



Cyclic pressurization assisted extraction of lipids from microalgae for biodiesel production: Non-equilibrium and equilibrium data



Gustavo Batista^a, Guilherme A.S. Surek^a, Cristina Benincá^b, Marcos L. Corazza^a, Everton F. Zanoelo^{a,*}

^a Federal University of Paraná, Department of Chemical Engineering, Curitiba, PR, Brazil

^b Federal University of Rio Grande, School of Chemistry and Food, Santo Antônio da Patrulha, RS, Brazil

HIGHLIGHTS

- Cyclically pressurized extraction of lipids from microalgae was investigated.
- Alkyl esters were produced from the extracted lipids.
- Equilibrium data were obtained from kinetic curves.
- The yield of lipids by cyclic pressurization extraction was up to 53% higher than that at atmospheric pressure.
- The yield of ester was $\approx 89\%$ from lipids extracted with ethanol at atmospheric pressure and under cyclic pressurization.

ARTICLE INFO

Article history:

Received 29 July 2015

Received in revised form 18 September 2015

Accepted 21 September 2015

Available online 1 October 2015

Keywords:

Lipids

Microalgae

Extraction

Kinetics

Equilibrium data

Biodiesel

ABSTRACT

The extraction of lipids from a microalgae (*Chlorella pyrenoidosa*) was experimentally investigated at a constant temperature of 30 °C with ethanol. The experiments involved a bed of dry solids loaded in a cylindrical vessel with approximately 23.5×10^{-3} kg of solvent. At the top of the cylinder there was a moving piston to allow extraction at cyclic or constant pressurization. A set of 10 kinetic extraction experiments was performed by varying the solid–liquid feed ratio at 5 levels under atmospheric pressure (91.4 kPa) and cyclic pressurization (300 s at 91.4 kPa + 300 s at 200 kPa per cycle) for 25,200 s ($5^1 \times 2^1 = 10$). At $p \leq 0.1$ a significant statistical effect of cyclic pressurization and solid–solvent ratio on mass fraction of extracted lipid at equilibrium (y_{Ae}) was observed, while the influence of pressure at cyclic pressurization on the same response was negligible. y_{Ae} was a parameter of a reliable first-order kinetic model tuned on different extraction curves involving a full set of almost 500 experimental kinetic data. Based on these equilibrium data, the highest yield and efficiency of lipid extraction were 11.3% d.b. and 72%, respectively. The slopes of tie lines in rectangular equilibrium diagrams of Ponchon–Savarit for extraction at atmospheric pressure and cyclic pressurization confirm that lipids in microalgae are not easily removed from the insoluble solid. The fraction of microalgae lipids converted to biodiesel by an esterification reaction based on the method of Hartman and Lago was approximately $89 \pm 7\%$.

© 2015 Elsevier Ltd. All rights reserved.

1. Introduction

Microalgae is a renewable important source of lipids (i.e.; ≈ 10 –30% in dry basis under normal growth conditions) that has been extensively explored in laboratory-scale for biodiesel production [1–3]. The advantages of using microalgae instead of classical biofuel crops for biodiesel production are well-documented in the literature [4–7]. However, it is also

well-known that the low yield of lipid extraction from microalgae is likely the most important limitation for commercial production of biodiesel from that solid, so it has been a matter of concern of many investigations [4,5,8–10].

Among the modern procedures of lipid removal from microalgae with solvents, extraction with supercritical fluids [3,6], assisted by pulsed electric field [11,12], by microwave [4,7,8,13] and by ultrasound [2,4,8,13] have been already considered. Many different solvents have been tested with discrepancies in terms of yield of lipid extraction, such as supercritical CO₂, *n*-hexane, *n*-heptane, chloroform, *iso*-propanol, methanol, ethanol and mixtures of them [3,4,6–8].

* Corresponding author. Tel.: +55 41 33613583; fax: +55 41 33613674.

E-mail address: everton.zanoelo@ufpr.br (E.F. Zanoelo).

Nomenclature

k	rate of extraction (s^{-1})	N	inverse of the solution holding capacity of the insoluble matrix defined in Eq. (3) ($kg\ kg^{-1}$)
m_A	mass of lipids A in the spent microalgae at equilibrium (kg)	P	pressure (kPa)
m_{Ae}	dry mass of extracted lipids used in the esterification reaction (kg)	R	wet solid to solvent mass ratio ($kg\ kg^{-1}$)
m_B	mass of inert B in the spent microalgae at equilibrium (kg)	R^2	coefficient of determination
m_C	mass of ethanol C in the spent microalgae at equilibrium (kg)	t	time of extraction (s)
m_{ds}	mass of dry microalgae fed into the batch extractor (kg)	T	temperature of extraction ($^{\circ}C$)
m_e	mass of produced esters from the esterification reaction (kg)	U	uncertainty in the values of y_{Ae} ($kg\ kg^{-1}$)
m_L	mass of ethanol fed into the batch extractor (kg)	x_{Ae}	equilibrium solute mass fraction in the spent microalgae on an inert-free basis ($kg\ kg^{-1}$)
m_S	mass of microalgae fed into the batch extractor (kg)	y_A	solute mass fraction in the ethanol phase at time t ($kg\ kg^{-1}$)
M	mass ratio of retained ethanol to inert ($kg\ kg^{-1}$)	y_{Ae}	equilibrium solute mass fraction in the ethanol phase ($kg\ kg^{-1}$)
n	number of experimental kinetic data of lipid mass fraction in the ethanol phase at a particular extraction run	Y_e	yield of lipid extraction (%)
		Y_{es}	yield of esters by esterification of extracted lipids (Eq. (7)) ($kg\ kg^{-1}$)

In spite of its efficiency of solute removal comparable to those obtained by ultrasound assisted extraction [14], an emerging technology that has never been applied for lipid extraction from microalgae is the cyclically pressurized extraction [15,16]. It is specially attractive for such a purpose if one considers that it demands low operating costs due to the low temperatures often applied [15,16]. In other words, it may contribute to reduce the energy consumption in the operation of extraction, a critical obstacle for a large-scale production of biodiesel from algae [4,7,17]. It also presents the advantage of using moderate pressures (<1013 kPa), so the capital cost is expected to be lower than that required to build and put in operation a plant that uses supercritical CO_2 extraction, microwave or ultrasound assisted extraction.

Based on all the alleged benefits of solid–liquid extraction assisted by cyclic pressurization, it was currently tested for removal of lipids from *Chlorella pyrenoidosa* with ethanol. In particular, the aim of the investigation was to verify the effect of pressure, pressure cycles and solid–liquid ratio on the solute mass fraction in the liquid phase at equilibrium. Based on these responses, the yield and the efficiency of lipid removal at equilibrium were estimated. The efficiency was calculated by considering that exhaustive lipid extraction was achieved in a Soxhlet at the boiling point of ethanol at atmospheric pressure. The potential of alkyl esters production from the extracted lipids was also investigated by involving a reliable approach based on the extensively reported method of Hartmann and Lago [18].

Ethanol was preferred to many other organic solvents because of its wide availability at low cost, its high vapor pressure and low toxicity that allow its easy removal from the extracted oil and the use of the spent biomass for animal feed, respectively [19–21]. As it happens with methanol [22–24], it may be used as a reactant for the lipid conversion to alkyl esters, a relevant advantage when the extraction of lipids is for biodiesel production [25–27]. Its polar nature may be interpreted as a negative point, but it allows to disrupt some lipid–protein associations that makes possible to extract a particular fraction of neutral lipids [5,28]. In spite of all the given reasons for using ethanol as solvent, the most important motivation to do that was the highest amount of lipids always extracted with ethanol when samples of microalgae identical to those currently used (*Chlorella pyrenoidosa* obtained under identical growth and drying conditions) were also exposed to hexane at the same circumstances [29].

2. Materials and methods

2.1. Extraction experiments and kinetic data

Commercial samples of *Chlorella pyrenoidosa* (Lot 049277, Florian, Piracicaba, Brazil) with particle size between 106 μm and 150 μm , measured by a screen analysis with a 150 and a 100 mesh screen of the Tyler standard screen series (Bertel, Caieiras, Brazil) were used in all the extraction experiments. The same solid presented a moisture content close to $2.50 \pm 0.03\%$ in wet basis, determined by oven drying it under vacuum at ≈ 67 kPa and $60^{\circ}C$ for 24 h (440–1D, Ethik Technology, Vargem Grande Paulista, Brazil).

Extraction always took place in a bath cylindrical chamber made of polycarbonate and internally equipped with a moving piston. To set the pressure at a desired value the piston was forced through the cylinder with a hydraulic press (15 tons, Schulz, Joinville, Brazil) up to a position defined by the Boyle's law. To have extraction at a constant temperature of $30^{\circ}C$, the extraction vessel was kept in a digital-controller bath (SL 152/10, Solab, Piracicaba, Brazil) for the entire period of lipid removal equal to 25,200 s. Extraction was carried out with a single sachet of solid loaded in the cylinder already filled with a known mass of ethanol (23.5×10^{-3} kg) (purity $\geq 99.8\%$, Neon, São Paulo, Brazil). To avoid the transport of insoluble solids to the liquid phase all the sachets were made of paper filter with an average pore size (14 μm) much smaller than the smallest particle of microalgae used in the experiments (106 μm). To monitor the kinetics of extraction liquid samples were periodically removed from the extraction chamber. In almost all the examined cases, Δt was 1200 s or 3600 s. Total soluble compounds in ethanol were determined by a gravimetric method involving a digital balance with a precision of $\pm 10^{-7}$ kg (AG-200, Gehaka, São Paulo, Brazil) and an oven to solvent evaporation for 24 h at $55^{\circ}C$ (400–2ND, Ethik Technology, Vargem Grande Paulista, Brazil).

A full set of 10 kinetic experiments was performed at 5 levels in terms of solid–liquid feed ratio under constant atmospheric pressure (91.4 kPa) and cyclic pressurization from 91.4 kPa to 200 kPa. To do that the mass of *Chlorella* in the sachets was varied between 1×10^{-3} and 6×10^{-3} kg by a step of 1.25×10^{-3} kg. In the case of cyclic pressurization extraction, the frequency of cycles was always equal to 1:600 per second (i.e.; 300 s at 91.4 kPa + 300 s at 200 kPa for 25,200 s). To assess the effect of pressure

Download English Version:

<https://daneshyari.com/en/article/205495>

Download Persian Version:

<https://daneshyari.com/article/205495>

[Daneshyari.com](https://daneshyari.com)