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Effects of pre-treatments on the lipid extraction and biodiesel production from municipal WWTP sludge

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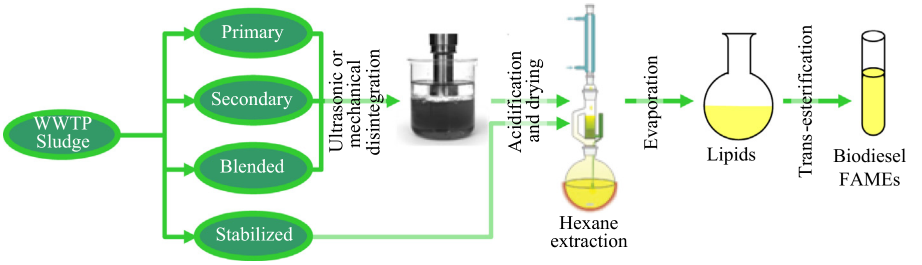
HIGHLIGHTS

- One of the first attempts made in Europe to convert municipal sludge to biodiesel.
- First attempt to optimise the yield of lipids extracted using pre-treatments.
- First attempt to compare four sludge of different characteristics from the WWTP.
- Primary sludge has a conversion to FAME of 19% in dried sludge basis.
- Pre-treatments do not affect essentially the conversion and composition of FAME.

GRAPHICAL ABSTRACT



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ABSTRACT

Biodiesel production is currently limited due to high raw material costs. The potential of using sludge from municipal wastewater treatment plants as an alternative lipid feedstock was investigated. Four different types of sludge (primary, secondary, blended and stabilised) were tested in lipid extraction by Soxhlet using hexane, and biodiesel production by acid catalysis. To improve the extraction efficiency, the influence of pre-treatment methods (ultrasonic and mechanical disintegration) and duration of these treatments were investigated. Finally, the effect of sludge acidification with concentrated HCl was also evaluated. The pre-treatment methods did not increase significantly the amount of extracted lipid as well as biodiesel yield. Previous sludge acidification showed lower yield of lipids from primary, secondary and blended sludge. However, the amount of saponifiable lipids was higher, giving the overall biodiesel yield almost unchanged. Among the four sludges tested, primary sludge achieved the greatest lipid and biodiesel yields, 27% and 19% respectively, on the basis of dry sludge. The highest biodiesel yields obtained from blended, secondary and stabilised sludge amounted to 15%, 4% and 2% respectively, on the basis of dry sludge. No significant influence of the pre-treatments and acidification on the fatty acid composition was found. At least 8 fatty acids were determined, with a predominance of palmitic (C16:0), stearic (C18:0) and oleic acid (C18:1). The comparison of sludge fatty acids profile with common biodiesel feedstocks showed suitability of WWTP sludge for production of biodiesel.

1. Introduction

Biodiesel is one of the most promising renewable fuels as it is biodegradable, less toxic than fossil diesel, compatible with current commercial diesel engine and refuelling technology, and it has low

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emission profile. Additionally, it has excellent lubricating properties and it could provide energy density similar to diesel [1–4]. Biodiesel is generally produced by transesterification of vegetable oils or animal fats, yielding fatty acids methyl esters (FAMEs) from the lipid fraction. The production of biodiesel in the EU increased from 3.6 (2005) to 10.7 billion litres in 2010 [5]. However, nowadays the competitive potential of biodiesel is limited due to high cost of common lipid feedstocks (soybean, canola, rapeseed, sunflower, palm, and coconut oils), which constitutes 70–85% of the overall biodiesel production cost, strongly influencing the final price of this biofuel [2,3,6,7]. In fact, the production of biodiesel decreased by 10% in 2011 as compared to 2010 [5]. In addition, lack of agricultural lands for growing biodiesel feedstocks limits biodiesel expansion and has contributed to the increase of food prices over the past few years, raising the concerns of food shortage versus fuel crisis [3]. Thus, there is an urgent need to find an alternative, cheaper feedstock, non-edible, readily available and in large quantities.

In contrast, municipal sewage sludge that is gaining more attention nowadays in biodiesel production can meet the requirements of lipid feedstock [3,4,8]. Sewage sludge is a waste, formed during treatment of wastewater in wastewater treatment plants (WWTPs) that needs specific treatment before disposal and represents a major cost in WWTP operation. In addition, WWTPs annually produce higher amounts of sludge due to the expansion of urbanised and industrialised areas. Each year, higher quantities of sludge are produced and the number is estimated to increase from 10 million tons (2005) to 13 million tons in 2020 in the whole of EU [9]. Additionally, dry sludge could comprise up to 30 wt% of lipids [10–12], which could be converted into FAMEs. Recent studies have indicated that lipid contained in sewage sludge could be potential feedstock for biodiesel [1,6,7,11,13]. Nevertheless, production of biodiesel from sludge poses great challenges for fast commercialisation. The optimisation of lipid extraction is a major challenge that may affect the economy of the process [7].

It has been demonstrated that ultrasonic pre-treatment [14,15] and acid hydrolysis [16] are able to increase the lipid extraction yield from biologic samples but their utilisation to improve lipid extraction from sludge has not been reported. These pre-treatments are able to release the lipids from other macromolecules which are not available to solvent in bonded form. Therefore, the utilisation of the sludge pre-treatments could also improve the efficiency of extraction. The most common methods of sludge pre-treatment are ultrasonication and mechanical disintegration, commonly used to enhance biogas production [17–20]. Ultrasonic energy is able to disintegrate sludge flocs and disrupt large organic particles, breaking down bacterial cell wall and releasing intracellular substances and extracellular polymeric substances into aqueous phase [14,15,17,20]. Mechanical disintegration is used to reduce size of the sludge particles, disintegrate cells and release organic components into sludge [18,19]. On the other hand, acid hydrolysis of sludge is another pre-treatment method used to increase the solubility of the organic matter contained within sludge and thus to reduce the amount of sludge and improve its dewaterability [21]. As sewage sludge is a processed sample, in which lipid can be bonded to proteins, carbohydrates and/or minerals, the proposed pre-treatment methods could facilitate the extraction of lipids by sludge disintegration.

The purpose of this study was to investigate the influence of sludge type (primary, secondary, blended and stabilised) and sludge pre-treatments (ultrasonic and mechanical), combined with or without sludge acidification, on the yield of lipid extracted as well as biodiesel (FAMEs) produced. Finally, the composition of FAMEs was determined and compared with common biodiesel feedstocks.

2. Materials and methods

2.1. Chemicals

Lipid extraction experiments were conducted using hexane of laboratory reagent grade (ref: 208752) and magnesium sulphate monohydrate (ref: 434183) purchased from Sigma–Aldrich. Fuming hydrochloric acid (ref: 84418) used for sludge acidification was purchased from Fluka. Transesterification experiments were carried out using hexane (ref: 34859), anhydrous methanol (ref: 322415) and sulphuric acid (ref: 33974) from Sigma–Aldrich at the highest purity available. Sodium chloride (ref: 71379), sodium bicarbonate (ref: S6297) and anhydrous sodium sulphate (ref: 239313) were provided by Sigma–Aldrich. Standard used for identification and quantification of fatty acid methyl ester (FAMEs) was supplied by Supelco (37 component FAMEs mix, ref: 47885-U). Analytical standards of free fatty acids (FFA) were provided by Sigma–Aldrich (C12 ref: L556, C14 ref: 70082, C15 ref: 96125, C16 ref: P0500, C16:1 ref: P9417, C18 ref: S4751, C18:1 ref: O1008, and C18:2 ref: L1376. High Performance Liquid Chromatography (HPLC) grade toluene (ref: 650579) used for preparation of FFAs solution was also provided by Sigma–Aldrich.

2.2. Sludge collection, handling and characterisation

Primary, secondary, blended and stabilised sludge were collected from the municipal WWTP in Reus (Tarragona, Spain) with a capacity to process near 25,000 m^3 of wastewater per day. 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 in the feed of the anaerobic reactor. Stabilised sludge, produced by an anaerobic digestion was sampled after belt filter press dewatering. Sludge samples were taken every 2–3 weeks and the sampling was done four times. The samples were immediately delivered to the laboratory and stored at 4 °C prior to use (maximum storage time 7 days).

Each sample of received sludge was characterised in order to determine total solids (TS) and volatile solids (VS) content, both according to standard method 2540G [22]. Chemical oxygen demand (COD) was measured in a UV-spectrophotometer (DINKO UV–VIS 800 spectrophotometer) according to standard method 5220D [22]. The Sludge characteristics are given in Table 1. As the sludge composition varies during the wastewater treatment and depends on the specific treatment applied, therefore the stabilized sludge gave the largest content of TS and VS due to the water elimination by filter press system, and the primary sludge gave higher quantity of TS, VS and COD than blended and secondary.

2.3. Pre-treatment of sludge samples

Before the extraction, primary, secondary and blended sludge were pre-treated using ultrasonic and mechanical disintegration methods. Due to its solid appearance, anaerobically stabilized sludge was used as received without previous disintegration.

The ultrasonic disintegration experiments were carried out using the procedure previously described elsewhere [23]. The mechanical disintegration experiments were carried out using a mechanical homogenizer (Taurus, Turbo-rotation system) at 600 W of the input of energy at room temperature. 200 ml of sludge was used for each test of each disintegration method. In order to optimise the disintegration time, the blended sludge was disintegrated by both methods for 5, 10, 15 and 20 min.