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Direct liquid–liquid extraction of lipid from municipal sewage sludge for biodiesel production

Magdalena Olkiewicz [a], Martin Pablo Caporgno [a], Agustí Fortuny [b], Frank Stüber [a], Azael Fabregat [a], Josep Font [a], Christophe Bengoa [a,*]

[a] Departament d'Enginyeria Química, Universitat Rovira i Virgili, Av. Països Catalans 26, 43007 Tarragona, Spain
[b] Departament d'Enginyeria Química, Universitat Politècnica de Catalunya, Av. Víctor Balaguer S/N, 08800 Vilanova i la Geltrú, Spain

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ABSTRACT

Municipal sludge from wastewater treatment plants is a promising lipid feedstock for biodiesel production as it contains a significant amount of lipids. However, the energy necessary to remove its high water content is a major inconvenience for scaling up because of the high associated cost. In addition, the expensive conventional sludge drying methods are not effective enough for lipid recovery, thus reducing the potential biodiesel production. This study explores an alternative method, the direct sequential liquid–liquid extraction, which was performed in a batch mixer–settler reactor at room temperature, using hexane as a solvent, after previous sludge acidification showed significant increase in the lipid efficiency. The optimisation study demonstrated that, after three stages, 91% of lipid from primary sludge was recovered. The optimised extraction gave slightly higher lipid (27%, dry sludge) than the standard method (25%, dry sludge), supporting the suitability of the proposed process. Finally, this work demonstrates that the residual lipid-extracted sludge is still a good feedstock for energy production via anaerobic digestion. Anyway, the economic and environmental aspects of biodiesel production from sewage sludge could be improved.

1. Introduction

The global continuous growth of energy demand poses urgent problem due to the fossil fuels' depletion, as they currently represent about 75% of all energy consumed worldwide [1]. One of the most promising renewable fuels proposed as an alternative is biodiesel that can be directly used with current engine and refuelling technology [1–3]. However, the competitive potential of biodiesel is currently limited by the price of the common lipid feedstocks, which constitutes 70–85% of the overall biodiesel production cost, thus strongly influencing the final price of this biofuel and raising the concerns of food shortage versus fuel crisis [1].

In turn, municipal sewage sludge from wastewater treatment plants (WWTPs) is gaining more attention nowadays as a lipid feedstock for the production of biodiesel as the dry sludge can contain up to 30 wt.% of lipids [1–6]. In fact, sewage sludge is a waste that needs specific treatment before disposal and represents a major cost in the WWTP operation. In addition, the WWTPs annually produce higher amounts of sludge due to the expansion of urbanised and industrialised areas. Therefore, the sewage sludge can be envisaged as a relatively cheap, readily available, and in abundance feedstock, which can make the biodiesel production profitable. Furthermore, it is one possible alternative to take advantage of the excess sludge, reusing it as a source of lipid for the production of biodiesel, consequently lowering the WWTP operation cost. Nevertheless, the production of biodiesel from sludge poses great challenges for a fast commercialisation. The main challenge to be faced by biodiesel production from waste sludge is an efficient lipid extraction from water, as water can account for up to 95–98 wt.%, so dewatering and drying constitute more than 50% of total biodiesel production cost [4]. This makes the production very expensive and difficult to scale up due to the cost of the energy necessary for water removal step.

Most of the literature reports only the utilisation of dry sludge in the extraction of lipid by an organic solvent [3,4,6]. Recently, some works have used dewatered primary [5] and secondary sludges [7] by centrifugation, but the energy of dewatering still constitutes 14% of the total biodiesel production cost [4]. On the other hand, the direct transesterification of sewage sludge into biodiesel has been also reported "in situ" on dry [2,4] and dewatered sludges [7]. Interestingly, the biodiesel yield obtained from dewatered sludge was about 20% lower than from dried sludge [7]. The "in situ" process can reduce the time and amount of solvent, however, after transesterification, a solvent recovery step is then needed, adversely affecting the overall cost of biodiesel.

Moreover, water elimination from biomass by conventional thermal drying or freeze-drying results in the loss of valuable organic compounds [8,9]. This fact can also provoke the loss of lipids in sewage

* Corresponding author. Tel.: +34 977 558619; fax: +34 977 559667.
E-mail address: christophe.bengoa@urv.cat (C. Bengoa).

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sludge hence decreasing biodiesel production yield. Nevertheless, the influence of sludge drying on the lipid extraction efficiency has not been yet evaluated. Therefore, the effect of common sludge drying methods on the lipid extraction efficiency as well as the fatty acid composition still needs to be examined.

Surprisingly, the direct liquid–liquid extraction has neither been reported, so the sludge drying and dewatering would thus become unnecessary. Thus, the main objective of this study was to explore this alternative and to demonstrate its feasibility. Three types of sludge generated in WWTPs were tested. Optimisation of liquid–liquid extraction was studied varying the ratio sludge/hexane, time of contact, and number of consecutive batch extraction steps in order to get the most favourable process. In addition, as the residual sludge after lipid extraction is still a potential biomass for energy recovery, the residual sludge can be used as feed for anaerobic digestion, which is widely implemented in municipal WWTPs. Therefore, the lipid-extracted sludge was subjected to anaerobic digestion to check out its potential for biogas generation. Finally, a simplified energy consumption estimation of the biodiesel production via liquid–liquid extraction was conducted.

2. Materials and methods

2.1. Reagents

The transesterification/esterification experiments were carried out using anhydrous methanol and sulfuric acid from Sigma-Aldrich at the highest purity available. Standard used for identification and quantification of fatty acid methyl esters (FAMEs) was supplied by Supelco (37 component FAMEs mix, ref: 47885-U). For the free fatty acid (FFA) analysis, 0.5 M potassium hydroxide volumetric solution was purchased from Fluka. All other solvents and reagents were high performance liquid chromatography grade and analytical reagent grade provided by Sigma-Aldrich.

2.2. Sludge collection and handling

Primary, secondary and blended sludges were collected from the municipal WWTP in Reus (Tarragona, Spain) with a capacity to daily process 25,000 m^3 of wastewater. Fig. 1a shows a schematic diagram of the WWTP, illustrating the step where these different types of sludges are generated. Primary sludge was collected after partial gravity thickening. Secondary sludge, produced by an activated sludge process, was collected after partial thickening by flotation. Blended sludge was collected after the combination of primary and secondary at a ratio of 65:35, v/v. The collected sludges were immediately stored at 4 °C prior to use. Because the sludge properties could be changed during long storage time, fresh sludge was always used for each experiment.

The inoculum used in anaerobic digestion tests was sludge collected from a mesophilic anaerobic digester in the same facility.

2.3. Sludge drying

2.3.1. Primary sludge — evaluation of drying methods

According to standard method 5520E [10], sludge was dried using magnesium sulfate monohydrate but without previous acidification. Using the referenced method, the sludge sample was considered as completely dried.

Oven drying method was conducted using an universal oven ULE400 (Memmert GmbH, Germany) at two different temperatures, 105 °C for 2 days based on standard method 2540G [10] or 70 °C for 3 days [3].

Freeze-drying method was conducted by using the method presented elsewhere [2]. At first, sludge was centrifuged and then allowed to freeze for 2 days at −20 °C. Afterwards, the frozen sludge was freeze-dried in an automatic vacuum freeze dryer, model FT33-A (Armfield Limited) for 2 days.

In the sun drying method, the sludge sample was left outside for 10 days, where the temperature was in the range of 25–35 °C.

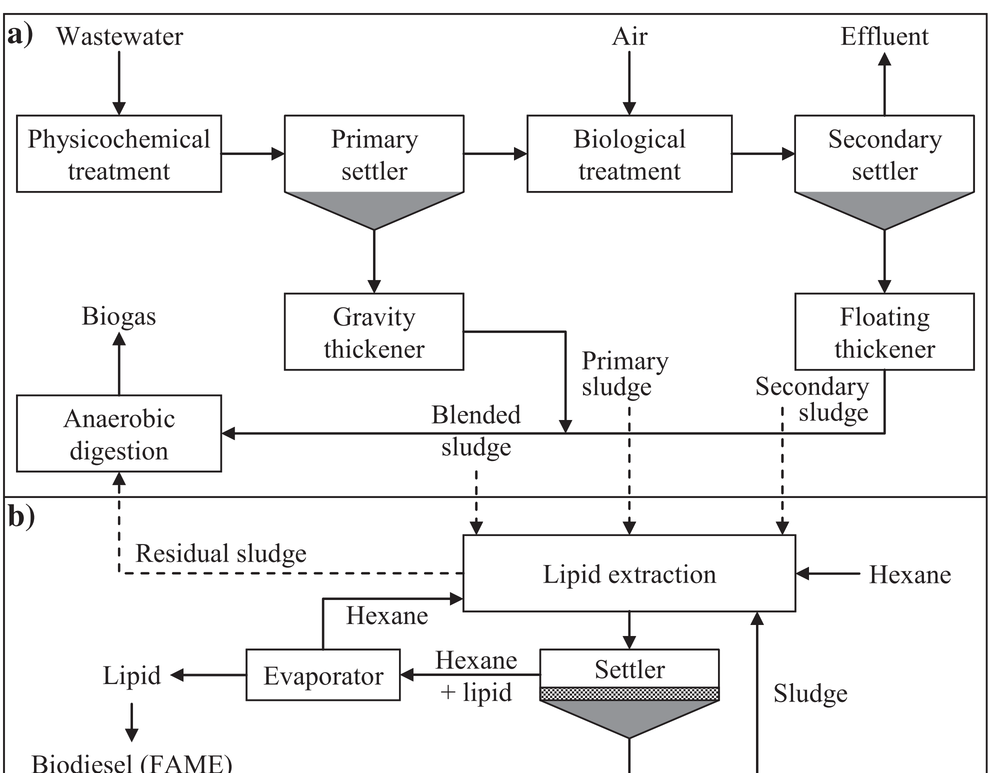

Fig. 1. Diagram of the wastewater treatment plant (a) and schematic diagram of the experimental liquid–liquid extraction setup carried out in the present study (b).