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ORIGINAL CONTRIBUTION

Pre-treatment and Characterization of Feed Stock for the Production of Biodiesel from Sewage Sludge - a Review

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ABSTRACT

Today, biodiesel can be a promising alternative of fossil fuels because of its noble properties, such as biodegradable, sustainable, non-toxic, etc. But the major problem associated with biodiesel is its production cost, especially the cost of feedstock. A cheap and non-consumable feedstock is highly recommended for lowering the expense of manufacturing and to ease up the competition with petroleum based fuel. Municipal sewage sludge may be a potential feedstock for the biodiesel production of biodiesel because of its exceptionally high oil yield and zero cost availability, in comparison to conventional biodiesel feedstock. It is abundant and comprises notable lipid concentration that can ensure to beneficially produce biodiesel from sewage sludge. This being an assured waste, released in ample amount while treating wastewater, the biomass production cost is eliminated.

Consequently, the sewage sludge can be expected to be an inexpensive, easily accessible and non-edible feedstock, which can manufacture biodiesel profitably. In addition, sludge usage as a source of lipid to synthesize biodiesel is also another means to make use of the excess amount of waste sludge released in waste water treatment plants (WWTPs). Extraction of lipids from sewage sludge happens to be the initial step while producing biodiesel from these wastes. Therefore, attaining the optimal condition of extracting lipids from sewage sludge is a crucial threat that may influence the economy of the entire process. Hence, the valorisation of sewage sludge in order to manufacture biodiesel is concentrated mainly on the extraction of lipids from such wastes and additionally on the synthesis of biodiesel from the extracted lipids. Before using sewage sludge as feedstock pre-treatment and characterization is necessary to make it suitable for the production of biodiesel. This review presents an overview of the pre-treatment and characterization of sewage sludge as a prominent feedstock to produce biodiesel. The aim is to give a clear idea about the commonly employed pre-treatment methods along with the essential parameters for characterising sewage sludge.

Keywords: Biodiesel, Feedstock, Sewage sludge, Lipids, Extraction, Wastewater, Non-edible.

1. INTRODUCTION

Sewage sludge generally contains large quantities of impurities. Before using the sewage sludge as feed stock, it should be pre-treated to remove undesired substances. Much attention has been paid to the pre-treatment technologies and their relevant studies since the late 1970s. The first applications of sludge pre-treatment were based on thermal processes. Those proved to be interesting alternatives to improve sludge de-waterability and anaerobic biodegradability. Other processes have been studied and proposed as alternatives (Haug, 1978).

There have been a growing number of publications on sludge pre-treatment methods since the mid-90s, which could be related to the need for sustainable sludge management methods in the wake of more restrictive legislation and bans on practices like ocean dumping. Sludge pre-treatment has become an important area of research as can be noted by the growing number of publications in recent years (Neumann, 2016). Pre-treatment is generally required for the sewage sludge before being used as a pure feedstock in the production of biodiesel.

Commercial and industrial facilities utilize pre-treatment to eliminate hazardous adulterants prior to discharging into a sewer system under the monitoring of a publicly owned treatment works (POTW). Conversion of SS for lipid is regarded by far as one of the finest planning in order to manage sludge. However, the prevailing drawback of lipid synthesis from sludge is the poor yield due to the reduced availability of easily consumed substances in sludge. Pre-treatments are useful means to decompose complex matter present in sludge as well as upgrade bioavailability of sludge. Pre-treating of SS helps in significantly accelerating the liberation of total nitrogen (TN), total phosphorus (TP) from solids and the soluble chemical oxygen demand (SCOD). Once pre-treated the sludge also achieves appreciable betterment in lipid yield and sludge minimization (Chen, 2020). Pre-treatment can be applied to primary, secondary or mixed sludge. Mostly this is oriented towards waste activated sludge (WAS) pre-treatment, followed by mixed and primary sludge. It involves single or combined physical, chemical and biological means to disrupt the flock structure of sludge and hydrolyse organic matter. This provides significant enhancements in terms of solids reduction, biogas production and digested sludge properties (Sperling, 2007). The importance of this pre-treatment can be realised by the fact that - by placing adequate controls and/or limits on the levels of certain contaminants in the sewage sludge (SS) will not only prevent interference with the concerned waste water treatment plant (WWTP) but also block the entry of pollutants that could pass through the WWTP untreated into the receiving container of SS. This in turn will improve opportunities for reuse or recycling of wastewater and SS.

In this paper we have focused on different techniques for the pre-treatment of sewage sludge before using it as a feed stock for biodiesel production. Along with pre-treatment characterization of the feedstock is also necessary to maintain the quality of the feedstock. Different methods of pre-treatment and characterization techniques are discussed here in details.

2. PRE-TREATMENT OF FEEDSTOCK

The sewage sludge selected as the feedstock undergoes different stages of pre-treatment to

yield biodiesel of required quality. Several pre-treatment techniques are employed to obtain the pure SS. Generally pre-treatment process include four steps, these are: (1) separation, (2) drying, (3) size reduction and (4) size separation. Under these main pre-treatment methods, there are a few variations or stages to carry out the required operation. Each of these stages has important roles to play during the pre-treatment of the feedstock.

2.1 Method of separation

Once the sewage sludge is collected from the municipal waste water treatment plant (WWTP), several separation processes is done to get rid of contaminants like sand, grit, paper, plastics and water, etc. Separation can be done by means of screening (Sondhi, 2020), sedimentation (Mondala, 2009), filtration (Qi, 2016) and centrifugation (Choi, 2019). The above separation processes are applied for the removal of unwanted materials from the sludge sample and make the sample ready for transesterification reaction.

2.1.1 Screening

This is an important separation method used in sewage sludge treatment. It eliminates objects like rags, paper, sticks, plastics and metals from effluent sewage sludge. If the large solids are not screened properly there is a chance of clogging or damage in pipes and downstream equipment.

2.1.2 Grit Removal

The screened sludge then allowed to flow through an aerated grit chamber that ensures the sedimentation of heavier inorganic materials, such as grit and sand by curtailing the sludge velocity.

2.1.3 Gravity Settling

This is a most popular thickening process. This provides another means to separate solids and liquids present in the sewage sludge flocks. Owing to gravity the solids settle down below and thus get separated from the liquid.

2.1.4 Filtration

Sludge filtration facilitates to filter out the suspended solids from the liquids using mechanical means.

2.1.5 Centrifugation

This is a high speed dewatering process where the solids get separated by means of size, density and viscosity from the sewage or wastewater liquids to produce a non-liquid material referred to as cake. Thus the volume of residuals (sample) can be reduced and improvement of further operations has been ensured. The force of a rotational cylindrical bowl is used to do the separation. This dewatering method can extract more water than other methods.

2.2 Moisture removal techniques

Once separation of sample from contaminants has been completed the sludge sample is sent to the dryer to cut down the amount of bound water. Here basically moisture is removed from the sludge with the help of a desiccator, or freezer or by thermal heating (Melero, 2015). Free water can be separated by mechanical dewatering process but to achieve the removal of bound water a drying process is necessary.

2.2.1 Thermal drying

Thermal sludge drying is a process where the dewatered sludge sample is dried using external heat to produce a moisture free sample. The dryer can be operated in a temperature regime of 65^o C (Choi, 2019) to 105^o C (Jazie, 2019). Sometimes, the bound moisture is removed by evaporation at room temperature. Drying time (24 hours to 90 hours) has also an important role in curtailing the amount of moisture present in the dewatered sludge sample.

2.2.2 Freeze-drying

Freeze drying is basically a dehydration process carried out at lower temperatures. It comprises product freezing, followed by pressure reduction and then ice removal via sublimation. Contrary to dehydration, which is a conventional process, this method does not evaporate water with the help of heat. Instead of thermal drying freezer can also be used for removal of moisture present in the dewatered sludge sample. The temperature can be maintained in the range of -20^o C (Kumar, 2016) to -18^o C (Mondala, 2009) for operating the freezer.

2.3 Size reduction method

After removing the moisture from sewage sludge it is important to reduce the size of these solid particles. Size reduction process enhances the reaction (trans-esterification) rate by improving the surface area per unit volume of the solid sludge sample. This can also help in

handling of the solid sample. The size of the solid sludge can be reduced (in the range of 0.5 to 1.0 mm) (Melero, 2015) by means of crushing (Melero, 2015) or grinding (Wu, 2016).

2.3.1 Crushing

It can be applied for the size reduction of dried sludge sample. Solid particles are disintegrated by two rigid forces. Crushing has been performed by using compression mechanism.

2.3.2 Grinding

This is an abrasive process by which solid blends are turning into a granular form. Impact is applied as the primary size reduction mechanism, where a single rigid force is hammered to obtain the desired size of solid sludge sample.

2.4 Size separation technique

Before using in the trans-esterification reaction, the crushed solids are to be separated according to their size. For this purpose sieve can be used widely. The ground sample is passed through a sieve of the required mesh to ensure uniformity in size of the fine powder of the sample. Homogenization is achieved by means of sieving separation (Hatami, 2019).

3. CHARACTERISATION OF FEEDSTOCK

It is important to understand the quality of feedstock (sewage sludge) after pre-treatment and before starting the trans-esterification process. It is necessary to check whether the desired parameter of the respective feedstock is suitable or not. During the trans-esterification process, tri-glycerides react with alcohol to yield FAME that is the end product. But if there are any traces of foreign undesired particles in the tri-glycerides, it may cause problem during the reaction and it will also retard the reaction rate. Therefore, characterisation is done after the pre-treatment of feedstock in order to control the limitation of certain parameters so that they do not exceed or fall short of the desired measurement. Some important parameters of sewage sludge are discussed here below.

3.1 pH

In the trans-esterification process, a rapid increase is followed by a gradual decrease in pH until a constant value is reached. A low pH indicates high conversion. Thus, maintenance of low pH of feedstock is required. Kumar et al.

(Kumar, 2016) have found the feedstock to contain a pH of 6.9 ± 0.2 .

3.2 Crude Fat

Crude fat is essential for bio-diesel yield because during trans-esterification process the lipid extraction ultimately results in more bio-diesel yield. Thus, more the amount of crude fat present in the feedstock, more will be the yield of bio-diesel obtained. In most reactions, the crude fat is around $16.1 \pm 0.1\%$ (Zhang, 2020).

3.3 Lipid Content

As stated before the bio diesel yield is totally dependent on lipid. This high lipid content is expected for better result. Hatami et al. (Hatami, 2019) have revealed this lipid value ranges from $21 \pm 1.05\%$. While Sondhi et al. (Sondhi, 2020) have found it to be around 30% and Wu et al. (Wu, 2016) confirmed the average lipid content is about 9.4% .

3.4 Moisture Content

Biodiesel has a tendency to absorb more moisture than petroleum diesel. High moisture content in biodiesel may lead to problems such as water accumulation and microbial growth in fuel handling, storage, and transportation equipment. Thus, we have to check the moisture content in the feedstock so that it would not create any problem to the end product. In most cases the moisture content is around $83.0 \pm 0.5\%$ (Zhang, 2020) or about less than 1% (Araza, 2016).

3.5 Total Organic Content

The next most highly recommended parameter is total organic content (TOC). TOC needs to be checked carefully as it results in pollution. The TOC in most cases is nearly about 32% (Kumar, 2016).

3.6 Ash Content

Ash has adverse impact on feedstock. It displaces valuable carbohydrate and decreases pre-treatment efficacy. The high proportion of ash in fuel reduces its higher heating value. Ash has negative effects on the heating surfaces as it increases their erosive wear and can cause the formation of slag and ash deposits. Studies confirm that the ash content varies from $17.8 \pm 0.2\%$ (Araza, 2016).

3.7 High Heating Value (HHV)

The HHV that is High Heating Value is an important property of biodiesel. It defines the energy content efficiency of fuels. Higher the value of HHV more will be the efficiency of biodiesel. HHV is a very good indication of the suitability of a proposed fuel, from the available energy standpoint. Zhang et al. (Zhang, 2020) have affirmed the HHV value is approximately 18.5 MJ kg^{-1} .

3.8 Volatile Matter

High volatile matter in feedstock is undesirable as it produces more tar, causing problems to the reaction. Due to high content of volatile matters sewage sludge can ignite at low temperatures. The volatile content usually varies from 65.3% (Wu, 2016) to $73.06 \pm 1.84 \text{ wt}\%$ (Hatami, 2019).

3.9 Acid Value

The acid value denotes the percentage of free fatty acids (FFA). High acid value result in corrosion and engine deposits in the fuel injectors. The acid value also plays a significant role in the quality control of feedstock. Generally the feedstock should have lesser acid value. Higher acid value lowers the biodiesel yield. The Acid value is found $102.8 \pm 1.8 \text{ mg KOH/g}$ (Qi, 2016).

3.10 Ultimate Analytical Characterisation

A fuel which has high oxygen content is most likely to cause an increase in combustion reaction rates. The presence of nitrogen and sulphur content in fuel may cause emissions of harmful gases. The nitrogen content is usually around $7.8 \pm 0.3 \text{ wt}\%$, the sulphur value is generally about $(0.8 \pm 0.2) \text{ wt}\%$ and the carbon content values are from $(39.4 \pm 0.7) \text{ wt}\%$, the hydrogen content is around $5.6 \pm 0.1 \text{ wt}\%$ (Araza, 2016).

3.11 Other Characterization

Other parameters that are to be checked are amount of crude proteins present in feedstock. Zhang et al. (Zhang, 2020) have revealed the value of crude protein is approximately $26.2 \pm 0.82\%$. Different techniques for the pre-treatment and characterization of feedstock are presented in Table 1 based on the reported articles.

Table 1: Different techniques of pre-treatment and characterization of feed stock as reported in the literature

Method of separation	Moisture removal technique	Size reduction method	Final size of particle	Parameter study	Ref.
Gravity settling	Vacuum filtration		40 mesh (0.425 mm)	Moisture content=less than 1%, Volatile matter=73.06 ± 1.84, Lipid fraction=21 ± 1.05%	Hatami,2019
Gravity settling		Mortar and pestle		Total organic matter=58%, Total organic carbon (TOC)=32%, Lipid content= 22.5%, pH - 6.9 ± 0.2	Kumar,2016
	Vacuum rotary evaporator	Vibration mill		Moisture content=83.3%, Average lipid content=9.4%	Wu,2016
Settling			0.5 to 1.0 mm		Melero, 2015
		Vibratory mill		Moisture content=83.0 ± 0.5%, VolatileSubstance=65.3 ± 0.6%, Crudefat=16.1 ± 0.1%, Crude protein=26.2 ± 8.2%, TOC=32.7 ± 0.2%	Zhang,2020
Centrifugation		Grinding		Lipid content=9-11% w/w dry matter	Chen, 2018

4. CONCLUSIONS

From overall literature review it is clear that pre-treatment is necessary for the sewage sludge before using it as feedstock for biodiesel production. In broad the pre-treatment process is having four steps;these are separation, drying, size reduction and size separation. There are different methods developed to carry out these four steps. In addition characterization of sewage sludge is

also an essential before using it as the feed stock. Characterization is the next step of pre-treatment. Different parameters, such as lipid, moisture content, ash content, crude fat, TOC, etc should tested for the sewage sludge to confirm the quality of the feedstock.

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