhancement.
- Tests at laboratory and pilot scale have been performed.
- Several commercial polymeric membranes and two microalgae species were tested.
- Dynamic membrane filtration reduces cost compared with conventional one.

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## ABSTRACT

The effect of shear-enhanced filtration by vibratory process in microalgae dewatering is presented in this paper. The aim of this research was to investigate the technical performance and improvement of vibrating membrane filtration compared with conventional tangential cross-flow filtration in microalgae concentration.

An industrial-scale available commercial set-up was used. Several membrane materials as polyethersulfone, polyacrylonitrile, etc., and mean pore sizes (from 7000 Da to 0.2 μm) were tested and compared in both filtration set-ups. Experiments were carried-out with *Nannochloropsis gaditana* and *Phaeodactylum tricornutum* microalgae.

It has been demonstrated that, even if the choice of the membrane depends on its cut-off, its material and the type of microalgae filtrated, dynamic filtration is always the best technology over a conventional one. If with conventional filtration permeability values were in the vicinity of 10 L/h/m$^2$/bar in steady state phase, with dynamic filtration these values increased to 30 L/h/m$^2$/bar or more.

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## 1. Introduction

The interest for microalgae lies in their potential utilization in several fields. First, studies were addressed to mariculture feed, fine chemicals and health food industries (omega 3 oil, chlorophyll, livestock feed). Later, the interest for this raw material started also in pharmaceutical and nutraceutical industries, in agriculture as biofertilizer, as bioremediation of water pollution and, finally, as a power source to obtain different kinds of products as biomass to produce hydrogen, biomethane, bioethanol and biodiesel (Harun et al., 2010; Kim et al., 2013a; Mata et al., 2010; Rawat et al., 2011). Nowadays, to allow the process being economically feasible, all microalgae metabolic products should be extracted as final usable products to take advantage of all their properties within a biorefinery concept (Šoštarič et al., 2012).

There are several advantages using microalgae as a feedstock instead of terrestrial plants; several vegetable oils are used as typical raw materials of biodiesel. But, an advantage over these is that, for microalgae there is no need to encroach valuable crop and virgin land nor it is necessary to fertilize soils. Algae can grow practically anywhere, where there is enough light. Algae can grow in fresh or saline water and can be produced in the laboratory. This is essential also because all parameters can be controlled, and, if marine algae are used, there is no need to carry large quantities and frequent supplies of freshwater (Sun et al., 2010; Williams and Laurens, 2010). Another advantage for using microalgae as raw material is also that they can be converted with a large number of different methods to obtain liquid fuel and gas, by using biochemical or thermo-chemical processes. The former will

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produce ethanol and biodiesel; the latter will produce oil and gas. Microalgae can also be directly combusted, even if it is not convenient because of high water content.

The process to produce biodiesel from microalgae consists of four main steps: cultivation, concentration, lipid extraction and, finally, transesterification. After the cultivation process a dewatering step is required. Common methods are: flocculation/sedimentation (Şirin et al., 2013), dissolved air flotation (DAF), centrifugation and filtration processes (Kim et al., 2013b). Novel methods are also being investigated such as using magnetic nanoparticles (Hu et al., 2013). The flocculation/sedimentation process refers to the aggregation of microalgae in suspension to form masses that can subsequently settle. Its efficiency is increased by using several flocculants. This process reduces the need of energy intensive separation mechanisms like centrifugation. Even if flocculation is an economic method, the concentration obtained is low (<10% of solids content) (Williams and Laurens, 2010). This means that further concentration is necessary by using other methods. For this reason, it is commonly used as an initial dewatering step and centrifugation is the most combined method use with it. Centrifugation is the preferred one, but the shear forces during the process can disrupt cells and costs are high because of energy intensive.

Alternatively, a membrane microfiltration (MF) and ultrafiltration (UF) process can be performed, which are more suitable for fragile cells and small-scale production processes (Zhang et al., 2010). For microalgae harvesting, studies are mainly addressed to MF and UF membranes, this is because of their size (in general higher than 4 μm and up to hundreds of microns, depending on the species), but they involved mostly cross-flow filtration processes.

It was demonstrated that algal filtration causes noticeable fouling and too large cake resistance (Babel and Takizawa, 2010). Membrane performance is mainly assessed by its permeability that decreases significantly with time due to these phenomena. Dynamic filtration has been proved to be a successful method in several processes since high shear rates can reduce fouling resistance (Ríos et al., 2012). In particular, vibratory shear enhanced process (VSEP) has been studied in drinking water purification, pervaporation, baker's yeast microfiltration and skim milk ultrafiltration, landfill leachates, etc. (Shi and Benjamin, 2011). In fact, it was demonstrated that increasing the shear rate makes it harder for algae to deposit on membrane; this means that a higher flux can be obtained with the consequent reduction of concentration polarization and cake build-up (Zhang et al., 2010). To obtain this result without decreasing trans-membrane pressure a dynamic or shear-enhanced filtration can be used. Three types of dynamic filtration have been studied so far: with rotors between fixed membranes and rotating or vibrating membranes (Jaffrin, 2008). The characteristics of these types of dynamic filtrations have been studied in various models produced by different manufacturers, with polymeric or ceramic membranes (Rios et al., 2010). Others studies in this field demonstrated that further improvement can be obtained adding straight vanes of various height to rotating disks fixed in a dynamic filtration module (Jaffrin, 2008). Studies showed that this system yields higher permeate fluxes than conventional cross-flow filtration (Bott et al., 2000), but at the same time it is more expensive and complex, and it consumes more energy.

A step of membrane characterization should be considered, also to determine or to prove commercial membranes characteristics. Many parameters can be studied, but the most important are: zeta potential, hydrophilicity/hydrophobicity, molecular weight cut-off (rejection), pore size, porosity and distribution (Martin et al., 2003; Palacio, 1999). Studies regarding membrane characterization and modification are addressed to the permeation flux and fouling phenomenon with special considerations on cut-off, constitutive materials and surface properties (charge, hydrophilicity).

In this work, a comparison between the performances of vibrating and conventional cross-flow filtration systems has been made from culture growth both in laboratory and in pilot plant. Several experiments with different membranes have resulted in an understanding which membrane material and cut-off could be the best for this application.

## 2. Experimental

### 2.1. Materials

#### 2.1.1. Microalgae

Experiments were carried-out with two types of microalgae strains: *Nannochloropsis gaditana* Lubián, with spherical cells and an approximate diameter of 3–4 μm, and *Phaeodactylum tricornutum* Bohlin, with fusiform cells an approximate length of 40 μm and width of 4 μm. Images of both species can be observed in the Electronic Annex 1. These strains have long been used in aquaculture and as they are very diverse in size and shape they offer two different particle characteristics that are interesting to compare in filtration. Algal strains were kindly provided by the Institut de Recerca i Technologia Agrolimentaries in Sant Carles de la Ràpita (Tarragona, Spain). Algae were grown in 300 L polyethylene bags with seawater (37 g $L^{-1}$ salinity) filtered through 25, 10, 5 and 1 μm pore size filters (polyKLEAN, MICRO-KLEAN, 3 M/Cuno), UV sterilized and enriched with commercial fertilizer (0.3 mL $L^{-1}$ of Codafol 14.6.5, Sustainable Agro Solutions S.A., Lleida, Spain). Cultures were kept at 25 °C (±2), aerated and illuminated (16:8 light:dark cycle) with daylight fluorescents which gave an irradiance of 200 μmol photon $m^{-2}$ $s^{-1}$ at the bag surface. In all experiments recollection was performed at the stationary phase, which was reached by all cultures after a period of about 10 days. Before each experiment the concentration of the culture was measured and this resulted in about $21 \pm 2.7 \times 10^6$ cells/mL (mean and standard deviation, as in the entire article) for *N. gaditana* and approximately $21 \pm 1.8 \times 10^6$ cells/mL for *P. tricornutum*. These concentrations were measured with a microscope Carl Zeiss AxioScope A1 by hemocytometer.

#### 2.1.2. Membranes

Commercial polymeric membranes were used and their properties are listed in Table 1. Their filtration area was 0.0155 $m^2$ for conventional cross-flow filtration module, with a rectangular shape, and 0.0446 $m^2$ for a dynamic one, with a circular shape. Membranes of several materials and with lower porous size than microalgae were chosen.

### 2.2. Methods

#### 2.2.1. Membrane morphological characterization

The morphology of the membranes was studied by using scanning electron microscopy (JEOL JSM-6400 Scanning Microscopy Series, with a working voltage of 15 kV) to obtain cross-section or surface micrographs of the membranes. Samples were wetted into ethanol and immersed into a liquid nitrogen bath to freeze them, which allowed the membranes to be broken preserving the internal porous structure. After that, samples were covered with a gold layer with sputtering process in order to make them conductive (Torras et al., 2007).

#### 2.2.2. Membrane electrokinetic potential

The membranes surface charge was calculated from the zeta potential measure as a function of pH over the range 2–10 with a 1 mM KCl electrolyte solution by SurPASS Surface Potential Analyzer (Anton Paar). The membranes were immersed in the

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