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Evaluation of different sludges from WWTP as a potential source for biodiesel production

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Abstract

Biodiesel expansion is currently limited due to high raw material costs for its production. The potential of using sludge from municipal wastewater treatment plants as an alternative lipid feedstock for biodiesel production was investigated. Experiments were conducted to evaluate the suitability of four different types of wastewater sludges for biodiesel production. Lipids were extracted from primary, secondary, blended and stabilized sludge in a Soxhlet extractor, using hexane as a solvent. Finally, the lipids were converted by acid catalysis transesterification into their corresponding fatty acid methyl esters (FAMES) - biodiesel. Results indicated that among four sludge tested, primary sludge achieved the greatest lipids and biodiesel yields. The amount of extracted lipids for primary sludge was 25.3% compared to 21.9%, 10.1% and 9.1% (dry wt) for blended, stabilized and secondary sludge, respectively. The FAMES yields obtained in this study were 13.9%, 10.9%, 2.9% and 1% (dry wt) for primary, blended, secondary and stabilized sludge, respectively. The estimation of annual biodiesel production based on the sludge generated in WWTP of Reus was evaluated, showing that primary sludge consists of 87% of the total biodiesel among the wastewater sludges. Gas chromatography analysis of the FAMES revealed a similar fatty acids composition for all sludge tested with a predominance of palmitic acid, stearic acid, oleic acid and linoleic acid. Comparison of sludge fatty acid profile with common biodiesel feedstocks showed their suitability for the production of biodiesel.

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1. Introduction

Nowadays, there is an urgent need for alternative cheap and renewable energy resources with no environmental impact like a biodiesel, bioethanol and biogas derived from renewable biomass. Among them, the greatest demand is currently observed for biodiesel. In 2009, biodiesel represented about 75% of total biofuels produced in The European Union (EU) [1]. Biodiesel is generally produced by the base or acid catalyzed transesterification of vegetable oils and animal fats, which yields the fatty acids methyl esters (FAMES) of the lipid fraction. Biodiesel represents an excellent alternative to conventional diesel because it is renewable, biodegradable, less toxic, and has low emission profiles. It has excellent lubricity and could provide similar energy density to diesel. It can be used directly without any engine modification and does not require new refueling station [2-4]. Due to the advantages of biodiesel mentioned above, the production of this biofuel in the EU and in the world is increasing. For example, the production of biodiesel in the EU has increased from 3.6 billion liters in 2005 to 10.7 billion liters in 2010 [5]. However, the increasing demand for biodiesel has done that the demand for lipid feedstocks such as soybean, canola, rapeseed, sunflower, palm, and coconut oils have also increased, and constitute between 70-85% of the overall biodiesel production cost [6,7]. Therefore, the biodiesel production is currently limited due to high raw material costs. In addition, lack of agricultural lands for growing biodiesel feedstocks limits its expansion and has impacted on the food price increases over the past few years. This fact can provoke that providing enough feedstock to cater to the increasing demands for biodiesel in the next few years will be at the expense of the food manufacturing industry. Thus, it is needed to find a cheap alternative feedstock, uncompetitive with food market, readily available and in large quantities. The search for cheaper feedstocks for the production of biodiesel has turned attention to various forms of waste alternative raw materials, including waste animal fats [8], waste cooking oil [9] and now lipids in sewage sludge [10,11].

Sewage sludge is a waste formed during treatment of wastewater. As shown in Fig. 1, wastewater treatment plant (WWTP) produces primary and secondary sludges, generally after primary and secondary treatment with significant differences between their compositions. The primary sludge is a combination of floating grease and solids collected at the bottom of the primary settler after screening and grit removal. The secondary or activated sludge is composed mainly of microbial cells and suspended solids produced during the aerobic biological treatment and collected in the secondary settler. A portion of the collected secondary sludge is recycled back to the aeration basins to maintain a sufficient concentration of microorganisms [7]. The remainder of secondary sludge, after thickening is mixed with thickened primary sludge and blend of them is the by-product after wastewater treatment. The sludge produced after wastewater treatment needs to be processing to facilitate its handling or disposal [12]. The blended sludge feeds aerobic or anaerobic digester (WWTP, Reus), and after this process stabilized sludge is obtained.

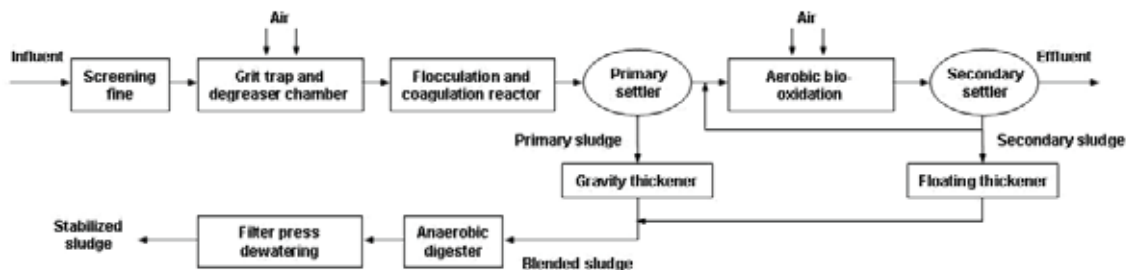


Fig. 1. Schematic diagram of municipal wastewater treatment plant in Reus, Spain.

Wastewater treatment plants produce each year higher quantities of sludge due to the increase of urbanization and industrialization, it can be considered as a continuous process in the future. It was estimated the total sludge generation in the EU in 2020 will be approximately 12.9 million tones of dry sludge per year, compared with 10 million tones in 2005, an overall increase by about 30% [13]. Additionally, the sludge could comprise up to 30 wt% of a lipid fraction [10], which could be converted into FAMES. The extraction and transformation of the lipids could yield an unexploited source of cheap and readily available feedstock for biodiesel production. Furthermore, it is one possible alternative to take advantage of the excess of sludge, reusing it as a source of lipid for the production of biodiesel.

The purpose of this research was to investigate the influence of sludge type on the lipid extraction and biodiesel yields. Because of the difference between the composition of primary, secondary and stabilized sludge the influence of sludge type on the fatty acids composition of biodiesel was also examined. In addition four different types of sludge were tested in order to evaluate the suitability of the wastewater sludges for biodiesel production. Finally, the paper provides comparison results on the composition of fatty acids in the lipid fraction of sludge to soybean and lard, in order to demonstrate that sewage sludge can be a potential feedstock for the production of biodiesel.

2. Materials and methods

2.1. Chemicals

Hexane laboratory reagent grade and magnesium sulphate monohydrate were purchased from Sigma-Aldrich. Fuming hydrochloric acid was purchased from Fluka. Transesterification experiments were carried out using hexane, anhydrous methanol and sulfuric acid from Sigma-Aldrich at the highest purity available. Sodium chloride, sodium bicarbonate and anhydrous sodium sulfate were provided by Sigma-Aldrich. The standard used for identification and quantification of fatty acids was supplied by Supelco (37 component FAMES mix, 47885-U).

2.2. Sludge collection and handling

Primary, secondary, blended (blend of primary and secondary in ratio 65:35) and stabilized sludge samples were collected from the municipal WWTP in Reus (Tarragona, Spain) with a capacity to process near 25,000 m³ of wastewater per day. As it can be observed in Fig.1, primary sludge samples were collected after gravity thickening. Secondary sludge samples, produced by an activated sludge process, were collected after partial thickening by flotation. Stabilized sludge, produced by anaerobic digestion of blend of primary and secondary, was sampled after belt filter press dewatering. The collected sludges were immediately delivered to the laboratory and stored at 4 °C prior to use (maximum storage time 4 days). The samples of sludge were taken every 2-3 weeks, and the sampling was done four times. The sludges were analysed in order to determine the total solids content (TS) according to the standard method 2540G [14]. The average TS content of four sludge samples used in this study can be seen in Table1.

2.3. Soxhlet extraction

Before soxhlet extraction, primary, secondary, and blended sludge were disintegrated using an ultrasonic disintegrator UP200S (Hielscher Ultrasonics GmbH, Germany) at 24 kHz of working frequency and 4 W/cm² of power intensity (50 W of ultrasonic power). 200 mL of sludge sample was placed in a 250 mL vessel and the sample was disintegrated at room temperature for 10 min. To avoid the

sample heating a water bath was used. Anaerobically stabilized sludge, because of its solid appearance, was used as received without previous disintegration.

The extraction procedure was carried out according to the standard method 5520E [14]. 20 g of sludge sample was acidified by adding 0.3 mL of fuming hydrochloric acid. After the acidification, to dry the sample, 25 g of magnesium sulfate monohydrate was added. The mixture was sired to obtain a smooth past and left until solidified. The mixture was stored in a desiccator at room temperature overnight. Next day, the sample was ground using a pestle and mortar until the sample was as homogenous as possible. For the sohxlet extraction, the homogenous sample was placed in a cellulose thimble (Filter Lab, ANOIA S.A.) and glass wool was added to the top of the thimble to avoid the scattering of the solid. 200 mL of hexane was added into a 250 mL round-bottomed flask previously weighed. The heating process was monitoring to allow 80 cycles of the extraction in approximately 5.5 hours. After the lipids extraction, the hexane was removed from the flask by using a rotary evaporator. The lipids were stored in a desiccator overnight and weighed the next day. The yield of extracted material was determined gravimetrically and expressed as a gram of extractable lipids per gram of dry sludge. After the quantification, the lipids were dissolved in hexane, and kept frozen at -20°C until further analysis.

2.4. Transesterification

The conversion of the lipids extracted from the sludge to FAMES (biodiesel) was carried out through acid catalysis transesterification using a modified version of Christie's method [10,15]. The lipid sample (up to 50 mg) was placed in a vial and dissolved in 1 mL of hexane. After that, 2 mL of 1% sulfuric acid in methanol was added to the vial. The vial was capped and heated overnight at 50 °C. Afterwards, to recover the FAMES, 5 mL of 5% sodium chloride in water was added and the FAMES were extracted 2 times with 5 mL of hexane, using a vortex to provide efficient mixing in the vial. The hexane phase was washed with 4 mL of 2% sodium bicarbonate in water and dried over anhydrous sodium sulfate.

2.5. FAME analysis

The FAMES produced by transesterification were analyzed using an Agilent gas chromatograph 6890GC with a flame-ionization detector (FID). The separation was achieved in a HP-INNOWax column 30m × 0.32 mm × 0.25 µm (Agilent Part No. 19091N-133), with helium as carrier gas and with a constant injector temperature 260°C. The injection volume of sample was 1.5 µL with a split ratio 20/1. The FID was at 260°C for the duration of the analysis. The oven temperature programme was start from 150 °C, holding for 1 min and then increased by 2.9 °C/min to 230 °C, holding for 1 min. A 37 component FAMES standard mixture was used for the calibration of the instrument. All calibration curves were linear with a correlation coefficient of 0.999 or better. Results of the gas chromatography runs were used to calculate the amount of saponifiable material in extracted lipids. Calculation based the amount of lipids extracted and the percentage of saponifiable material in the lipids allowed calculating the mass of biodiesel (FAMES) produced from dry sludge.

3. Results and discussion

3.1. Lipids and biodiesel yields

Table 1 shows the amount of total solids, extracted lipids, saponifiable materials, and overall biodiesel yields obtained from four types of sludge. The values represent the average of the results obtained from four different samples collected in several days. Each sludge sample was analysed three times.

Table 1. Total solids content, extraction and transesterification yields of sludges.

Sludge type	Total solids (%)	Lipids yield ^a (%)	Saponifiable ^b (%)	Biodiesel yield ^a (%)
Primary sludge	4.2 ± 1.2	25.3 ± 1.3	55.0 ± 4.8	13.9 ± 0.7
Secondary sludge	3.2 ± 0.7	9.3 ± 1.3	31.3 ± 0.6	2.9 ± 0.5
Blended sludge	3.1 ± 0.7	21.9 ± 1.9	49.9 ± 4.3	10.9 ± 1.7
Stabilized sludge	25.3 ± 4.4	10.1 ± 1.5	10.0 ± 0.7	1.0 ± 0.2

^a Lipids and overall biodiesel yields were calculated based on weight of dry sludge.

^b Percentage of transesterified lipids on the mass basis.

Values are presented as means ± SD, n = 4.

As it can be seen in Table 1, the primary sludge achieved the greatest lipids yield (25.3%) followed by blended (21.9%), stabilized (10.1%) and secondary (9.3%). The transesterification also gave the highest saponifiable matter in the lipids from primary sludge sample (55%), resulting the highest overall biodiesel yield. Comparison between stabilized and secondary sludge indicates that although the amount of lipids extracted from stabilized sludge was higher than that in secondary sludge, the secondary sludge gave a higher overall biodiesel yield owing to a much larger amount of saponifiable matter in the lipids extracted, 31.3% comparing to 10% in the lipids from stabilized sludge. This shows that an extraction from stabilized sludge produce lipids heavily contaminated with nonsaponifiable material, causing a lower productivity of biodiesel.

Among the four sludges tested, the primary achieved the greatest lipids and biodiesel yields. Primary sludge consists of organic matter originating from raw wastewater, is a combination of floating grease and solids. The secondary sludge is composed mainly of microbial cells and suspended solids produced during the aerobic biological treatment of the primary treated wastewater. Stabilized sludge comes from digestion process during which the organic matter is mineralized. Thus, it is logical that primary sludge gave the highest lipids yield and then, the highest biodiesel yield. As blended sludge is a mixture of primary and secondary, with a higher fraction of the first one, it is natural that its results are slightly lower than of the primary.

Comparing overall biodiesel yields of four sludge types, the results shows that primary sludge achieved the greatest biodiesel yield, 13,9% (Table 1), hence it is more beneficial to use this sludge to produce biodiesel. Furthermore, blended sludge needs to be stabilized before its handling or disposal usually by aerobic or anaerobic digestion. It was demonstrated that significant amount of lipids in sludge can affect both aerobic and anaerobic process [16] and can inhibit methanogenesis during anaerobic digestion [17]. Elimination of lipids form primary sludge can significantly reduce the amount of lipids in blended sludge. Therefore, reduction of lipids in sludge before digestion process can improve the production of energy from sludge; production of biodiesel from lipids fraction and improvement of anaerobic digestion process for biogas production.

3.2. FAME analysis of biodiesel produced form four types of sludge

Results of the gas chromatography runs were used to calculate the mass percentages of fatty acids presented in the extracted lipids of primary, secondary, blended and stabilized sludge. Some of the compounds detected in the chromatogram but not identified or not quantified were presented as total not identified (N.I.). Fatty acid profiles of biodiesel produced from primary, secondary, blended and stabilized sludge are presented in the Fig. 2. All types of sludge have a significant amount of palmitic acid (C16:0), oleic acid (C18:1), stearic acid (C18:0) and linoleic acid (C18:2). However, stabilized sludge contains only the fatty acids from C16:0 to C18:2 comparing with other types of sludge.

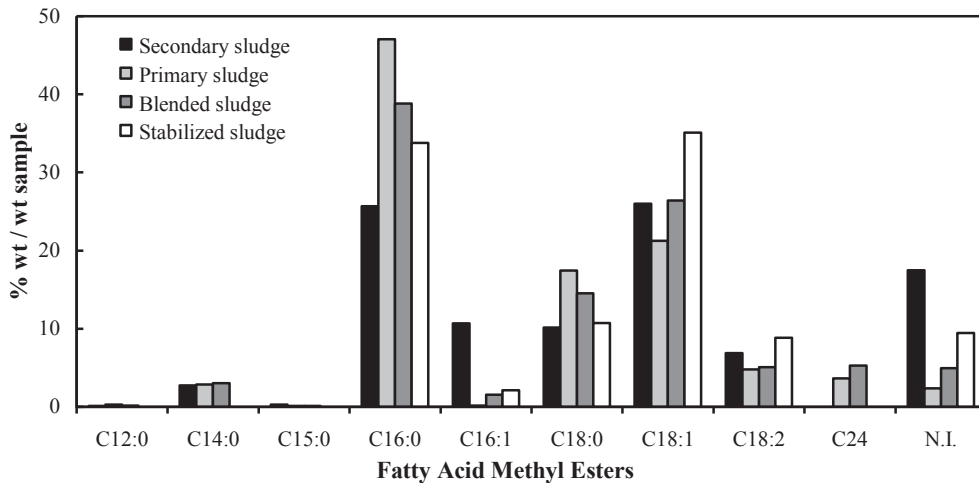


Fig. 2. FAME composition of biodiesel obtained from four type of sludge.

Profile comparison between the primary, secondary and blended sludge indicates that there is no substantial difference in their fatty acids composition. The slight difference can be observed in the appearance of around 11% of palmitoleic acid (C16:1) in the secondary sludge as compared to 0.2% in the primary and 2% in blended sludge. Another difference can be seen in the mass percentages of palmitic acid (C16:0), around 26% for secondary sludge as compared to 47% and 39% in the primary and blended sludge respectively. Furthermore, there is no appearance of lignoceric acid (C24:0) in the fatty acids profile of secondary sludge.

Despite the slight differences in the fatty acids composition, at least 10 fatty acids were identified in the biodiesel from sludges, ranging from C10 (traces) to C18 for secondary sludge and from C10 (traces) to C24 for primary and blended sludges. These fatty acids are excellent in the production of biodiesel [6]. In addition, the European Standard EN 14103 method for the determination of composition and quantity of methyl esters in biodiesel is suitable for FAME containing methyl esters between C14 and C24 [18]. However other study showed that C12 could also be determined using EN 14103 method [19]. Thus, the fatty acid profile of biodiesel produced from sludge can be easily analyzed according to EN 14103 to determine all FAMES content.

3.3. Comparison of sludge fatty acid profiles with soybean and lard

Fatty acids composition of primary sludge was compared with common biodiesel feedstocks; soybean as a vegetable oil and lard as an animal fat [20], as shown in Fig. 3. We can observe that majority of fatty acids found in the sludge are the same as fatty acids of soybean and lard; palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1), and linoleic acid (C18:2). However, the fatty acids profile of primary sludge is more related to that of lard, concluding that most of the lipids of sludge come from animal fats.

The notable difference is observed in the high amount of linoleic acid (C18:2) and the presence of linolenic acid (C18:3) in the biodiesel from vegetables. These polyunsaturated fatty acids can undergo reactions such as autoxidation due to the bis-allylic position of the carbon double bonds, provoking a destabilisation of the biodiesel [21]. The oxidation stability is one of the biodiesel properties which is standardised and need to meet the specifications of EN 14214 [22].

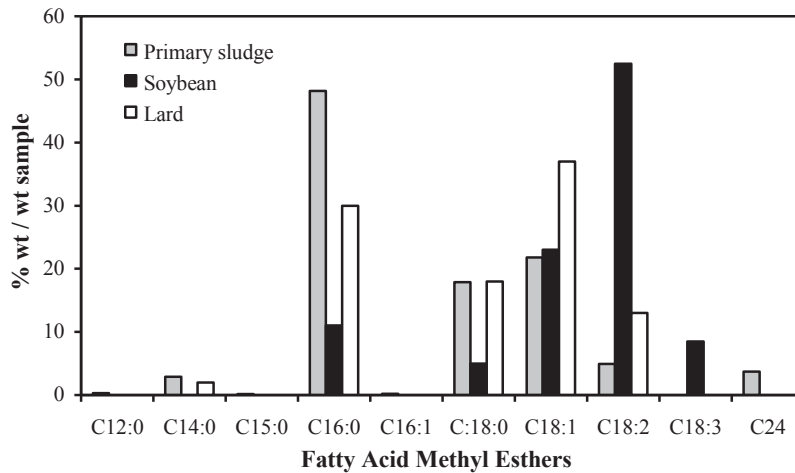


Fig. 3. Fatty acid profile comparison of primary sludge to soybean and lard.

However, it is well known that it is very difficult to achieve the minimum limit of six hours for oxidation stability for biodiesel derived from common vegetable materials such as soybean, sunflower, grape, olive, palm and rapeseed oil [19]. The oxidation stability decreases with the increase of the content of polyunsaturated fatty acids. Therefore, vegetable oils rich in polyunsaturated fatty acids (linoleic acid and linolenic acid) tend to give biodiesel with poor oxidation stability. On the other hand, feedstocks rich in saturated or monounsaturated fatty acids give biodiesel with higher oxidation stability [19,20]. As the polyunsaturated fatty acids in the biodiesel from primary sludge constitute only 5% as compared to 61% in the biodiesel from soybean, the production of biodiesel using sewage sludge as a lipid feedstock will improve the oxidation stability of biodiesel.

On the other hand, the level of saturated fatty acids found in the sludge is 73%. It is much higher than the saturation level of lard and soybean, 50% and 16% respectively. The significant amounts of saturated fatty compounds may present a problem in cold flow properties of biodiesel, increasing the temperature at which a liquid biodiesel, when cooled, becomes cloudy due to formation of crystals and solidification of saturates [20,21]. Thus, biodiesel produced from vegetable oils achieves better cold flow properties than this from animal fats [20]. According to this, the biodiesel produced from sewage sludge with large amount of saturated fatty acids will give a problem in cold weather owing to gelling. However, the cold flow problem can be overcome by using branched chain alcohols instead of methanol in the reaction of transesterification [20,21,23].

3.4. Biodiesel potential of sewage sludge generated in wastewater treatment plant of Reus, Spain

The annual biodiesel potential of Reus municipal wastewater sludges is presented in Table 2. The municipal WWTP in Reus have a capacity to process near 25,000 m³ of wastewater per day of 200,000 population. Annually, the WWTP of Reus produces a total of 94,487 tons of primary and secondary sludges. Based on the present research, the estimation of lipids potential derived from primary and secondary sludge was 524 and 135 tons, respectively. The annual biodiesel potential was estimated at 288 ton from primary and 42 tons from secondary sludge, resulting on total 330 tons of biodiesel produced

from wastewater sludge in Reus. The most beneficial sludge for biodiesel production was primary sludge consisting 87% of the total biodiesel value among the two Reus sludge evaluated.

According to Corporation of Strategic Reserves of Oil-based Products (CORES, by its Spanish acronym), Spain consumed 26,000 tons of pure biodiesel in 2011 [24]. As a result of this, biodiesel production potential from wastewater treatment sludge of small Reus population may replace 1.3% of current biodiesel consumption in Spain. Thus, sewage sludge is a large potential feedstock for future biodiesel production. Moreover, biodiesel production from common vegetable feedstocks is not economically feasible because of high cost of these raw materials. The cost of biodiesel is currently 1.5-3 times higher than the petroleum diesel in developed country [25]. As sewage sludge is a waste produced in large quantities, reutilizing these wastes as a source for alternative energy can give financial and environmental benefits for biodiesel production. Additionally, the economic study of Mandola et al. estimated the price of the sludge biodiesel is lower than average price of diesel fuels and refined soy biodiesel [7]. Thus, the utilisation of sewage sludge as a lipid feedstock may reduce the price of biodiesel.

Table 2. Estimation of annual biodiesel production based on the sludge generated in Wastewater Treatment Plant of Reus, Spain (200,000 inhabitants).

Sludge type	Sludge generation (tons/year)		Lipids potential		Biodiesel potential	
	Total	Dry material	(tons/year)	%	(tons/year)	(%)
Primary sludge	49,275	2,070	524	25	288	14
Secondary sludge	45,212	1,447	135	9	42	3
Total	94,487	3,517	659	19	330	9

4. Conclusions

The results have showed that all types of municipal sludges produced during wastewater treatment are a potential source of suitable lipids for the production of biodiesel.

Gas chromatography analysis of the FAMES indicated a similarity between the fatty acids composition of the four sludges evaluated. All types of sludge have a significant amount of palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1) and linoleic acid (C18:2) which are essential for the production of biodiesel.

Higher amounts of lipids were obtained from primary (25.3%, on the basis of dry sludge) and blended sludges (21.9%) compared with stabilized (10.1%) and secondary sludges (9.1%). About biodiesel yields, primary sludge also reached the maximum biodiesel yield, 13.9% based on dry weight of sludge. Blended, secondary and stabilized sludge achieved 10.9%, 2.9% and 1% respectively. Thus, it is more beneficial to use primary sludge to produce biodiesel.

Estimation of annual biodiesel production based on the sludge generated in WWTP of Reus have showed that biodiesel from primary sludge consists of 87% of the total amount produced.

Comparison of primary sludge fatty acid profile with common biodiesel feedstocks has showed their suitability for the production of biodiesel. However, higher concentration of saturated fatty acids was found in the biodiesel from sludge and it could be a problem in cold countries. On the other hand, the higher saturated content in the biodiesel from sludge will produce biodiesel with better oxidation stability.

The results indicated that sewage sludge is a potential candidate as raw material for the production of biodiesel. Furthermore, sludge is a cheap and available raw material and, the production of biodiesel could reduce excess sludge.

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