Scale-up and economic analysis of biodiesel production from municipal primary sewage sludge

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Abstract

Municipal wastewater sludge is a promising lipid feedstock for biodiesel production, but the need to eliminate the high water content before lipid extraction is the main limitation for scaling up. This study evaluates the economic feasibility of biodiesel production directly from liquid primary sludge based on experimental data at laboratory scale. Computational tools were used for the modelling of the process scale-up and the different configurations of lipid extraction to optimise this step, as it is the most expensive. The operational variables with a major influence in the cost were the extraction time and the amount of solvent. The optimised extraction process had a break-even price of biodiesel of 1232 $/t, being economically competitive with the current cost of fossil diesel. The proposed biodiesel production process from waste sludge eliminates the expensive step of sludge drying, lowering the biodiesel price.

Keywords

Sewage sludge, Lipids, Biodiesel, Process modelling, Economic evaluation

1. Introduction

Biodiesel is one of the most promising renewable fuels that is biodegradable, less toxic, may generate similar amount of energy to fossil diesel and can be directly used with current engine and refuelling technology/infrastructure without major modification (Siddiquee and Rohani, 2011; Kwon et al., 2012; Atabani et al., 2012). Biodiesel, *i.e.*, fatty acids methyl ester (FAME), is mainly produced from edible vegetable oils, however the high cost of vegetable oils which constitutes between 70-85% of the overall biodiesel production cost, strongly influences the final price of this biofuel, limiting its expansion (Mondala et. al., 2009; Siddiquee and Rohani, 2011; Kwon et al., 2012; Atabani et al., 2012). Furthermore, the cultivation of edible oilseeds for biofuels raises the concerns of food shortage, which competes with fuel production (Atabani et al., 2012; Khan et al., 2014).

The possibility of using municipal sewage sludge as non-edible lipid feedstock is gaining more attention due to the large amounts of sludge generated in the developed countries, and high amount of lipids contained within these wastes, up to 30 wt% (Dufreche et al., 2007; Olkiewicz et al., 2014, 2015a; Tyagi and Lo, 2013; Yi et al., 2016). The amount of lipids strongly depends on the sludge type. The lipid yield in secondary sludge was found in the range of 2-12 wt% (Huynh et al., 2010; Siddiquee and Rohani, 2011; Tyagi and Lo, 2013; Olkiewicz et al., 2012, 2015a), while in primary sludge usually ranges between 15-30 wt% (Willson et al., 2010; Siddiquee and Rohani, 2011; Pastore et al., 2013; Olkiewicz et al., 2015a, 2015b; Yi et al., 2016).

On the other hand, the sludge formed during treatment of wastewater needs specific management before disposal and represents a major cost in wastewater treatment plant

(WWTP) operation (Dufreche et al., 2007; Pastore et al., 2013). Therefore, the sewage sludge can be envisaged as a low-cost, readily available in abundance and non-edible feedstock, which can make biodiesel production profitable. Recent studies have indicated that the lipid contained in sewage sludge could be a potential feedstock for biodiesel production (Dufreche et al., 2007; Mondala et al., 2009; Huynh et al., 2010; Willson et al., 2010; Siddiquee and Rohani, 2011; Kwon et al., 2012; Pastore et al., 2013; Olkiewicz et al., 2012, 2014, 2015a). Nevertheless, the cost of energy necessary to eliminate the high water content (95-98 wt %), before lipid extraction, is the main limitation to scale-up, as dewatering and drying constitutes more than 50% of the total biodiesel production cost (Dufreche et al., 2007; Mondala et al., 2009). On the other hand, previous research demonstrated the feasibility of lipid extraction from liquid sludge (~96% of water) by direct liquid–liquid extraction using hexane as a solvent (Olkiewicz et al., 2014). Since the production of biodiesel from liquid sewage sludge is feasible, the expensive sludge drying step can be eliminated, and therefore the overall biodiesel production cost can be reduced. However, in order to confirm the stated hypothesis, the economic feasibility of the wet process (direct use of liquid sludge) and its comparison with dry process (use of dry sludge) has to be done.

Economic analysis of the production of biodiesel from dry sewage sludge has already been reported. Dufreche et al. (2007) estimated the cost of biodiesel production from dry sludge by direct *in situ* transesterification, without the extraction step, to be 933 $/t. However, in this research short-cut economic methods were used without giving details about the cost of methods used. A more detailed breakdown of estimated costs also for *in situ* transesterification was calculated by Mondala et al. (2009), based on data published by others, *e.g.*, the cost of sludge drying was taken from Dufreche et al. (2007). They obtained a break-even price of biodiesel of 970 $/t. Pokoo-Aikins et al. (2010) presented a full economic feasibility study, based on process design and simulation, to choose the best option to produce

biodiesel from sludge, using two-step process: preliminary lipid extraction, evaluating four solvents (hexane, toluene, methanol and ethanol), and subsequent conversion of the lipids into biodiesel. The results indicated that hexane and toluene were cheaper solvents, giving 868 and 838 $/t of biodiesel, respectively. These excellent results were obtained considering that dry sludge was free of cost, charging the sludge drying to WWTPs. Certainly, if sludge drying were also taken into consideration, the final price of biodiesel would increase significantly.

In short, on the one hand, in the aforementioned economic studies, some assumptions were underestimated and in some cases not all process steps were considered for the estimation of the final biodiesel cost. Therefore, to fairly estimate the biodiesel production cost from sewage sludge, all assumptions must be taken with a constructive criticism and include realistic values. On the other hand, the biodiesel production from wastewater sludge has a promising future but it is still in research stage. Therefore, further large scale studies are required to realize the benefits of this new biotechnology.

The purpose of this research is to critically review the biodiesel production from sewage sludge using the know-how acquired at bench scale experimental work. Laboratory scale data obtained in our previous study (Olkiewicz et al., 2014), where the feasibility of lipids extraction directly from liquid sludge was demonstrated, is analysed by computational tools in order to carry out the scale-up of this novel biodiesel production process. In particular, the lipid extraction from liquid primary sludge is optimised by using computational tools to model the process performance and the economic evaluation of the process alternatives. Process options are envisaged to estimate a realistic scenario considering the technology currently available. Finally, the optimised biodiesel production process from liquid sludge is compared to the *in situ* and two-step processes using dry sludge (also simulated in this study) in order to decide on the most economically favourable process.

2. Materials and Methods

A production plant with a capacity of around 4000 t/year of biodiesel produced from primary sewage sludge is studied. The capacity of the facility will depend on the sewage sludge availability. In this sense, a nearby urban waste water treatment plant (WWTP) to feed 60 m^3/h of primary sewage sludge was considered. This set-up (Fig. 1) can eliminate the cost of transporting the sludge feedstock into the biodiesel production facility, which therefore was not taken into account in the economic study as well as the cost of raw sludge, which is a waste generated during treatment of wastewater. The proposed process aims to improve the biodiesel production from sewage sludge, *i.e.*, lipid extraction process, by the elimination of the energy intensive step of sludge dewatering and drying and also the elimination of the heating process during extraction. Particularly, the process developed is compared with those described in other works, whose main differences are: on the one hand, the use of sludge previously dehydrated, with the consequent increased costs of the raw material, that in some assessments seem to be understated or dismissed (Pokoo-Aikins et al., 2010; Zhang et al., 2013; Dufreche et al. 2007; Mondala et al., 2009); and on the other hand, the use of heating during extraction, which also increases the cost of the process (Pokoo-Aikins et al., 2010; Zhang et al., 2013; Dufreche et al. 2007); and finally, the conversion of all lipids into biodiesel (Zhang et al., 2013), since based on experimental studies approximately 70-85% of lipids can be converted into biodiesel (saponifiable lipids) (Pastore et al., 2013; Olkiewicz et al., 2014). The economic evaluation of the process and its potential alternatives is performed based on the previous results experimentally tested (Olkiewicz et al., 2014).

2.1. Approaches and assumptions

The presented study aims to put some light in the potential of the wet route that so far has been underrated with respect to dry routes. The hypothesis considers experimental results in

laboratory and also the pilot-scale experience with other lipid sources in water solutions of similar features (microalgae, vegetal oils, *etc.*). In this sense, although the economic analysis to probe the feasibility of the process is based on conceptual design (specifically in laboratory-scale experiments), it is in line and even more complete than previously mentioned studies about better-known dry options that can be found elsewhere in the literature. The simulation software supports the customization of unit processes allowing the implementation of specific complexities that the simulator cannot solve in a realistic way related with the sewage sludge properties. Two main objectives are pursued, on the one hand, obtaining preliminary profitability indicators of sewage sludge-based biodiesel processing through wet pathway; and on the other hand, comparing the wet route with dry alternatives (*in situ* and two-step processes using dry sludge) under the same basis, that are the same process simulation and economic modelling procedures.

2.1.1. Primary sludge

The calculations of the economic feasibility study were performed with the data of primary sludge collected from the municipal WWTP in Reus (Tarragona, Spain) with a capacity to process near 25,000 m^3 of wastewater per day, which serves 200,000 inhabitants. The WWTP of Reus produces an average of 135 m^3/day of primary sludge. For the calculations, the flow rate was approximated to 60 m^3/h to assimilate the production of a big town, as for example the WWTP near Barcelona, which serves approximately to 2 million inhabitants.

Fig. 1(a) shows a schematic diagram of the WWTP in Reus, illustrating the sludge generation in the WWTP facility. The primary sludge was sampled after partial gravity thickening. The composition of primary sludge used for the analysis in AspenHysys V8® simulator was determined as described by Olkiewicz et al. (2015b), and is shown in Table S1.

2.1.2 Lipid extraction from liquid primary sludge

149 The calculations of the economic feasibility study were performed using directly the
150 experimental values obtained in laboratory, with a bench scale experimental device, by our
151 research group (Olkiewicz et al., 2014). In the experimental procedure, the sequential liquid-
152 liquid extraction of lipid was conducted in a batch mixer-settler reactor at ambient
153 temperature, using hexane as a solvent, where nine consecutive extractions stages were
154 carried out. In the simulation of the process scale-up, the lipid extraction takes place in a
155 liquid-liquid extraction series of mixers, where hexane is used as solvent, but in a continuous
156 counter-current system, as depicted in Fig. 1(b). Thus, the main difference between the flow
157 diagram presented in Fig. 1(b) and previously presented by Olkiewicz et al. (2014) is the
158 extraction system.
159 A maximum of five mixers were modelled in the AspenHysys V8® simulator, as more than
160 90% of lipids were extracted after 5 consecutive extraction stages in the experimental study
161 (Olkiewicz et al., 2014). According to the experimental study, in order to extract all lipids
162 from liquid sludge, sludge acidification until pH 2, prior to extraction is required (Olkiewicz
163 et al., 2014). In order to minimize the addition of acid, sludge acidification to pH 4 was also
164 studied. The sludge was acidified to pH 2 and pH 4 by addition of concentrated HCl. Due to
165 the primary sludge pH varies between 5.8 and 6.5, according to experimental results, it was
166 assumed that the required concentration of HCl in liquid sludge is approximately 1.5% or 0.8
167 % v/v to attain the pH 2 and pH 4, respectively (data experimentally tested).
168 After sludge acidification, the lipid composition consists of 20% non-saponifiable and 80%
169 saponifiable lipids (convertible to biodiesel). As shown in Table S1, the saponifiable lipid
170 consists mainly of free fatty acids (FFAs) and traces of triglycerides (TG), suggesting that the
171 main reaction during the conversion of sludge lipids into biodiesel is esterification of FFAs.

Based on the laboratory experiments, six different process configurations (Table 1) were selected to perform the comparative economic study with the aim to optimise the number of extraction stages and extraction conditions, to find the most economically favourable process.

2.1.3. Biodiesel production

Due to the high content of FFAs in the sludge lipids, the acid catalysis esterification/transesterification was selected to convert saponifiable lipids into biodiesel. Acid catalyst, *i.e.*, H_2SO_4, is used in the simultaneous esterification of FFAs and transesterification of glycerides avoiding soap formation, which takes place in the case of conventional alkali catalyst (*e.g.*, NaOH). The assumptions applied in the esterification/transesterification reaction were taken from Zhang et al. (2013), modifying the temperature of the reaction.

According to Zhang et al. (2013), the sludge lipids conversion is considered as 99% under the following conditions: 6:1 methanol to lipids molar ratio, 1% (v/v) of H_2SO_4 as catalyst in methanol, temperature reaction of 50ºC and 4 hours of residence time. However, the laboratory experimental test applied to the primary sludge lipids, using these conditions but taking into account the correction for saponifiable lipids (*i.e.*, 6:1 methanol to saponifiable lipids molar ratio), gave only 88% of reaction efficiency. An increase in the temperature to 60 ºC, showed an increase in the reaction efficiency to approximately 99%. According to the experimental results, the reaction was simulated at 60 ºC.

As only the saponifiable part of sludge's lipids is convertible into biodiesel, the separation of non-saponifiable lipids was performed after biodiesel production, in the purification step, as commented on in subsection 2.2.2 of this paper.

2.2. Process simulation

196 The economic characterization approach is based on the steady-state simulation of the
197 plant in AspenHysys V8®, in a continuous process. Although most of the chemical
198 components involved in the simulation are defined in the AspenHysys® component library,
199 some compounds have been defined as hypothetical solids, as potassium sulphate, the
200 inorganic matter or ash content in the sludge and the organic matter different from lipids, for
201 which average molecular weights and mass densities are defined. Moreover, certain
202 compounds are used in representation of similar substances. More precisely, a mixture of M-
203 palmitate and M-oleate was selected to represent the biodiesel product, the palmitic acid
204 represents the FFA content, the triolein plays the role of triglycerides and a saturated fatty
205 acid ester was selected to represent the non-saponifiable lipids (*i.e.*, cetyl palmitate). The
206 palmitic acid was selected due to the predominance of this acid in the sludge lipids and thus in
207 the biodiesel produced (Olkiewicz et al., 2014, 2015a). The primary sewage sludge (and its
208 lipids composition) was simulated according to the sludge characterization presented in Table
209 S1. The Peng-Robinson Soave (PRSV) equation of state was the fluid package selected to
210 predict the physicochemical properties of the chemical components involved, including
211 solutions such as 95% concentrated sulfuric acid. The Peng-Robinson (Peng and Robinson,
212 1976) versions have been the most successful in vapor-liquid equilibria calculations of
213 conventional as well as non-conventional mixtures of fluids (Ghosh, 1999). The selection of
214 the PRSV method takes into account that extends the original PR method for moderately non-
215 ideal systems and because it performs rigorous separation for aqueous systems containing
216 methanol, glycols plus hydrocarbons in second liquid phases. These features have been
217 probed adequate for the stages of reaction, separation and purification. Since the simulation
218 software was not able to reproduce accurately the performance of the lipids extraction phase,
219 user customized units were used to model this particular stage due to the presence of sewage
220 sludge solution, as it is explained in subsection 2.2.1.

The detailed flow diagram obtained directly with AspenHysys® simulation package, and the characterization of the main material streams involved can be seen in Fig. 2. The detailed list of main equipments used in the AspenHysys® simulation model is presented in Table 2. The simulation is based on the current available technologies. The process simulation is structured in two main sections: the lipid extraction from the liquid (96% water content) primary sludge and the acid-catalyzed esterification/transesterification process. In the following sections a detailed description of both process sections is explained.

2.2.1. Lipid extraction from liquid primary sludge

The primary sludge is stabilized with acid before the extraction (V-100), as commented on in subsection 2.1.2. After that, the lipid extraction takes place in a liquid-liquid extraction series of mixers (CSTR-100, 101), where hexane is used as solvent in a counter-current system. The equilibrium and operating data were taken from the experiments carried out by Olkiewicz et al. (2014). In order to find the number of stages that optimize the economic results, different alternatives are modelled regarding the working pH for the sludge, residence time during the extraction stage and the ratio between sludge and hexane, depending on the ratio of hexane used (Table 1). The separation units are totally controlled by the model programmed in a custom-fitting spreadsheet, building tailored units instead of using predefined operations from the simulator toolbox. The model imports the variables of mass flows and composition of the inputs (sludge and solvent) and exports to the separation units the values of splits and separation efficiencies. These output variables are calculated through mathematical expressions of the material balances and equilibrium equations of the extraction based on the adjustment of the experimental results in the corresponding conditions (solvent ratio, mixing time and pH). The design of the extraction is based on a short-cut method comparable to the McCabe-Thiele stepwise calculation for distillation columns (Perry et al., 1999). A constant flow rate of feed solvent and extraction solvent is assumed, and the solute

concentrations are given as the weight ratios of solute to feed solvent and extraction solvent in the raffinate and the extract phases, respectively. The compositions (raffinate and extract) of each extraction stage (operation line) are calculated with the equilibrium curve equation obtained by the fit of experimental data. The curve fitting equations and their coefficients of determination for each extraction configuration are presented in Table 1.

The raffinate composed by the rest of biomass and more than 96% water can be recycled to the WWTP, to be used as a substrate to produce biogas by anaerobic digestion (the process widely implemented in municipal WWTPs, presented in Fig. 1(a)), avoiding the generation of a new waste sludge. It has been already demonstrated that the residual lipid extracted sludge can be easily anaerobically digested, producing biogas which maintains a similar composition, *i.e.*, methane content, to that coming from raw excess sludge (Olkiewicz et al., 2014).

On the other hand, the extract is led to an equilibrium-flash separator V-101 where over 99% of hexane is recovered and recycled to the extraction.

2.2.2. Biodiesel production

Acid catalyzed reaction system is proposed for the production of FAME using methanol as reactant, where two reactions take place (CRV-100): the acid esterification of the FFA to give FAME and water, and the acid transesterification of the triglycerides to obtain FAME and glycerol. Based on experimental results, near 99% is achieved for the sludge's lipids esterification/transesterification under the conditions described in subsection 2.1.3.

The products stream is forwarded to a decanter (V-104) where the contact with washing water forces the separation of two-phases. The light phase is conducted to a flash separation (V-107) to reduce further the amount of hexane and traces of methanol that accompanied the obtained biodiesel. The heavy phase includes water, methanol, a low quantity of glycerol as by-product of the transesterification reaction, and the acid used as catalyst. To recover the methanol for its recycling as excess reagent in the esterification/transesterification reaction,

the heavy phase is first neutralized (CRV-102) by the addition of potassium hydroxide obtaining a salt, *i.e.*, potassium sulphate, that is removed (X-101) and that may be considered as a valuable by-product. Then, the neutralized stream is forwarded to a distillation column (T-102) to recover the methanol (79% of the total used in the process) and to obtain a stream of water with traces of glycerol (0.4 wt %) that might be recycled to the WWTP. As the obtained biodiesel contains also non-saponifiable lipids, a crystallization fractionation is applied to split biodiesel into a liquid (low-melting point) and a solid fraction with high melting point, *i.e.*, sterols and/or waxes, achieving a product of more than 98% of FAME. Particularly, the traditional fractionation consists of two stages, the crystallization under strictly controlled cooling rate combined with gentle agitation, and the separation by filtration (Knothe et al., 2005). This process was simulated by units V-106 and X-102 (see Fig. 2 and Table 2), to coarsely estimate the costs derived from the energy and equipment requirements.

On the other hand, during the design of the process, energy integration strategies were applied in order to reduce the energy consumption in certain stages of the process that were specially energy consuming. For example, during the hexane recovery and the product purification the streams that leave the separation units at high temperature are used to exchange heat with the input streams so the heating and cooling requirements are reduced.

2.3. Dry routes alternatives

The wet extraction route, optimised in this study, is compared with the dry route extraction where the sewage sludge is previously dried. The sewage sludge drying process was modelled in a similar way to that used for microalgae biomass harvesting, which consists in a two-staged dewatering process: centrifugation from a concentration of 5 to 20% of solids, and drying in a spray dryer till 95% of solids (Lassing et al., 2008). Two dry routes are considered in the economic comparison. On the one hand, the conventional (two-step) dry route, based on

operating data given by Zhang et al. (2013), uses hexane in a ratio of 10 L/kg dry sludge in a mixture with methanol and acetone 3:1:1, achieving 96% of extraction efficiency at 50ºC and a residence time of 1 hour. After the solvent recovery, the esterification/transesterification reaction is carried out using the same approach as in the wet route process. On the other hand, a dry route alternative is also assessed where the extraction and the acid catalysed reaction take place simultaneously, called *in situ* transesterification. This alternative was simulated using the operation data detailed by Mondala et al. (2009) including a mixture of 12:1:3.3 methanol:sludge:hexane mass ratio and 5% (v/v) of H_2SO_4 as catalyst in methanol. This alternative reduces the number of equipment involved and eventually the reduction of the costs associated is foreseen. The solvent recovery and product purification phases for both dry extraction alternatives are similar to that described for the wet route, with the only difference of impossibility of by-product formation during the *in situ* process. In this way, simulating the three alternatives (wet rout, two-step dry rout and *in situ* dry route) under common operational basis, *i.e.*, dry sludge composition, drying expenses, economic parameters, diesel specifications, etc., the results obtained are valuable especially in terms of comparison.

The detailed process flow diagrams obtained with AspenHysys® simulation package, and the characterization of the main material streams involved are presented in Fig. S1 and Fig. S2, for the conventional (two-step) and *in situ* dry routes, respectively.

2.4. Automated economic evaluation

The characterization of the process presented in this work is performed by the automated environmental evaluation tool (AEET) programmed in Matlab® R2010b (Torres et al., 2013a) taking advantage of the Component Object Module (COM) automation server capabilities to connect the simulator and Matlab®. In this tool the calculations are grouped into different modules, more precisely, it includes an inventory module that retrieves data from the process

simulation related with the inputs and outputs of materials and energy in the steady-state, while the economic module computes the capital investment, operating costs and profitability indicators. This tool was adapted to the presented case study; the particular complexity lay on the necessity of analysing a high number of alternatives as result of altering operating variables (solvent ratios, pH, residence time, etc.) and, in a greater extend, changes in topological variables, such as the number of extraction stages and plant configurations (one wet and two dry routes). The code of the module corresponding to the initialization of these parameters in the simulation, called the specifications module, includes the identification of the simulation file with the topological configuration to be assessed and the definition the set of variables and constraints that the simulation should meet. Besides, the modules for the calculation of the capital investment and manufacturing costs were complemented by a specific cost estimation procedure for the equipment in the extraction stages. The automation of the procedure makes the AEET a powerful tool for the evaluation of any process by the acquisition of realistic data from the simulation case. Besides, it allows performing additional analysis, emphasizing among others, generation and discrimination of alternatives (Torres et al., 2013b), retrofit and sensitivity analysis and coupling with external optimization algorithms (Torres et al., 2013a).

The profitability analysis module includes the calculation of the net present value (NPV) and the discounted payback period. However, taking into account that the purpose of this study is to determine whether the primary sludge can be a feasible feedstock for biodiesel production, the break-even price (BEP) is computed because it allows an easy comparison with the biodiesel main competitor, *i.e.,* fossil diesel. The calculations of the capital and production costs are based on Spain/European Union conditions (6% rate of interest for the capital investment and a plant life span of 20 years were assumed).

The total capital investment includes the fixed capital cost and the working capital cost, where the second is usually a fraction of the first (15% is used in this work). The fixed capital cost consists of the total bare module capital cost, the contingencies and fees and the auxiliary facilities cost. In this work, the equipment module costing technique is used to estimate the total bare module capital cost of the plant. This technique relates all costs back to the purchase cost of equipment evaluated for some base conditions that imply that the units are fabricated from the carbon steel and operated at near-ambient pressure. The deviation from these base conditions are handled using multiplying factors that depend on the equipment type, the system pressure and the materials of construction. Since cost estimation is a very specialized subject the complexities of a detailed profitability study are out from the scope of this study. For this reason, elements as pressure drops and potential safety issues are not itemized in the procedure. The analysis is based on the conceptual design of the process alternatives to compute an estimate of investment required and the rough cost of production in order to test the feasibility of the proposed process (wet route), optimize the design and decide between process alternatives.

The total manufacturing cost includes three different items: direct manufacturing cost (*i.e.*, raw materials, labour fees, utilities, maintenance and repairs, operating supplies, laboratory charges and patents and royalties), fixed manufacturing cost (*i.e.*, overheads, packaging, storage, local taxes, insurances and depreciation), and general expenses (*i.e.*, administration, distribution and selling, and research and development). Detailed information about the equipment module costing technique used to estimate the total bare module of the plant can be found in the literature (Turton et al., 2003; Torres et al., 2013c). For more detailed information, the prices of all raw materials, utilities used and by-product are listed in Table S2.

3. Results and discussion

The results of cost-effectiveness of the production of biodiesel, directly linked to the extraction of lipids are discussed in this section. Initially, the results of the production of biodiesel obtained from the different extraction configurations are evaluated (Section 3.1). Then, the economic results of the six extraction alternatives studied are discussed (Section 3.2). After that, the details of the best configuration are presented (Section 3.3). Finally, a comparison with production of biodiesel from dried sewage sludge, *i.e.*, conventional dry route and *in situ* dry route, and BEPs comparison are debated (Section 3.4 and 3.5).

3.1. Effect of the configurations of lipid extraction on biodiesel production

The extraction of lipids from sewage sludge is the most important point, as the reaction of esterification/transesterification is not optimised and its procedure is fixed (see subsection 2.1.3 and 2.2.2). The optimisation of the extraction was performed for six different configurations, presented in Table 1. These configurations were selected as the best alternatives regarding the experimental results obtained in laboratory work (Olkiewicz et al., 2014).

Fig. 3a presents the annual values of biodiesel production obtained for the six configurations depending on the number of mixers used for the lipid extraction. As it can be seen all configurations present the same behaviour, the production of biodiesel grows with the number of mixers used during the extraction stages. The optimisation was limited to 5 mixers as the costs of the extraction process is directly related to the number of mixers and, the increase in the quantity of produced biodiesel by the last units is much smaller in comparison with the increase of the costs (law of diminishing returns). As stated in subsection 2.1.2., more than 90% of lipids were extracted after 5 consecutive extraction steps in the laboratory work.

As shown in Fig. 3a, the highest productions of biodiesel were attained with 5 mixers for the configurations CS1, CS2 and CS3, approximately 4600 tonnes of biodiesel per year. Slightly lower amount, approximately 4400 of biodiesel per year, was achieved by configuration CS6. Comparing the extraction configuration CS3 with CS6, where the only difference is the sludge pH, it can be concluded that the increase of pH from 2 to 4 does not have significant impact on the biodiesel production. Additionally, the use of less acid in the process will reduce the biodiesel production cost (discussed in further detail in section 3.2). The lowest results were obtained in the case of CS4 and CS5, approximately 3600 and 3300 tonnes of biodiesel per year, respectively. These two configurations have a mixing time of only 20 min, suggesting that the extraction time seems to be the most important parameter. The lower the extraction time, the lower the yield of lipid extracted and, therefore the lower the amount of biodiesel produced. However, in the case of CS1, also 20 min of mixing, the time is compensated by the higher amount of solvent, ratio sludge/hexane of 1/2. This configuration is able to extract high quantities of lipids, but by using a high quantity of solvent, which is not economically favourable for the process (discussed in further detail in section 3.2). The detailed values of biodiesel production for each configuration in each extraction stage are presented in Table S3 and discussed in the following section.

3.2. Optimisation of lipid extraction configuration

In order to find the most profitable extraction conditions in biodiesel production from liquid primary sludge, the value of break-even price (BEP), for all configurations tested at each number of mixers was evaluated. The BEP is defined as:

$$BEP(\$/t) = \frac{\text{Manufacturing costs}(\$/y) - \text{Byproducts sales}(\$/y)}{\text{Production of biodiesel}(t/y)} \quad (1)$$

Where the manufacturing costs is the total manufacturing cost as defined in section 2.4; by-products sales are the revenue from the sale of by-products generated during the process (in

this case only one by-product is generated, *i.e.*, potassium sulfate; production of biodiesel in tonnes of biodiesel produced per year.

Fig. 3b presents the evolution of the break-even price for the six configurations studied as a function of the number of mixers used. The six configurations show exactly the same behaviour, the BEP decreases from one to two mixers, giving a minimum value for two mixers, and then increases constantly until the five mixers. The detailed values of break-even price as well as the investment and manufacturing cost for each configuration in each extraction stage are presented in Table S3. As shown in Table S3, independently of the configuration used, the total manufacturing costs increase faster in each stage than the production of biodiesel. The low increment of biodiesel production from 2 to 5 mixers is not compensated by the high increase in the manufacturing costs, resulting in continuous increase of BEP from 2 to 5 mixers (Fig. 3b). Furthermore, using more extraction stages (mixers) entails also the increase of total investment cost, which consequently prolongs the payback period, *i.e.,* time to achieve benefits by the plant. As shown in Fig. 3b, the results of BEP indicates that for all configurations tested, two extraction stages (mixers) are sufficient to extract enough lipids to make biodiesel production profitable. Although additional mixers increase the amount of lipids and, thus the amount of biodiesel in the process, the installation of more than two mixing equipment is not cost-effective.

As presented in Fig. 3b and in more detail in Table S3, the lowest BEP (1232 $/t) and therefore the best profitability was obtained by the configuration CS6 using two mixers. The detailed economic results for each configuration using two mixers are shown in Table S4. The best configuration, *i.e.*, CS6, works using two times less solvent than CS2 and CS4 and four times less solvent than CS1. Consequently, the cost of total manufacturing for the CS6 was lower, mainly due to the lower amount of solvent that has to be handled (and recovered, and recycled). Despite the fact that the biodiesel production was increased by 19% in the case of

CS1 and CS2 compared to CS6, the manufacturing cost increased by about 28% and 26% respectively, leading to higher BEP. In addition, the total investment cost related mainly to storage and separation was also lower in the case of CS6 than the CS1, CS2 and CS4. Although the cost of mixing equipment, in the case of CS1 and CS4 was lower than for the best configuration (CS6), due to the lower residence time which permits to use smaller equipment, the cost of other investments was much higher resulting in higher overall investment cost. Continuing with the comparison, the configuration CS5, where the only difference is lower residence time with respect to CS6, allows a reduction in total manufacturing cost of 12% and also in investment cost of 10%. However, the reduction of extraction time resulted in a significant decrease of biodiesel production by around 25%, giving therefore higher BEP than the best configuration (CS6). Looking at the configuration CS3, which has only lower pH than the configuration CS6, the investment cost was almost equal but the manufacturing cost was slightly increased by about 7% due to the higher amount of acid required. The increased of manufacturing cost for this configuration was not compensated by the insignificant rise of biodiesel production (≈ 4%), thus having higher BEP than CS6. It can be concluded that the optimized configuration, *i.e.*, CS6, does not only allow to minimize the biodiesel production cost, but also to reduce the solvent and acid use, making the process less detrimental for equipment and environment.

3.3. Details of the best configuration

The optimized extraction process (*i.e.*, configuration CS6 with two mixers) allows the plant to produce 3546 tonnes of biodiesel per year, with the minimum biodiesel cost of 1232 $/t. The biodiesel production plant is divided into four processes: extraction (lipid extraction from liquid sludge), recovery (recovery of lipids from solvent), reaction (biodiesel production), and purification (biodiesel separation, purification and catalyst neutralization). In

order to establish the cost requirement of each process in the whole biodiesel production plant, the detailed economic analysis was distributed and the results are presented in Table 3. Regarding the total investment cost, the extraction step is responsible for 54% of the total investment followed by purification (29%), recovery (10%) and reaction (7%). With respect to manufacturing cost, the extraction step is also the most expensive, representing 48% of the total, while purification, recovery and reaction account for 24%, 7% and 21%, respectively. This economic analysis of biodiesel production demonstrates that the proposed direct extraction from liquid sludge is the most cost intensive step for the investment as well as for the manufacturing of the process. Therefore, any improvement of the lipid extraction step would have a high impact on the final profitability of the process and finally on the biodiesel price. On the other hand, the purification step is the second expensive one due to the high amount of water used for the separation and purification of the final product. It is well known that in the conventional synthesis of biodiesel, using acid or basic catalyst, the separation and purification of final product from catalyst is difficult and required high amount of energy (Siddiquee et al., 2011; Andreani and Rocha, 2011). Thus, the improvement of the separation step by using better catalyst for the reaction, easy to separate and reuse, could also reduce the final cost of biodiesel production from liquid sludge. However, this implies the employment of new biodiesel manufacturing technologies based on *e.g.*, heterogeneous catalysis (Siddiquee et al., 2011) or ionic liquid catalyst (Andreani and Rocha, 2011), capable to overcome the problems related to conventional catalyst but that, to our knowledge, are not ready to be commercially used (or are not economically attractive).

3.4. Comparison with dry routes processes

In order to decide on the most economically favourable process, the economic data of the wet route *i.e.*, the optimized biodiesel production process with extraction configuration CS6,

was compared to conventional (two-step) dry route (extraction from dry sludge and consequent conversion of lipids into biodiesel) and to *in situ* dry route (direct production of biodiesel from dry sludge). The detailed process flow diagrams were modelled with AspenHysys® simulation package, and the characterization of the main material streams involved are presented in Fig. 2, Fig. S1 and Fig. S2 for wet, dry conventional and dry *in situ* routes, respectively.

As shown in Table 4 the wet route process is much more cost-effective than both dry routes. With respect to investment cost, both dry routes have a total investment cost around twice higher than the wet route. The wet route process requires large equipment size for lipid extraction step due to large volume of liquid sludge associated, and consequently with a higher mixing cost. However, the cost of dewatering and drying equipment in the dry routes is six times higher as compared to mixing equipment used in wet route, making the total investment cost of wet route more profitable. Regarding the manufacturing cost, dry processes are also more costly, with the total manufacturing cost twice higher in the case of dry conventional and approximately four times higher in the case of dry *in situ* (in both cases compared with the wet route). The much higher manufacturing cost is mainly related to the cost required for sludge drying which represents about 20% (334 $/t of biodiesel) and 10% (314 $/t of biodiesel) of the total biodiesel cost for dry conventional and dry *in situ*, respectively. As can be seen in Table 4, although the biodiesel production is higher in both dry routes, owing to more efficient extraction of lipid from dry sludge than from wet one, the much larger increase of manufacturing cost results in higher BEP as compared to wet process. Therefore, the proposed process of biodiesel production using directly liquid sludge is more cost-effective than the conventional dry or *in situ* dry routes, giving the break-even price of 1232 $/t , 1656 $/t and 3145 $/t, respectively.

519 Comparing the dry routes, the conventional two-step dry route is more profitable than the
520 *in situ* dry route. This result is in agreement with other authors who demonstrated that two-
521 step process is more energetically favourable than the one-step *in situ* (Zhang et al, 2013;
522 Pastore et al., 2013).
523 It is interesting to compare the BEP of biodiesel produced following the *in situ* approach in
524 this work (3145 $/t) with the BEP obtained by Mondala et al. (2009) (970 $/t), as they differ
525 widely. On the one hand, the cost of sludge drying used in the present study according to
526 Lassing et al. (2008) was higher than the cost presented by Mondala et al. (2009), who based
527 their calculation on the cost of sludge drying given by Dufreche et al. (2007). Both authors
528 did not described details about the method and equipment used for drying, presenting such a
529 favourable drying cost (2.1 $/t of wet sludge) as compared to other authors, 4.3 and 52.2 $/t of
530 wet sludge reported by Lassing et al. (2008) and Kwon et al. (2012), respectively. Therefore,
531 the sewage sludge drying process was modelled in a similar way to the totally defined
532 procedure by Lassing et al. (2008), increasing the BEP as compared to the BEP obtained by
533 Mondala et al. (2009) for the same process. On the other hand, the purification of the final
534 product from non-saponifiable lipids, which was performed in the present study, but was not
535 mentioned in other works (Dufreche et al., 2007; Mondala et al., 2009; Zhang et al., 2013)
536 also contributes to the cost raising. Furthermore, the costs of raw materials presented by
537 Mondala et al. (2009) are also lower than the presented here and should be included in order
538 to compare the approaches. Finally, regarding the *in situ* studies (Dufreche et al., 2007;
539 Mondala et al., 2009), only economic evaluation was performed, however the whole process
540 was not modelled and simulated. The simulation of process scale-up leads to get a full techno-
541 economic evaluation of the plant and finally more realistic cost of biodiesel is obtained.
542
543 **3.5. Break-even prices comparison**

The optimised BEP of biodiesel produced from liquid primary sludge was estimated to be 1232 $/t. This value is comparatively lower than the current price of fossil diesel in Europe, estimated to be 1376 $/t on January 2016 (IEA, 2016). Therefore, it is evident that the biodiesel obtained from municipal primary sludge has a high potential to economically compete with fossil diesel.

On the other hand, it is interesting to compare the BEP of biodiesel from sewage sludge to that from microalgae, the alternative biodiesel feedstock that has been investigated extensively for a very long time but whose industrialisation is not yet economically viable (Rios et al., 2013). The BEP of biodiesel produced from sewage sludge is lower than the obtained from microalgae in different studies; 1344 $/t calculated with the best scenario available (Lassing et al., 2008), 2953 $/t for a biodiesel with microalgae produced in open ponds (Davis et al., 2011), and 5700 $/t obtained in an exhaustive study (Ríos et al., 2013). As municipal sewage sludge is a waste, provided for free from WWTP, the cost of biomass production is eliminated; which is not the case in microalgae biodiesel manufacturing, where biomass production accounts for 65% of the overall cost (Ríos et al., 2013).

4. Conclusions

The detailed techno-economic study indicates that the proposed biodiesel production process from liquid primary sludge is economically feasible and more cost-effective than alternatives from dry sludge. The required biodiesel selling price for the optimised lipid extraction step was estimated to be 1232 $/t, which is lower than the current cost of fossil diesel and the cost of biodiesel from microalgae. Thus, the municipal sludge has a large potential as cost-competitive, plentiful and non-edible feedstock for biodiesel production. Additionally, further improvement of the proposed process (especially lipid extraction from liquid sludge and biodiesel purification) could lower the biodiesel price even more.

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661

Figure captions

Fig. 1. Scheme of the current WWTP of Reus (Tarragona, Spain) (a) and the biodiesel production process (b).

Fig. 2. Flow diagram of the AspenHysys® simulation model for the biodiesel production process from liquid primary sludge (wet route).

Fig. 3. Influence of number of stages in the extraction of lipids from liquid primary sewage sludge (a) on the biodiesel production and (b) on the break-even price.

Table 1. Configurations of the lipid extraction process.

Configuration	Sludge/hexane volume ratio	pH	Mixing time (min)	Fitting equation		R^2	
				Concentration		Concentration	
				Low	High	Low	High
CS1	1/2	2	20	y=0.2554x+7e-5	y=0.0001exp(1483.6x)	0.916	0.989
CS2	1/1	2	60	y=0.8968x-0.0001	y=0.0002exp(1302.3x)	0.967	0.990
CS3	2/1	2	60	y=3.9716x-0.0016		0.997	
CS4	1/1	4	20	y=7e-5exp(956.94x)		0.954	
CS5	2/1	4	20	y=-238.81x2+4.3902x-0.0046		0.980	
CS6	2/1	4	60	y=3.9716x-0.0016		0.997	

Table 2. List of the main equipments used in the AspenHysys® simulation model for the biodiesel production process from liquid primary sludge (Fig. 2)

Name	Equipment	Purpose
V-100	Tank	Reception of sludge and mixing with acid
CSTR-100	Mixing unit	Representation of time and mixing needed for extraction (1st stage)
X-103	Separation unit	Representation of the separation controlled by "Equilibrium" (1st stage)
CSTR-101	Mixing unit	Representation of time and mixing needed for extraction (2nd stage)
X-104	Separation unit	Representation of the separation controlled by "Equilibrium" (2nd stage)
V-101	Separator	Flash separation of solvent from lipids after extraction
V-102	Tank	Feed tank of hexane
MIX-102	Mixing unit	Collect hexane streams from the recovery equipment
V-103	Tank	Feed tank of methanol
MIX-101	Mixing unit	Mixing methanol with sulfuric acid to reach the appropriate reaction conditions (controlled by "Reaction inlet")
CRV-100	Reactor	Transesterification of lipids with methanol and sulfuric acid
V-104	3-phase separator	Separating light and aqueous phase before purification
CRV-102	Reactor	Neutralizing acid before recovering methanol from aqueous phase
X-101	Solid separator	Separating neutralization product
T-102	Distillation column	Recovery of methanol from the aqueous phase
V-107	Separator	Purification of light phase removing remaining methanol from FAME
V-106	Refrigeration tank	Adjusting temperature of product to achieve winterization
X-102	Solid separator	Removing heavy fractions (waxes) from FAME product

Table 3. Disaggregated results for the optimal configuration CS6[1] as a function of the process steps.

		Unit ($)				
	Item	Extraction	Recovery	Reaction	Purification	Total
Investment costs ($)	Total bare module	2270511	410244	316032	1229418	4226204
	Centrifugation and drying	0	0	0	0	0
	Reactors	0	0	129214	33843	163057
	Distillation columns	0	0	0	687314	687314
	Flush & other separation equipment	249299	63451	0	74147	386897
	Mixing units	942909	96465	9563	13670	1062606
	Heat exchangers	0	239677	0	227158	466835
	Pumps	26143	10652	4035	13823	54652
	Storage	1052160	0	173100	173340	1398600
	Fix. cap. costs (bare, cont. aux.)	3482963	629315	484793	1885927	6482997
	Working capital	522444	94397	72719	282889	972450
	Total investment costs	4005408	723712	557512	2168816	7455447
Manufacturing costs ($/y)	Raw materials	271648	0	313255	53435	638338
	Utilities	269715	44350	60	53497	367623
	Steam	0	44346	0	50944	95290
	Cooling	0	0	59	2378	2437
	Electricity	269715	5	1	1	269722
	Makeup water	0	0	0	174	174
	Fuel (spray dryer)	0	0	0	0	0
	Operation labour	270000	40500	155250	195750	661500
	Direct manufacturing cost	811363	84850	468565	302683	1667461
	Overhead (fix. & gen. exp. costs)	1312587	210534	440997	753629	2717746
	Total manufacturing costs	2123950	295384	909562	1056311	4385208

[1] ratio sludge/hexane 2/1, pH 4, 2 mixers and 60 min of mixing

Table 4. Comparison of optimal configuration CS6[1] (wet route) with dry routes.

	Item	Process ($)		
		CS6 (wet route)	Dry route (conventional)	Dry route (*in situ*)
Investment costs ($)	Total bare module	4226204	9780923	11290902
	Centrifugation and drying	0	6400000	6400000
	Reactors	163057	189218	1196930
	Distillation columns	687314	688299	316706
	Flush & other separation equipment	386897	384994	437067
	Mixing units	1062606	255744	339856
	Heat exchangers	466835	420554	265841
	Pumps	54652	43513	56962
	Storage	1398600	1398600	2277540
	Fix. cap. costs (bare, cont. aux.)	6482997	15003935	17320243
	Working capital	972450	2250590	2598036
	Total investment costs	7455447	17254525	19918280
Manufacturing costs ($/y)	Raw materials	638338	978234	3910752
	Utilities	367623	2617825	5263030
	Steam	95290	558431	3075539
	Cooling	2437	28423	156467
	Electricity	269722	92086	92363
	Makeup water	174	245	21
	Drying: Fuel (spray dryer) and Electricity (centrifuge)	0	1938641	1938641
	Operation labour	661500	717750	508500
	Direct manufacturing cost	1667461	4313809	9682283
	Overhead (fix. & gen. exp. costs)	2717746	5174616	6574965
	Total manufacturing costs	4385208	9488425	16257248
	Biodiesel production (t/y)	3546	4700	4425
	Break-even price (BEP) ($/t)	1232	2014	3674

[1] ratio sludge/hexane 2/1, pH 4, 2 mixers and 60 min of mixing

Fig. 1.

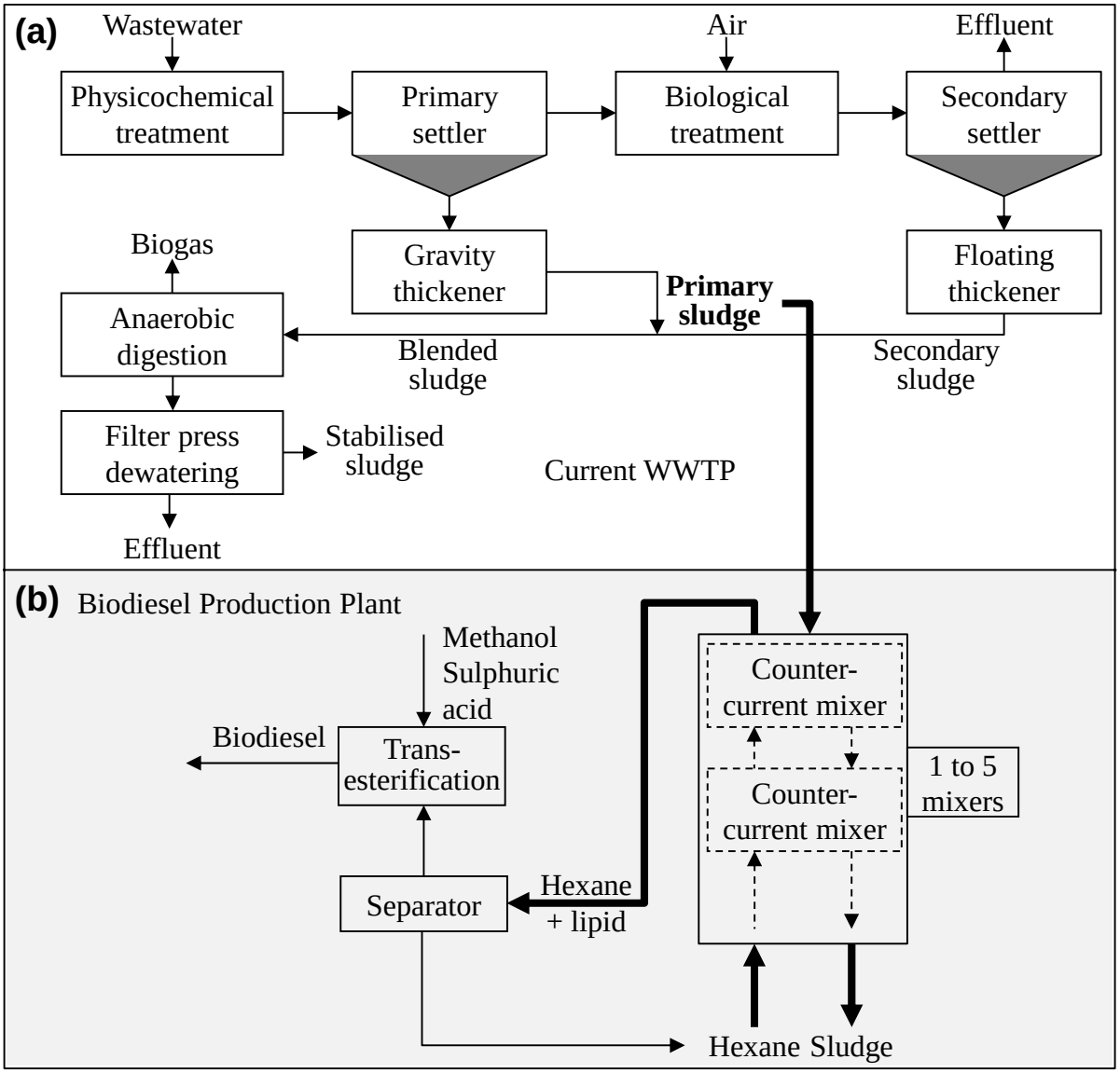
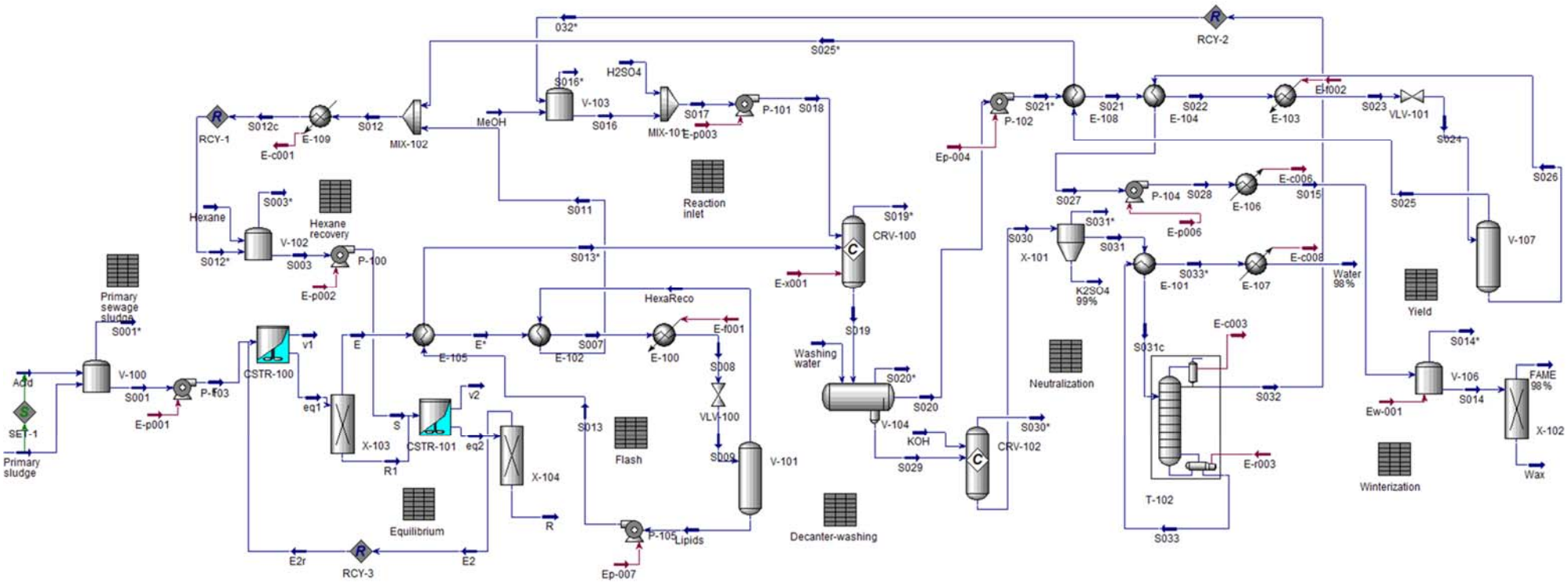

(a)
Wastewater
Air
Effluent
Physicochemical treatment
Primary settler
Biological treatment
Secondary settler
Biogas
Gravity thickener
Floating thickener
Primary sludge
Anaerobic digestion
Blended sludge
Secondary sludge
Filter press dewatering
Stabilised sludge
Current WWTP
Effluent
(b)
Biodiesel Production Plant
Methanol
Sulphuric acid
Biodiesel
Trans-esterification
Counter-current mixer
Counter-current mixer
1 to 5 mixers
Separator
Hexane + lipid
Hexane
Sludge

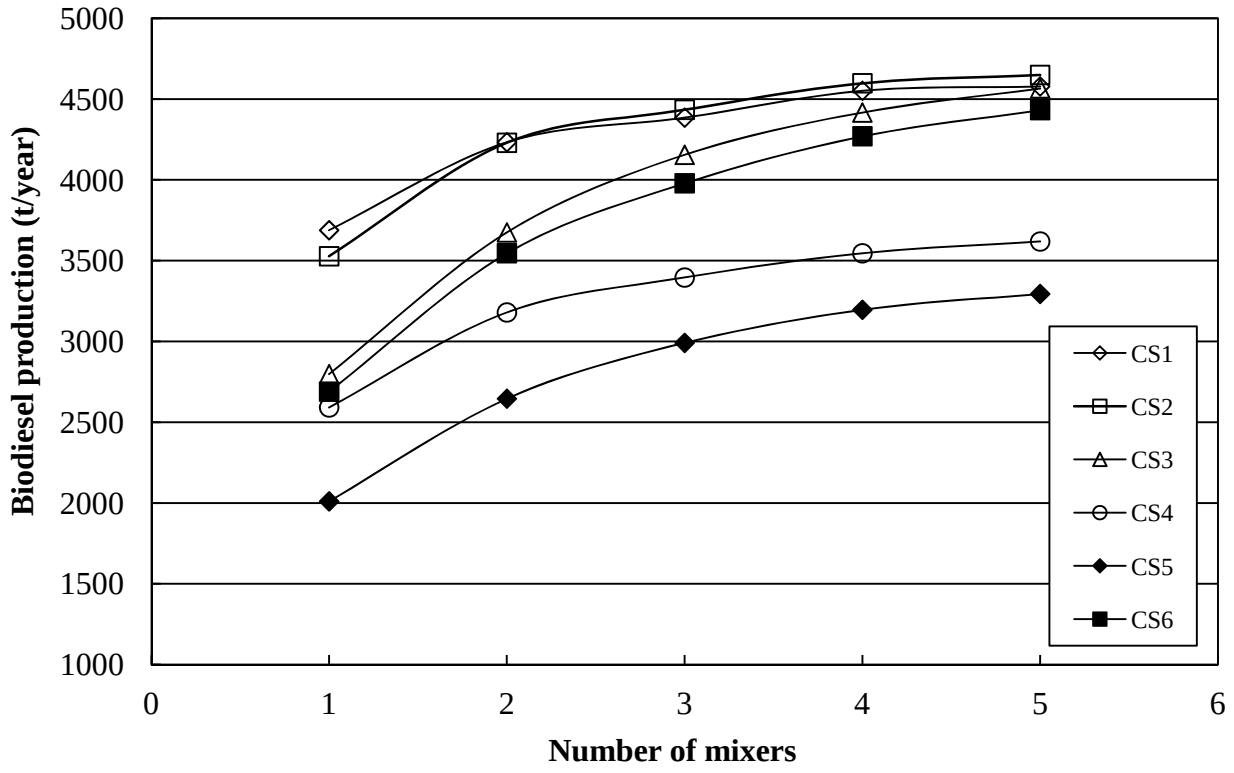
Fig. 2.



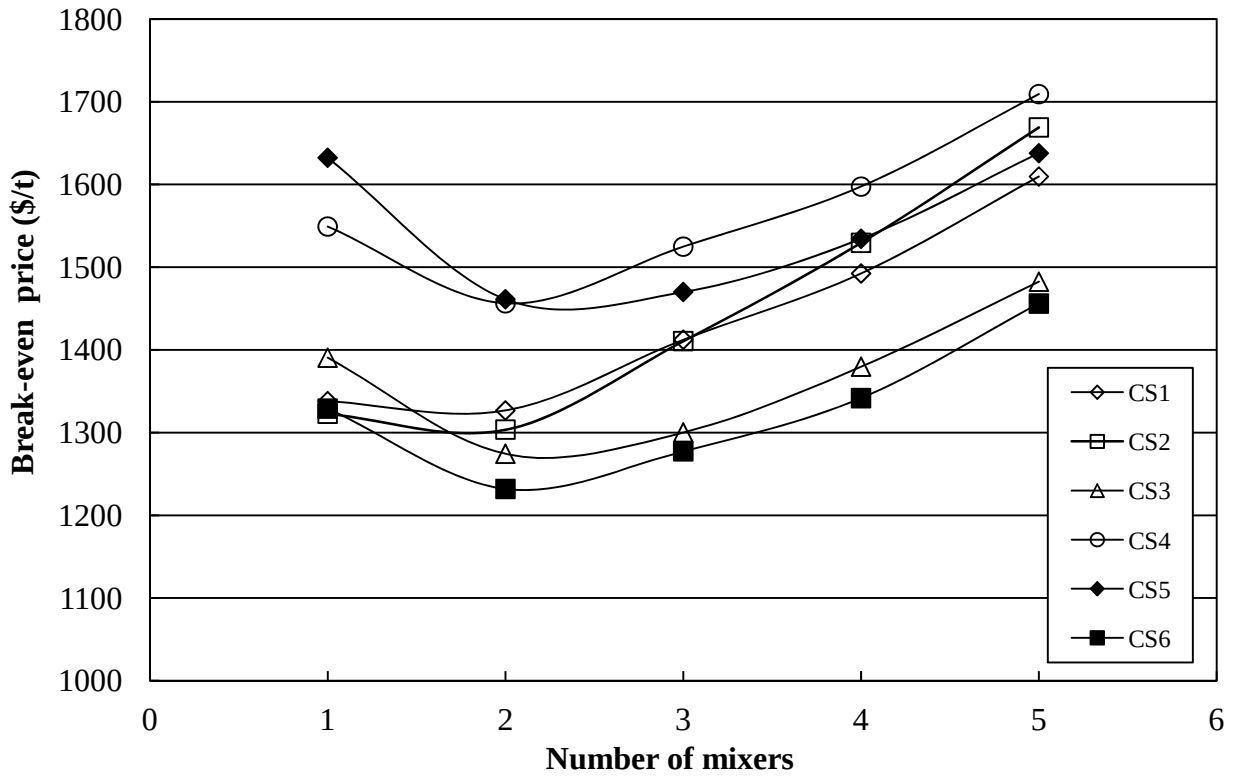
Stream name	Primary sludge	F	E	R	S	S012	S013	S015	S017	S019	S020	S025	S031	S032	FAME 98%	Water 98%	K_2SO_4 99%
Temperature (ºC)	25.0	24.9	25.9	31.7	60.6	61.8	89.3	35.0	59.6	60.0	61.3	168.9	41.4	69.7	5.0	35.0	41.4
Pressure (kPa)	101.3	115.0	105.0	105.0	120.0	80.0	101.3	101.0	101.0	101.0	101.0	80.0	101.0	101.0	101.3	110.0	101.0
Mass Flow (kg/h)	60000.0	60319.6	19925.3	59858.6	19464.9	19454.1	588.4	501.0	292.1	880.6	618.2	117.2	610.6	224.2	406.4	386.5	11.2
Components mass fractions																	
H_2O	0.9600	0.9549	0.0000	0.9623	0.0001	0.0001	0.0000	0.0001	0.0360	0.0409	0.0019	0.0098	0.6376	0.0466	0.0001	0.9804	0.0001
Palmitic acid	0.0084	0.0084	0.0183	0.0024	0.0000	0.0000	0.6167	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Triolein	0.0004	0.0003	0.0008	0.0001	0.0000	0.0000	0.0257	0.0003	0.0000	0.0002	0.0002	0.0000	0.0000	0.0000	0.0004	0.0000	0.0000
Rest Biomass	0.0205	0.0204	0.0000	0.0205	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Ash	0.0086	0.0085	0.0000	0.0086	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
Non-saponifiable lipids	0.0022	0.0022	0.0048	0.0006	0.0000	0.0000	0.1609	0.1887	0.0000	0.1075	0.1531	0.0010	0.0000	0.0000	0.0000	0.0000	0.0000
HCl	0.0000	0.0053	0.0000	0.0053	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
n-Hexane	0.0000	0.0000	0.9762	0.0000	0.9993	0.9993	0.1967	0.0218	0.0000	0.1314	0.1872	0.8943	0.0000	0.0000	0.0169	0.0000	0.0000
Methanol	0.0000	0.0000	0.0000	0.0001	0.0004	0.0004	0.0000	0.0008	0.9425	0.2593	0.0140	0.0702	0.3598	0.9534	0.0010	0.0156	0.0000
H_2SO_4	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0215	0.0071	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
M-Palmitate	0.0000	0.0000	0.0000	0.0000	0.0001	0.0001	0.0000	0.7582	0.0000	0.4346	0.6191	0.0244	0.0000	0.0000	0.9446	0.0000	0.0000
M-oleate	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0001	0.0301	0.0000	0.0171	0.0244	0.0003	0.0000	0.0000	0.0371	0.0000	0.0000
Glycerol	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0018	0.0000	0.0000	0.0026	0.0000	0.0000	0.0040	0.0000
KOH	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
K_2SO_4	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000	0.9999

Fig. 3.

(a)


Biodiesel production (t/year)
5000
4500
4000
3500
3000
2500
2000
1500
1000
0
1
2
3
4
5
6
Number of mixers
CS1
CS2
CS3
CS4
CS5
CS6

(b)


Break-even price ($/t)
1800
1700
1600
1500
1400
1300
1200
1100
1000
0
1
2
3
4
5
6
Number of mixers
CS1
CS2
CS3
CS4
CS5
CS6