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Research article

## Feedstock and process influence on biodiesel produced from waste sewage sludge

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### ABSTRACT

Disposal of sewage sludge is one of the most important issues in wastewater treatment throughout Europe, as EU sludge production, estimated at 9.5 million tons dry weight in 2005, is expected to approach 13 million tons in 2020. While sludge disposal costs may constitute 30–50% of the total operation costs of wastewater treatment processes, waste sewage sludge still contains resources that may be put to use, like nutrients and energy, that can be recovered through a variety of approaches. Research has shown that waste sewage sludge can be a valuable and very productive feedstock for biodiesel generation, containing lipids (the fats from which biofuels are extracted) in amounts that would require large areas cultivated with typical biodiesel feedstock, to produce, and at a much lower final cost. Several methods have been tested for the production of biodiesel from sewage sludge. To date, among the most efficient such process is pyrolysis, and in particular Microwave-Assisted Pyrolysis (MAP), under which process conditions are more favorable in energetic and economic terms. Sludge characteristics are very variable, depending on the characteristics of the wastewater-generating service area and on the wastewater treatment process itself. Each sludge can be considered a unique case, and as such experimental determination of the optimal biodiesel yields must be conducted on a case-by-case basis. In addition to biodiesel, other pyrolysis products can add to the energetic yield of the process (and not only). This paper discusses how feedstock properties and process characteristics may influence biodiesel (and other products) yield from pyrolytic (and in particular, MAP) processes, and discusses future possible technological developments.

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### 1. Introduction

Safe disposal of sewage sludge is one of the most important issues in the wastewater treatment cycle: at the European Union level, the 2005 sludge production was estimated in 9.5 million tons dry weight, up more than 54% in ten years, expected to reach 12.9 Mt by year 2020, a further increase of more than 30% (David et al., 2008). Sludge disposal costs may constitute up, and sometimes above 50% of the total cost of operation of an urban wastewater treatment plant, and include common disposal options like: landfilling, disposal in agriculture (about 40% EU-wide), incineration or co-incineration, use in industrial production of bricks, asphalts, concrete. Sewage sludge, however, still contains beneficial resources such as nutrients, that can be recovered through specific processes (e.g. precipitation as struvite) and energy, recoverable

through a variety of approaches.

On the other hand, to reduce fossil fuels consumption and carbon emissions, energy industry and government policies worldwide are promoting the use of biodiesel and other non-petroleum biofuels. The current European Union (EU) commitment under Directive 2003/30/EC on the promotion of biofuels for transport, sets a target of 5.75% of all transport fuels by 2010, increased to 10% by 2020 by the recent European Commission energy roadmap (Raboni et al., 2015). Based on life cycle analysis (LCA), it has been estimated that replacement of petroleum-derived diesel fuel with biodiesel would reduce greenhouse gas emission by up to 45% due to the carbon neutrality of the latter (Basha et al., 2009).

A “biofuel” is defined as a fuel that is produced through contemporary biological processes, rather than by geological ones, such as those involved in the formation of fossil fuels. An ongoing debate on biofuels sustainability has emerged since the biofuels industry gathered momentum. While 1st generation biofuels were (and are) mainly produced with feedstock with food value for

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humans or animals, 2nd generation biofuels are increasingly being produced from lignocellulosic and waste materials and could enable far greater reductions in greenhouse gases (GHG) emission (Capodaglio et al., 2016a). In 2009, biodiesel represented about 75% of total biofuels produced in the EU. If produced to comply with ASTM D6751 (the international standard of biofuels), it can be used for transportation and industrial purposes, and represents a valid alternative to conventional diesel because is renewable, biodegradable, less toxic, and has low emission profiles. With a similar energy density to diesel, it can be used directly without any vehicle or engine modification, and does not require a new type of refueling infrastructure. For these reasons, the production of biodiesel in the EU has increased from 3.6 billion liters in 2005 to 10.7 billion liters in 2010 (EDB, 2015; Thevenieau and Nicaud, 2013), initially from feedstocks such as soybean, canola, rapeseed, sunflower, palm, and coconut oils. Still, biodiesel production is currently limited due to high raw material costs, lack of agricultural lands and perceived conflict and impact on food prices. The search for cheaper feedstocks has turned towards various forms of waste, alternative raw materials, including waste animal fats, cooking oils (Bhatti et al., 2008; Hossain and Mekhled, 2010; Wallace et al., 2016) and lipids in sewage sludge (Dufreche et al., 2007; Zhang et al., 2014a). Kwon et al. (2012) showed that sewage sludge could produce significant amounts of lipids at low cost, with high yields of fatty acids methyl esters (FAMEs), which are the main ingredients of the fuel.

Traditionally, sewage sludge has been processed for decades in anaerobic tanks to produce biogas, a mixture of CH<sub>4</sub> and CO<sub>2</sub> that can be used as such or further refined to obtain bio-methane, a renewable fuel with characteristics practically identical to those of fossil methane (Zhang et al., 2014b; Capodaglio et al., 2016b). However, recent work has demonstrated that the production of biodiesel using the lipids extracted from sewage sludge could be economically feasible because of the remarkably high yield of oil and low cost of this feedstock (wastewater sludge can be considered “free”, except for transportation costs), as compared to conventional biodiesel feedstocks (Siddiquee and Rohani, 2011).

The purpose of the study, having already demonstrated the possibility of recovering biodiesel from waste sludge materials by microwave-assisted pyrolysis (MAP) process (Xie et al., 2014; Capodaglio et al., 2016c), is to attempt a more encompassing analysis of the process itself, in order to define its outcome limits and overall sustainability. In a previous experimental study (Capodaglio et al., 2016c), the authors concentrated on the sludge oil production potential by MAP, showing that under specific circumstances the process itself can be energetically positive, but contributions of generated syngas and char were totally neglected. These products can bring additional value to the process. Relationships between feedstock properties, process characteristics and products yields are further assessed in this work.

### 1.1. Waste urban sludge as a biodiesel feedstock

Feedstock selection is one of the most important aspects that should be taken into account, determining the economics of the entire biodiesel (and other resources) recovery process. As a general rule, in every production line, feedstock price should not be more than 50% of the production cost. Until recently, it was estimated that oils used as feedstock claimed about 80% of production costs: for this reason, many researchers believe that waste sludge has ideal characteristics for a basic biodiesel feedstock (Van Gerpen, 2004). Unlike other common feedstocks used for biodiesel production (food and non-food crops) which prices may experience severe fluctuations due to market conditions and competition with other uses, sludge is a waste product, abundantly produced in large quantities at an almost constant rate during the year,

uncompetitive with the food market, and readily available.

Currently, biodiesel diffusion is limited mostly by high raw material costs. Since waste sludge, generally, can be considered cost free (or even associated with a “gate fee” for its processing) and could be made by up to 30% (by weight) lipid fraction (Dufreche et al., 2007), which could then be processed into FAMEs, it could form an unexploited and almost endless source of cheap and readily available feedstock for biodiesel production.

Sewage Sludge contains a variety of organic and inorganic compounds originally contained in wastewater. The composition of sludge collected in primary sedimentation basins (primary sludge) consists to a high degree of undegraded organic matters. Secondary sludge, resulting from the interaction of microorganisms with organic matter in a process called activated sludge, exists normally in form of flocs, which besides living and dead biomass contain organic and mineral parts. Organic materials present in the waste water are detergents, pesticides, fats, oil and grease, colorings, solvents, phenols etc. Digested sludge is a mixture of primary and secondary sludge treated in an anoxic reactor to stabilize (reduce) its organic content, often with recovery of “biogas” (a mix of methane and CO<sub>2</sub> gases with other impurities). Less frequently, sludge can be stabilized aerobically, although this process is quite expensive and energetically disadvantageous. It has been claimed that the yield of FAMEs from primary sludge is greater than that of activated sludge. Lipid extraction from raw sludge would require large amounts of organic solvents in stirred and heated vessels. Dewatered sludge (solids content 10–20%) is denser and hinders lipid extraction, however, lipid extraction from dried sludge (solids content above 35%) is feasible, after adequate pretreatment (centrifugation and/or filtration processes) to obtain the proper humidity content.

Average characteristics and yields of different sludge types reported in literature are shown in Table 1. The wide lipid yields for primary sludges will depend on plant configuration, i.e. on mixing of recovered floating oil and grease with the primary sludge. Such configuration choices would have a significant effect on lipid yield.

### 1.2. Technologies for biodiesel production from waste sludges

Two main technologies are nowadays used for biodiesel production from wastewater sludge: lipid transesterification and pyrolysis. Several other technological processes have been studied and applied for biodiesel production from sludge and other feedstocks. Biodiesel consists of FAMEs, when methanol is the alcohol used in the transesterification process of lipids, or fatty acid alkyl esters (FAAEs) when produced via base-and/or-acid-catalyzed transesterification. In a typical process, triglycerides with low free fatty acid (FFA) (less than 0.5%) content are transesterified with methanol (MeOH) in the presence of alkaline homogeneous catalyst such as potassium hydroxide (Kwon et al., 2013).

Alternative approaches for dealing directly with dewatered sludge while minimizing the energy requirement of the entire process have been investigated: (i) direct methanolysis and (ii) preliminary extraction with hexane of the lipidic fraction and subsequent methanolysis into FAMEs (Pastore et al., 2013). Biodiesel yields from lipids extracted by different methods, i.e. acid hydrolysis, Soxhlet method, and water bath shaking method differ considerably: in a study by Zhu et al. (2014) they were 1.33, 6.73, and 4.92% (dry weight), with purity values, determined by GC–MS, of 97.5, 94.3, and 83.3%, respectively. Optimum production of biodiesel is faced with considerable challenges as lipids are extracted and then transesterified. In situ transesterification procedure have been developed where lipids are simultaneously extracted and transesterified, reducing reaction time and the amounts of solvent required, compared to the separate lipid extraction/

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