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RESEARCH-ARTICLE

Oil extraction from scum and ex situ transesterification to biodiesel

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ABSTRACT

The aim of this study is to investigate the extraction of oil from scum obtained from a municipal wastewater treatment plant. Various parameters such as solvent volume, temperature, agitation and moisture content were optimized to maximize the oil extraction efficiency using freeze-dried scum. The oil extraction efficiency was compared for each parameter using hexane and petroleum diesel as a solvent. The optimum physical parameters for oil extraction using freeze-dried scum were 75 g solids/L solvent, temperature 60 °C, agitation 300 rpm for 60 min, and maximum oil extraction efficiency of 100 and 94.3% w/w was obtained using hexane and petroleum diesel, respectively. The obtained results using 1 g of scum were further validated using 1 kg of wet scum under optimized conditions, and oil extraction efficiency of 94.1 ± 1.3 and 91.3 ± 4.2% w/w was obtained using hexane and petroleum diesel, respectively. Furthermore, ex situ transesterification was performed, and results showed that scum had a higher neutral lipid. In brief, scum is a potential feedstock for the production of biodiesel, and more work on this must be done in future.

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Introduction

Alternative technologies for biodiesel production are being improvised from the past 5 years of research. Biodiesel is one substitute for petroleum diesel (Ashnani et al., 2014). Biodiesel has been directly used for automobiles without any further engine modification, but in certain countries, biodiesel is blended with petroleum diesel at various levels (B2%, B3%, B4%, B5% and B10%) (Abedin et al., 2016). Consequently, with an increase in biodiesel utilization, there has been a decrease in greenhouse gas (GHG) emissions as less fossil fuel diesel has been consumed (Abedin et al., 2016).

Biodiesel is generally produced by transesterification of feedstock oil (plant oil, animal fats, cooking oil and other sources like microbial lipid) with alcohols in the presence of a catalyst (Gao et al., 2015). Current biodiesel production depends upon edible oil, and cannot be produced in quantities that can replace fossil fuels due to food versus fuel issues (Zhu et al., 2014). Further, this high-cost feedstock contributes up to 80% of biodiesel production costs (Moser et al., 2016). Biodiesel production using third-generation feedstocks (microbial lipids from microalgae, yeast and fungi) could be considered an effective alternative to replace edible-oil feedstocks due to the high lipid yield and abundance of industrial waste to be used as a carbon source for microbial lipid production. Various microorganisms such as *Cryptococcus*, *Chlorella*, *Yarrowia* and *Trichosporon* accumulate oil in the form of triacyl glycerides (TAG) (Bhatia et al., 2016). However, researchers are as yet unable to establish an industrially feasible technology for biodiesel production due to highly energy-intensive downstream processing as well as the use of cost-intensive organic solvents for oil extraction. Therefore, an alternative and low-cost feedstock is necessary for biodiesel production.

With the increase of metropolitan and cosmopolitan cities across Canada, there has been an accompanying increase in wastewater treatment plants. There are approximately 2303 municipal wastewater treatment facilities in the country, serving 36 million people (CCME 2014). In Canada, significant quantities of dewatered sludge solids are disposed of using landfills, incineration, underground disposal and lagoons. Management of the sludge solids causes environmental pollution and economic stress on wastewater treatment plants. Some studies have investigated sludge management by decreasing the contaminants and solids using a membrane reactor, ultraviolet rays, activated charcoal, etc. (Sepheri *et al.*, 2018; Johnson *et al.*, 2001). However, the cost of sludge management is one of the critical challenges for the wastewater industry. Therefore, an extensive approach is necessary to decrease the sludge management cost and GHG emissions (Demirbas et al., 2017).

In the wastewater treatment plant process, primary sludge is a result of the capture of suspended solids and organics in the primary treatment process through gravitational sedimentation, typically by a primary clarifier. The secondary treatment process uses microorganisms to consume the organic matter in the wastewater. The microorganisms feed on the biodegradable materials present in the wastewater in the aeration tank and then flow into a secondary clarifier where the biomass settles out and is removed as secondary sludge. Scum is a floatable material skimmed from the surface of primary and secondary settler tanks, especially from the surface of grit chambers in wastewater treatment plants. It is mainly composed of fats,

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Table 1. Comparison of solvent recycle using wet and dry scum with hexane or petroleum diesel as solvent.

	Recycle (no. of times)	Solvent	Solvent recovery % (v/v)	Oil extraction efficiency % (w/w)
Scum (dry)	0	Hexane	94.1	98.9
	1		87.4	94.2
	2		80.3	79.1
Scum (moist)	0		99.3	97.2
	1		96.9	95.5.
	2		95.1	88.1
Scum (dry)	0	Petroleum diesel	95.2	98.6
	1		89.2	95.2
	2		83.3	85.5
Scum (moist)	0		99.1	95.9
	1		97.3	93.2
	2		96.2	91.1

oil, and grease (FOG) which are rich in free fatty acids (Marufuzzaman et al., 2014) and have a high lipid content (Bi et al., 2015; Sangaletti-Gerhard et al., 2015). The possibility of using municipal sludge as a feedstock for biodiesel production is a critical approach to decrease the sludge management cost. Moreover, there is a significant availability of sludge with high oil content in the developed countries (Wang et al., 2016). The amount of oil present in sludge varies and is strongly dependent upon the population and urbanization of the city (Wang et al., 2015).

Thus, scum is more beneficial for biodiesel production than primary or secondary sludge. Wang et al. (2016) used freeze-dried scum solids for oil extraction using hexane as an organic solvent, and 91.2% (w/w) of oil was extracted. Further, in situ transesterification to obtain biodiesel from scum sludge accounted for 57.5 to 64.1% of unsaturated fatty acid esters. However, utilization of organic solvents can increase the biodiesel production cost. Yellapu et al. (2018) investigated petroleum diesel as a solvent for microbial lipid extraction from wet biomass, with an oil extraction efficiency of 94.2% w/w. Petroleum diesel is a non-polar solvent with a high boiling point and has low cost compared with organic solvent.

Therefore, the primary objective of this study is to develop a sustainable approach for using scum as a feedstock for biodiesel production without the use of any toxic organic solvent. In this study, petroleum diesel was introduced for the first time as a solvent, and results were compared using a standard solvent (hexane) to separate oil. The effects of different physical factors (temperature, agitation, time, solids concentration, solids moisture content and solvent recycling) on oil extraction efficiency were studied. The separated oil in the solvent was directly transesterified to biodiesel.

On the other hand, the challenge of this research is toward the development of alternate routes for sludge management in wastewater treatment plant and to introduce a solvent (petroleum diesel) to extract oil from scum, which can be further transesterified and directly used as a biofuel without any further blending.

Methodology

Sampling and characterization of wastewater sludge and scum

Scum was collected from a municipal wastewater treatment plant located in Quebec City, Canada. Wet scum was transferred to freeze-thaw trays and freeze-dried for 4 days to remove the total moisture. After freeze-drying, samples were used for oil extraction studies. The freeze-dried scum solids were characterized to determine the metal concentrations, oil concentration % (w/w) and free fatty acids (FFA) % (w/w) (Table 1). The chemicals and organic solvents used in this study were purchased from standard companies, and petroleum diesel was obtained from a local gas station in Quebec City, Canada.

extraction from sludge using hexane or petroleum diesel

The oil extraction was conducted using a modified method as described by Jarde et al. (2005). The freeze-dried scum solids were crushed using a mortar and pestle into a powder and dissolved in hexane or petroleum diesel to extract oil from different sludge samples or scum powder. One gram of scum powder was weighed in different screw-capped tubes (3 cm width and 50 mL working volume), and 15 mL of hexane or petroleum diesel was added and well mixed. The solvent and scum powder mixtures were incubated for 4 h at 70 °C with 400 rpm agitation in an orbital shaking incubator (Thermoscientific Inc., Canada). After incubation, the total mixture was filtered using a standard filter with mesh size 125 mm. In the case of hexane as a co-solvent, the filtrate was transferred into a pre-weighed tube (W_1) and oven dried at 60 °C until constant weight (W_2) was obtained. The oil content % (w/w) of the different freeze-dried scum was calculated as:

$$CO\ \% = \frac{W2-W1}{1} \times 100\% \qquad (1)$$

In Equation (1), CO% represents the % of oil content obtained by the conventional oil extraction method using hexane as solvent. W_1 represents the weight of the pre-weighed screw-cap tube, W_2 denotes the weight of oven-dried oil in the pre-weighed screw cap tube, and 1 denotes the dry scum powder weight in grams. The extracted oil was stored for further transesterification study.

However, in the case of petroleum diesel as a co-solvent, the filtered oil present in petroleum diesel (O_{pd}) was directly used for transesterification without drying. The oil transesterified to fatty acid methyl esters (FAMEs) was analyzed using FT-IR and obtained FAMEs were used to calculate the weight of the scum oil separated using petroleum diesel.

The total oil obtained by using petroleum diesel was calculated according to Equation (2):

$$\text{Oil extraction efficiency of petroleum diesel } (\%\ w/w) = O_{pd}/O_c \times 100\% \qquad (2)$$

where O_{pd} is the weight of oil (g) present in 15 mL petroleum diesel, and O_c is the dry weight of oil (Oc =0.23g) obtained using 1 g of dried scum powder with hexane as solvent.

Optimization of oil extraction parameters using dry scum

The physical parameters for oil extraction were optimized using freeze-dried scum powder. After that, the optimized parameters for freeze-dried scum were further validated using 1 kg of scum solids concentration.

Effect of temperature

The dried scum samples of 1 g each were weighed in different screw-capped tubes and mixed with 15 mL hexane or petroleum diesel. The tubes were incubated at different temperatures (30, 40, 50, 60, 70 and 80 °C) for 4 h with agitation at 400 rpm. After the reaction, the samples were filtered and analyzed as discussed in section 2.2.

Effect of agitation

The dried scum samples of 1 g each were weighed in different screw-capped tubes and were mixed with 15 mL hexane or petroleum diesel. The test tubes were incubated at the optimized temperature of 60 °C in an orbital shaker incubator (Thermo Scientific Inc.) with different agitation speeds of 100–400 rpm for 4 h. After the reaction, samples were filtered and analyzed as discussed in section 2.2.

Effect of extraction time

The dried scum samples of 1 g each were weighed in different screw-capped tubes and mixed with 15 mL hexane or petroleum diesel. The tubes were incubated at 60 °C temperature for a different incubation times of 30–240 min with an optimized agitation speed of 300 rpm. After the reaction, samples were filtered and analyzed as discussed in section 2.2.

Effect of moisture content

The oil extraction process using freeze-dried scum solids is a highly cost-intensive process; therefore, to save energy the direct use of wet scum is recommended to develop a cost-effective process for biodiesel production. However, the presence of moisture content in the scum solids can affect oil extraction efficiency. Therefore, to check the effect of different percentages of scum moisture content on the oil extraction efficiency, scum was initially freeze-dried. Different moisture contents in scum were adjusted using tap water. The dried scum samples of 1 g each were weighed in different screw-capped tubes, and moisture contents of 0, 20, 40 and 60% wt were adjusted with tap water. In another tube, scum without drying (72.9% moisture content) was added in equal weight to 1 g dry scum. Hexane or petroleum diesel at 15 mL/g dry scum was added, and the total mixture was incubated at 60 °C using an orbital shaking incubator for 1 h with an agitation speed of 300 rpm. After the reaction, the total mixture was filtered using a 125-mm mesh size filter, and total filtrate volume was estimated for solvent recovery %.

$$\text{Solvent recovery \% (v/v)} = \text{Filtrate solvent volume}/\text{Initial solvent volume} \times 100 \quad (3)$$

The filtrate was analyzed for oil content % wt as discussed in section 2.2.

Effect of sludge solids concentration

The different powdered dried scum samples (0.375, 0.75, 1.125, 1.5, 1.87 and 2.25 g) were weighed in their respective screw-capped tubes with constant solvent volume (15 mL), and the final scum solids concentration in the tubes was 25, 50, 75, 100, 125 and 150 g solids/L solvent, respectively. Hexane or petroleum diesel was added to each tube, and tubes were incubated at 60 °C for 4 h with agitation at 300 rpm. After cooling to room temperature, the total reaction mixture was filtered and analyzed as discussed in section 2.2.

Recycle capability of hexane and petroleum diesel as a solvent for oil extraction

The dried scum and wet scum (moisture content 72.3% w/w) samples of 1 g and 3.5 g (which is equal to 1 g dry weight), respectively, were weighed in four different screw-capped tubes in two sets and further mixed with 15 mL hexane or petroleum diesel. The reaction was performed under optimized conditions, and tubes were incubated at 60 °C for an incubation time of 60 min with an agitation speed of 300 rpm. After the reaction, the samples were filtered using a 125-mm mesh size filter, and the volume of filtrate (oil in petroleum diesel or oil in hexane) was measured for solvent recovery %. After that, 1 g of dried scum or 3.5 g of wet scum was again added into the different filtrates (oil in a solvent obtained by using dry or wet scum) and incubated under optimized conditions. The samples were further filtered, and the volume of filtrate was measured. The same process was repeated 2 more times (recycled 3 times in total) to increase the oil concentration, and this process is known as solvent recycling. The solvent recovery % and oil concentration in the solvent were calculated after each cycle as discussed in sections 2.2 and 2.3.4.

Validation of oil extraction using 1 kg scum

The experiment was conducted using a 15-L plastic bucket (oil extraction reactor) equipped with an agitator and temperature control jacket with knob. One kilogram of scum (moisture content 72.3%) was added to the oil extraction reactor, and solids concentration was adjusted to 75 g scum solids/L solvent (hexane or petroleum diesel), then closed with a tight lid and the container was sealed. After that, optimum parameters such as temperature 60 °C and agitation 300 rpm were controlled, and the reaction was terminated after 60 min. After oil extraction, the complete mixture was filtered using an 800-mm standard filter. The filtrate was collected in a beaker and allowed to settle for 2 h, and clear phase separation was observed in two phases. The top phase consists of scum oil dissolved in solvent and the bottom phase consists of water with some residual solids. The top phase was again recycled (0, 1 and 2 times) to extract oil repeatedly and to determine the saturation level of oil to be dissolved (or extracted) in solvent, and similar steps were followed to those explained above. After each cycle, the volume of the solvent (hexane or petroleum diesel) was measured, and transesterification was conducted to determine the oil-to-FAME conversion efficiency.

Figure 1. Effect of different factors on oil recovery from scum using hexane or petroleum diesel as solvents: (a) temperature; (b) agitation; c) time; (d) moisture content; (e) scum solids concentration.

Acid transesterification and biodiesel metal contaminants

The oil samples obtained using hexane or petroleum diesel were transesterified using acid as a catalyst. Acid catalyst was used for transesterification due to high free fatty acid (> 3% w/w) content in the extracted oil. The FAMEs obtained from oil separated using hexane as a solvent were analyzed with GC-FID as discussed in our earlier study (Yellapu et al., 2017). However, in the case of oil separated using petroleum diesel as a solvent, the obtained FAMEs were analyzed with FT-IR as discussed by Yellapu et al. (2018). The obtained FAMEs were characterized for metal analysis using ICP-AES axial vista.

Results and discussion

The characterization of scum reveals 29.3% w/w oil content and 12.5% w/w free fatty acid content in the extracted oil, and the oil was dark brown in color, with density greater than 1. The high concentration of oil and free fatty acids in scum was due to the presence of a mixture of FOG, cellulose fibers, hairs and other light solids (Demirbas et al., 2017). Moreover, the density higher than 1 may be due to contamination of the oil with organic matter from the scum solids.

Several researchers have reported the presence of oil content in scum and this may be due to the urbanization and industrialization of metropolitan cities (Yapıcıoğlu & Demir, 2017). Oil can be separated from scum with polar and non-polar solvents. Organic solvents (hexane) are expensive and cause concerns related to industrial safety; therefore, an alternative and low-cost solvent (petroleum diesel) was introduced to separate oil from scum. The oil extraction efficiency obtained using hexane as a solvent was considered to be 100%. The oil extraction efficiency using petroleum diesel as a solvent for scum was 97.8% w/w. Similar oil extraction results were obtained by Wang et al. (2016) using a combination of different solvents such as methanol, hexane, and acetone, and maximum oil contents of 33% w/w were obtained from scum solids using hexane as a solvent.

Effects of physical and chemical parameters for oil extraction

Effect of temperature

The oil extraction efficiency curve, illustrating the effect of different temperatures on freeze-dried scum with hexane or petroleum diesel as a solvent, is presented in Figure 1(a). The FOG bonded to scum solids was solubilized based upon the physical characteristics and nature of the oil. The solubility of oil embedded in the scum solids increased with an increase in temperature from 30 to 80 °C with constant reaction time of 4 h and 400 rpm agitation, and the maximum oil extraction efficiency of 98.63 ± 1.1% (w/w) was obtained at 60 °C. Mu et al. (2016) reported that scum oil is solid at room temperature, and that temperatures higher than 40 °C can liquefy oil. However, a temperature higher than 80 °C can cause solubilization of organic matter into the solvent (hindering the oil measurement), and it may also decrease transesterification efficiency.

Karlovic et al. (1992) investigated the effect of temperature on the kinetics of oil extraction from corn germ and reported that increasing the temperature enhances the capacity of solvents to dissolve the oil because of the thermal energy that overcomes the cohesive and adhesive interactions. Furthermore, the collision theory states that two molecules will only react if they have enough activation energy (Diphare & Muzenda, 2013). When the mixture is heated, the energy levels of the molecules increases and when the molecules are in their excited state, there will be more collisions between them. As a result, the rate of reaction or decomposition increases. Moreover, the energy input from heat provides the required energy to break the intermolecular forces of attraction between molecules, resulting in easy solubility in organic solvents.

Effect of agitation

The effect of agitation speed on oil separation using hexane or petroleum diesel is presented in Figure 1(b). Agitation increases the eddy diffusion and the transfer of FOG from the scum to the solvent (Diphare & Muzenda, 2013). The oil extraction efficiency increased from 64.16 to 98.98% (w/w) with an increase in agitation speed. The maximum oil extraction efficiency of 98.29% was obtained at 300 rpm with a constant optimum temperature of 60 °C within 4 h reaction time. However, for agitation speeds higher than 300 rpm, there was no significant increase in the oil recovery. The dependence of oil extraction efficiency on agitation shows that mass transfer plays a vital role in extraction.

Pilusa et al. (2013) reported that the role of agitation is to break down the grease molecules to liberate oil molecules as well as to increase its active surface area to interact with the solvent. Pilusa et al. (2013) further explained that once the bonds holding the scum matrix and oil are broken with the aid of agitation, oil floats in the mixture. Kadi and Fellag (2001) studied the effect of stirrer speed

Figure 2. Validation study using 1 kg scum with hexane (H) and petroleum diesel (PD): (a) solvent recovery percentage; (b) relative oil extraction and oil loss during solvent recycle.

on oil extraction from olive foot cake using hexane as a solvent. The oil content of 6.9 to 17.7% (w/w) was extracted at agitation speeds varying from 600 to 1000 rpm. This demonstrates that agitation is a crucial factor in the separation of oil from the scum or sludge solids.

Effect of time

Extraction time is an important parameter in the design and operation of extraction processes. The oil extraction efficiency increased with an increase in incubation time (Figure 1(c)). The maximum oil extraction efficiency of 98.97% (w/w) was achieved within 60 min incubation time at a constant temperature of 60 °C with agitation of 300 rpm.

The increase in extraction time above 60 min did not show any improvement in oil extraction. Various researchers have studied oil extraction from pulverized sludge solids at different reaction times, and the change in reaction time could be further dependent on specific factors such as reaction volume, solvent type, condition of solids (wet/dry) and physical parameters (Ibrahim & Hamza, 2017). Hence, optimization of extraction time is necessary for every oil extraction process.

Effect of moisture content

The effect of moisture content on oil extraction efficiency using hexane or petroleum diesel is presented in Figure 1(d). The oil extraction efficiency was similar to that seen with an increase in moisture content from 10 to 72.3% w/w using hexane and petroleum diesel as a solvent. Oil extraction efficiencies of 98.9 and 97.2% (w/w) were attained at 10 and 72.3% moisture content, respectively, using hexane as solvent. Similarly, oil extraction efficiencies of 98.6 and 95.9% (w/w) were obtained using petroleum diesel as a solvent with optimum parameters such as temperature 60 °C, agitation 300 rpm and time 60 min. However, the color of the filtrate (oil dissolved in solvent) was lighter with an increase in moisture content from 10 to 72.3% w/w. After reaction of solvent with scum solids containing different moisture contents, the solvent recovery percentage increased from 80 to 96% v/v with an increase in moisture content. This is explained by the fact that freeze-dried scum solids react with solvent during oil extraction, absorbing more solvent. The absorption capacity of the solvent to the sludge solids decreases with increased moisture content of the solids. Therefore, solvent recovery % was higher with an increase in moisture content of the scum solids.

Effect of scum solids concentration

The oil extraction efficiency using different concentrations of freeze-dried scum solids with hexane or petroleum diesel is presented in Figure 1(e). The maximum oil recovery was obtained by using 25–75 g scum solids/L solvent. After that, with an increase in solids concentration from 100 to 150 g solids/L solvent at optimum values of temperature 60 °C, 60 min, and agitation 300 rpm, a decrease in oil recovery from 93.5 to 90.8% w/w was observed using hexane as solvent. A similar scenario was found in the case of petroleum diesel as a co-solvent. This may be due to the impact of solids concentration on the energy transfer between the surfaces of the particles. With higher solids concentration, the energy requirement for liquid–liquid separation would be lower. Hence, at low solids concentration, the interaction between solvent and oil will be higher (Zhang et al., 2014). Therefore, the optimum solids concentration of scum to achieve maximum oil extraction efficiency was 75 g solids/L solvent.

Recycle capability of hexane and petroleum diesel

The solvent (hexane or petroleum diesel) can dissolve oil from sludge or scum solids. Reusing the same solvent (filtrate) to dissolve oil from scum or sludge repeatedly is known as solvent recycling. Table 1 presents the effect of solvent recycling on oil extraction efficiency as well as solvent recovery % using hexane or petroleum diesel as a solvent with dry and wet scum (72.3% w/w moisture content). The oil concentration in solvent was increased proportionally using hexane or petroleum diesel, and oil concentration of 18.18, 33.3 and 59.4 g oil/L solvent was observed in the case of dry scum, whereas with wet scum the oil concentration was 19.4, 39.3 and 64.2 g oil/L during 0, 1 and 2 recycles, respectively. The oil extraction efficiency % (w/w) and solvent recovery % (v/v) were almost the same for hexane and petroleum diesel. However, wet and dry scum drastically affected the solvent recovery % v/v and oil extraction efficiency % w/w at each recycle time. The solvent recovery and oil extraction efficiency decreased by 12–14% v/v and 13–19% v/v, respectively, with increasing recycle numbers of 0, 1 and 2 using dry scum and hexane or petroleum diesel as a solvent. However, in the case of wet scum, the solvent recovery and oil extraction efficiency decreased by 3–12%w/w and 4–9%w/w, respectively, with increasing recycle numbers of 0, 1 and 2 using hexane or petroleum diesel as a solvent. These results indicate that the solvent was not absorbed in the wet scum solids as

Figure 3. Comparison of fatty acid profiles of biodiesel obtained from and scum oil. FAMEs: Fatty acid methyl esters.

the non-polar nature of the solvent prevents the absorption along with moisture or water which is polar. Therefore, the use of wet scum for oil recovery can reduce the solvent contamination in scum solids.

Validation of oil extraction using 1 kg scum solids

The optimized parameters were validated using 1 kg of scum with moisture content of 73.8%. In this study, the results were almost the same as those obtained using 1 g of scum with hexane or petroleum diesel as solvents, and oil extraction efficiencies of 94.1 ± 1.3 and 91.3 ± 4.2% w/w, respectively, were obtained. The recycling capability of hexane and petroleum diesel was validated; the loss of solvent increased, and solvent recovery % decreased, with each recycle of 0 to 2 (Figure 2(a)). After each recycle, the relative oil concentration in the solvent increased using hexane or petroleum diesel (Figure 2(b)). However, the maximum oil concentration of 63.6 g/L was obtained using petroleum diesel. The relative oil concentration using hexane (57.5 g/L) was lower as compared to petroleum diesel as a solvent. In Figure 2(b) the relative oil concentration and solvent recovery % are interdependent. When the number of recycles was increased, the hexane recovery percentage decreased compared to that of petroleum diesel, and in that way the relative oil concentration in the solvent (g/L) also decreased in the case of hexane. The decrease in oil concentration can be explained by the physical properties of the solvent such as boiling point. The boiling point of hexane is 60 °C while that of petroleum diesel is 160–230 °C, and in this experiment the optimum temperature for oil extraction was 60 °C. Therefore, during the reaction (oil extraction) hexane was in the boiling stage and may have evaporated, and therefore solvent recovery % decreased during each cycle, which further affected the relative oil concentration. Therefore, petroleum diesel was found to be a more effective solvent for oil extraction from scum.

Ex situ transesterification and biodiesel metal contaminants

The oil recovered from scum was directly transesterified without any further drying (with solvent). The oil-to-FAME conversion efficiency of scum using hexane and petroleum diesel as co-solvent was 93.3 ± 1.9 and 95.2 ± 1.1% w/w, respectively. Figure 3 shows that oil extracted from scum oil was converted to C18:1 and C18:2. It explains neutral lipids are high in scum solids. The FAMEs obtained from high neutral lipids are highly combustible for engine ignition.

Table 2 displays the concentrations of metals present in scum solids (g/kg) and the final biodiesel product (mg/L). The concentrations of metals leached during extraction of oil and conversion to biodiesel for scum were different with hexane versus petroleum diesel as a solvent. The concentrations of metals present in biodiesel obtained by ex situ transesterification using hexane are as follows: biodiesel obtained from scum: Ca > P > Fe > Al > S > Mg > K. The metals were present in low concentrations in the biodiesel, converted using oil extracted with petroleum diesel as solvent as compared to biodiesel transesterified using oil extracted with hexane. The final obtained biodiesel was contaminated with metals, which may cause problems for engine ignition. As per biodiesel ASTM D6751, the metal concentrations need to be less than or equal to ASTM biodiesel norms (sulfur: 0.05mg/L; calcium: 5 ppm; magnesium: 5 ppm; phosphorus: 0.001% mass; sodium: 5 ppm) for biodiesel produced using scum solids. Therefore, to use biodiesel obtained from sludge in commercial and automobile sectors, further research on purification is underway to remove contaminants using dry washing methods with ion exchange resins such as Lewatit®, DW-R10 and SEPLITE®.

Conclusion

The scum from a municipal wastewater treatment plant was used for oil extraction, and the obtained oil was transesterified to biodiesel. The physical parameters significantly affected the oil extraction efficiency from scum using hexane and petroleum diesel as a solvent. The optimized parameters for maximum oil extraction efficiency were temperature 60 °C, agitation 300 rpm, time 60 min and 75 g/L solids concentration. The oil extraction efficiency obtained using hexane and petroleum diesel was almost the same in all cases. The moisture content in solids affected the solvent recovery percentage, but there was no impact on oil extraction efficiency. However, the solvent recovery decreased with an increase in the number of cycles, and solvent loss was greatr in the case of hexane as compared with petroleum diesel.

Ex situ transesterification without solvent evaporation (oil drying) gave >90% w/w FAME conversion. The presence of metals affected the quality of biodiesel. However, further research toward the purification of biodiesel to

Table 2. Characterization of metals in scum solids and biodiesel obtained using scum oil.

		Scum	
Metals	Scum solid (g/kg)	Biodiesel (mg/L) Hexane[a]	Biodiesel (mg/L) Petroleum diesel[a]
Al	3.55	0.80	0.54
Ca	16.52	3.72	2.67
Fe	4.13	0.93	0.51
K	0.98	0.22	0.18
Mg	1.06	0.24	0.15
Na	0.93	0.21	0.15
P	4.62	1.04	0.66
S	3.27	0.73	0.57

[a]After one time recycling solvent for oil separation from scum.

remove contaminants could be beneficial to establish scum oil as substantial feedstock for biodiesel production.

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