

A Simple Method To Extract Fats, Oil And Grease For Biodiesel Production From Grease Trap Waste

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Abstract— fats, oil, and grease (FOG), the main composition of dewatered grease trap waste (GTW), is the major cause of sewer pipe blockage that can lead to a costly treatment for councils as well as the households. To prevent this, grease traps are required to be installed in most of the hospitality services such as restaurants as well as food processing factories. As a result, a large amount of GTW is being collected and need to be treated that can cause many problems for most of the environmental services. This study investigated a potential and simple solvent extraction method to extract FOG from GTW. The resulting FOG was then used to produce biodiesel utilizing ethanol as a reagent. GTW was collected from an environmental service in Adelaide, South Australia, while hexane (HEX), diethyl ether (DEE) and a mixture of hexane - diethyl ether (HEX-DEE) were utilized as a solvent to extract FOG. The results showed that the extraction yield was increased significantly while utilizing the solvent in the following order: DEE > HEX-DEE > HEX. A 96% extraction yield was obtained under the optimum operating condition of 1:1 DEE-GTW ratio (vol/w), 30°C, 300 rpm and 360 min. In addition, all of the solvents are recycled and can be utilized continuously two to three times for FOG extraction with approximately to 88% extraction yield. **Keywords**—component; formatting; style; styling; insert (key words)

I. INTRODUCTION

Grease trap waste (GTW) is recently considered as a potential feedstock for biodiesel production [1-5], since it can be collected for free from wastewater sewerage systems of many food processing services. In fact, while many biodiesel producers have to spend money for other biodiesel feedstock, GTW collectors can also get pay from restaurants, food factories or household to treat grease that can cause pipe blockage

in the sewerage system. According to Haas and Foglia [6], the feedstock is responsible for up to 80% of the total operating cost to produce biodiesel. This is the main reason limiting the market growth of biodiesel in comparison to other fuels. Thus, while GTW can be considered as a potential feedstock, GTW collectors are prospective producers which can open a door for biodiesel to the global market. In the United States, an estimated 1.8 billion kg/year of lipids could be recovered from GTW, which could produce about 1.3 billion kg of biodiesel/year [7]. In Adelaide (South Australia), one of the major environmental services collected approximately 10 million liters of GTW in 2011. According to this service, this represents around 40% of the total GTW collected in Adelaide, resulting in an estimated total GTW generation of 25 million liters of GTW per year. After water is removed, this corresponds to annual 'brown grease' production of around 2.5kg per person per year. That was a great amount of feedstock for biodiesel production. Figure 1 shows the partially dewatered GTW obtained from an environmental service in Adelaide [8].



Figure 1. Dewatered grease trap waste

Recently, great efforts have been put into research that utilizes GTW as a feedstock for biodiesel production to reduce a feedstock cost [1, 3-5, 9, 10]. In practice, it has been announced that biodiesel has been

successfully produced from GTW utilizing methanol as a reagent and lead to a commercial product. However, there is limited study concentrated on the extraction of FOG which is a challenging process to make biodiesel from GTW. GTW is significantly more contaminated than waste cooking oil, with much higher levels of non-oil contaminants. This makes producing biodiesel from these feedstocks more technically challenging and costly. Another major issue with GTW is the very high level of free fatty acids (FFAs) present, formed by hydrolysis of triglycerides [9, 11, 12]. In previous studies, lipid separation from GTW is basically based on heating [13] that was not effective to extract the lipid fraction completely, while heating can also promote the hydrolysis process that can change the composition of the GTW in nature [3, 10, 14, 15]. Recently, attempts have been made to extract FOG from GTW using waste cooking oil (WCO) as a solvent [12]. It was reported that 95% extraction yield can be obtained after 240 min, at 70°C and the ratio between WCO-GTW is 3.2:1 (wt/wt). However, due to the fact that WCO is easy to be contaminated by GTW and lead to a costly refinery process as well as a moderate energy consumption is required, this method still not yet be applied in practical production. Moreover, the use of methanol, a toxic chemical, as a reagent for the biodiesel synthesis reaction is also a concern of the biodiesel industry. Therefore, the purpose of this study is to examine the use of popular industrial solvents, hexane and diethyl ether, to extract FOG from GTW and utilize it as a feedstock for biodiesel production. A very simple extraction process has been developed to meet the demand of industrial producers. The benefit of these solvents is that the extraction process can be conducted without heating which is a priority option for many environmental services, the most potential biodiesel producers in Australia, which were constructed mostly in the remote areas. Moreover, hexane and diethyl ether can be recycled and continuously used for the extraction process. On the other hand, ethanol was used as the main reagent for the biodiesel synthesis reaction to make biodiesel a greener product compared to the one in which methanol is utilized as a reagent.

II. MATERIALS AND METHODS

1. GTW collection and preparation

GTW, which contains very high FFA, is provided by an environmental service in Adelaide. A truck team operated by this service to collect GTW from grease traps that are installed in the sewerage system of food processing services around Adelaide. GTW is then pumped into the storage tanks awaiting for further treatment. In this study, GTW samples were taken from the top of the storage tanks where is rich of FOG and has less water compared to those at the middle or at the bottom of the tanks. After transferring to the lab, the raw GTW was dried in an oven at 110°C for 24 h to 48 h until the moisture of the sample dropped below 5%.

2. FOG extraction and analysis

2.1. FOG extraction

After being dried in the oven, FOG was extracted from GTW by the popular industrial solvent, hexane and diethyl ether. 500g of GTW was placed in a 1 L volume Pyrex reaction vessel that was equipped with a mechanical stirrer. The stirrer speed was set constantly at 300 rpm. The solvent was then added to the vessel with a different ratio based on GTW weight. The extraction process was conducted at room temperature in order to save energy and lower the cost of biodiesel making. Figure 2 shows the extraction procedure proposed in this study.

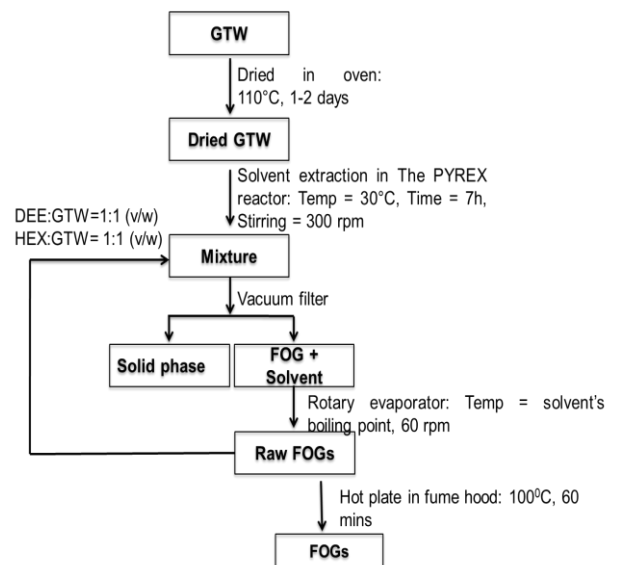


Figure 2. Flow-chart of the extraction of FOG from GTW

Since FFA is the main component of FOG, extraction yield was calculated based on the amount of extracted FFA compared to the initial one.

The extraction yield was calculated by formula 2.1 [12].

$$\text{Extraction yield} = \text{FFAi}/\text{FFAo} \times 100\% \quad (2.1)$$

where:

FFAi is the amount of FFAs measured at the sampling time i (g).

FFAo is the maximum amount of FFA found in the raw GTW sample, (g).

To measure the amount of FFA, standard methodology AOCS Ca 5a-40 (AOCS, 2009) was applied to determine the percentage of FFA in every sample via titration. Following equation was used to calculate the FFA (%):

$$\text{FFA}\% = (V \times N(\text{NaOH}) \times 25.6)/W \quad (1)$$

Where: V is the volume of NaOH recorded when the pink colour persisted in the sample (mL)

N(NaOH) is the normality of NaOH, W is the weight of the GTW sample.

Mass of FFA was following calculated by formula 2.2

$$\text{Mass of FFA} = \frac{C_{\text{KOH}} * (V_{\text{KOH}} - V)}{V_S} * V_2 * M.W_{\text{FFA}} \quad (2.2)$$

Where:

- C_{KOH} - Concentration of KOH used for the titration analysis, 0.1 mol/L
- V_{KOH} - Amount of KOH used for the titration of 1mL of FOG, L
- V - Amount of KOH used for the titration of 1mL of blank solvent, L
- V_S - Volume of sample, 1mL
- V_2 - Final volume of extracted FOG, mL
- $M.W_{\text{FFA}}$ - Average molecular weight of FFAs, 345.5 g/mol based on FFA profile calculation.

FFAo was determined by the FFA% obtained from the application of the Two-step Bligh and Dyer method [16]

2.2. Solvent recovery

After the extraction process was finished, all solvents were recycled using a rotary evaporator. The solvent in the extracted phase was first removed following by a solvent recovery for the solid phase. The amount of recovery solvent is the total obtained from both processes. Based on the boiling points, the recovery temperature was set at 45°C and 75°C for DEE and HEX respectively. The solvent was then reused for the extraction of FOG and the effect of recycled solvent was determined based on the extracted FOG yield.



Figure 3. Solvent recovery using rotary evaporator

2.3. Fatty acid profile

Fatty acids profile was determined by Perkin-Elmer Clarus 500 GC-FID instrument following a self-developed methodology that has been certified and validated by the analytical service of The University of Adelaide. To study the fatty acid profile, all FOG

samples were converted into methyl ester (using boron trifluoride in methanol 20%) and diluted with hexane before analyzing with GC-FID. The 24 mix FAME standard was purchased from Supelco while ethyl nonadecanoate (C19 ethyl ester) was applied as an internal standard.

2.4. Biodiesel production

The extracted FOG was converted to free fatty acid ethyl esters (FAEEs) via esterification reaction with the acid catalyst. 34.5g (0.1 mol) FOG was added to a capped vial heated and stirred by a magnetic hot plate. The magnetic stirrer was set constantly at 400 rpm while the reaction time, reaction temperature and FOG to ethanol ratio were changed respectively. After the FOG was heated up to a specific temperature, a mixture of ethanol and sulphuric acid was added. It was noticed that the temperature of the mixture will increase 10°C approximately when sulphuric acid was added. Therefore, the initial FOG should only be heated to 10°C lower than the expected temperature. The heating and stirring were stopped after the reaction has reached the determined reaction time. The temperature of the reactor was then quickly reduced by cold water. The mixture was allowed to settle for 1 h resulting in the separation of two distinct liquid phases. The excess ethanol in the top phase was removed using separating funnel, whereas the bottom product containing ethyl ester (biodiesel) and unreacted oil was collected. The biodiesel was washed 3 times with warm water and finally heated to 120°C to remove the remaining water before the mass of the product was measured. FAEEs conversion was estimated by gas chromatography (GC) analysis of the products.

III. RESULTS AND DISCUSSION

1. The compositions of GTW

3 samples were analysed for moisture content, glycerides, FFA level, and solids remain. The result is showed in Table 1. While the percentage of glycerides is similar to those reported in previous studies [11, 12, 17], the GTW collected in the top of the storage tank has lower moisture content and higher FFA level. The higher FFA concentration can be explained as the effect of cooking procedure and the hydrolysis caused by microbial activities during the disposal of GTW into the sewer pipes. This is a technical challenge for biodiesel production from trap grease since it easily causes the saponification when a transesterification reaction is performed.

Table 1. Some properties of GTW

| Sample | Moisture (%) | Glycerides (%) | FFA (%) | Solids remain (%) | Unmeasured part (%) |
|--------|--------------|----------------|---------|-------------------|---------------------|
| #1 | 38.7 | 0.17 | 39 | 21 | 2 |
| #2 | 39.5 | 0.18 | 37 | 19 | 3 |
| #3 | 41.8 | 0.23 | 36 | 17 | 3 |

2. FOG extraction

2.1. The effect of solvent - GTW ratio

Since this study is focused on reducing the energy consumption, all extraction experiments were conducted at the ambient temperature in Adelaide, South Australia (30°C). Each experiment was repeated 3 times and the average time for each experiment is 420 minutes (7 hrs), samples were taken every 60 minutes (1 hr), and the stirring rate was set continuously at 300 rpm. Figure 2 shows the setup for a batch of FOG extraction. The ratio between GTW and solvent like hexane (HEX) and diethyl ether (DEE) was set up as showed in Table 2.

Table 2. Solvent - GTW ratio setup

| Experiments | GTW - HEX (w/vol) | GTW - DEE (w/vol) |
|-------------|-------------------|-------------------|
| #1 | 1:1 | 1:1 |
| #2 | 1:1.5 | 1:1.5 |
| #3 | 1:2 | 1:2 |

The results of the extraction process using hexane and diethyl ether with a different solvent to GTW ratio were displayed in Figure 3.

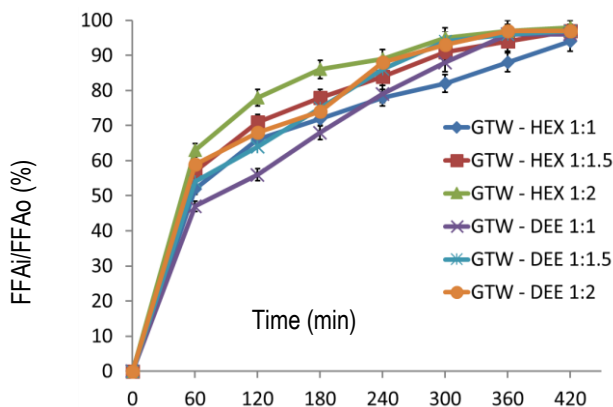


Figure 3. The effect of solvent - GTW ratio on the extraction yield

It can be seen in Figure 3 that a maximum of 96% of extraction yield can be obtained after 6 hours utilizing diethyl ether as a solvent with the GTW - DEE ratio is 1:1 (w/vol). The achieved extraction yield is higher to

compare with the one in the previous studies [18-22]. However, it was observed that there is an emulsion phase forming in the mixture of DEE - GTW after 1 hour of settling down as shown in Figure 4.



Figure 4. Emulsion phase observed in the FOG mixture extracted by DEE (middle tube)

By the contrast, there is no evidence of emulsion phase forming in the mixture of HEX - GTW as the mixture is clear. Therefore, to take advantage of the yield efficiency while eliminating the forming of emulsion phase, a mixture of hexane and diethyl ether was utilized with the ratio 1:1 (vol/vol). The optimum condition that obtained with DEE previously was applied for a batch extraction with this solvent mixture. The result is shown in Figure 5.

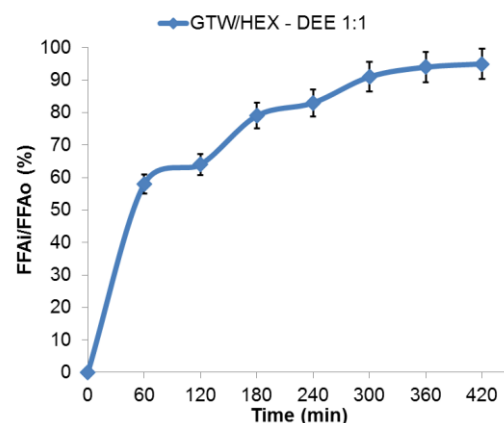


Figure 5. The effect of co-solvent DEE/HEX - GTW ratio on the extraction yield

By utilizing the mixture of DEE - HEX (1:1 vol/vol), the maximum extraction yield recorded is 94% at 420 minutes (7 hrs). Interestingly, no emulsion phase was observed as the sediment was settled down for separation. This can be explained as diethyl ether tends

to dissolve FFA, while hexane can also dissolve glycerides and other unknown parts of GTW [23].

2.2. Solvent recycling

Since solvent is a very important factor contribute to the production cost of biodiesel, all solvents were recycled using a rotary evaporator. Table 3 shows the average amount of solvent recovery after 3 times used for FOG extraction. The volume value of recovered solvent is based on 500ml of solvent applied for each extraction batch.

Table 3. Average amount of solvent recovery for every batch of FOG extraction

| Sample | Hexane (ml) | Diethyl Ether (ml) | Hexane - Diethyl Ether (ml) |
|-----------------|-------------|--------------------|-----------------------------|
| Original volume | 500 | 500 | 500 |
| 1st recovery | 465 | 415 | 453 |
| 2nd recovery | 451 | 406 | 427 |
| 3rd recovery | 443 | 401 | 412 |

It can be seen that diethyl ether is harder to be recovered compared to hexane due to its very low evaporation point. An average of 93% volume of hexane can be recovered while only 83% volume of diethyl ether can be recovered in the same condition. The recovery of hexane is similar to the one reported by Siddiquee and Rohani [20]. The loss of solvent can be explained as the result of evaporation and the absorption process of unknown solid remains in the solid phase of GTW. All recovered solvents were utilized for FOG extraction with the same GTW - solvent ratio (1:1). Figure 6 shows the efficiency of the extraction process after diethyl ether was used for 3 times at the optimum condition.

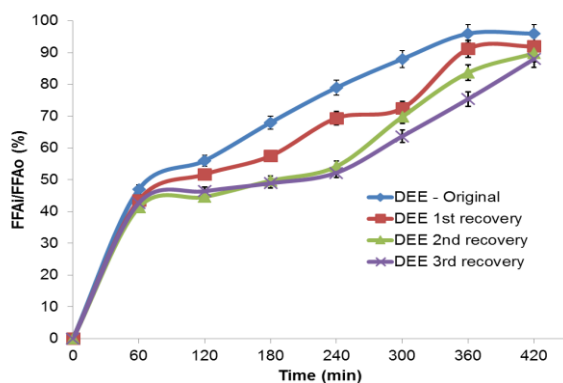


Figure 6. Yield efficiency of FOG extracted by recycled solvent

2.3. Fatty acid profile

FOGs extracted by hexane and diethyl ether were analyzed by GC-FID to determine the fatty acid profile. Surprisingly, the fatty acid profile is not significantly different between FOG extracted by hexane and diethyl ether. Hence, there is no selectivity in terms of fatty acid when using hexane and diethyl ether as the solvents for FOG extraction. Table

Table 4. Physical properties and the fatty acid profile of FOG

| Property | Amount |
|-----------------------------------|--------|
| Kinematic viscosity at 400C (cSt) | 31.2 |
| Water content (%wt) | 1.02 |
| Fatty acid composition (%wt) | |
| Octanoic acid (C8:0) | 0.4 |
| Decanoic acid (C10:0) | 1.3 |
| Lauric acid (C12:0) | 3.4 |
| Myristic acid (C14:0) | 0.4 |
| Palmitic acid (C16:0) | 21.9 |
| Palmitoleic acid (C16:1) | 1.2 |
| Stearic acid (C18:0) | 7.4 |
| Oleic acid (C18:1) | 47.6 |
| Linoleic acid (C18:2) | 13 |
| Linolenic acid (C18:3) | 1.8 |
| Arachidic acid (C20:0) | 1.0 |
| Behenic acid (C22:0) | 0.3 |
| Lignoceric acid (C24:0) | 0.1 |
| Average molecular weight (g/mol) | 345.5 |

3. Biodiesel synthesis from extracted FOG

To make biodiesel a greener product, ethanol was utilized as the reagent for the synthesis of biodiesel, while H₂SO₄ (the cheapest and most popular catalyst) was applied as the catalyst. The reaction was conducted in a 1 L Pyrex reactor equipped with a mechanical stirrer. The FOG – ethanol molar ratio was 1:3, 3% H₂SO₄ based on the amount of FOG, the reaction temperature was set at 65°C and the stirring speed was kept at 300rpm [24]. Ethyl nonadecanoate (C19 ethyl ester) was applied as the internal standard for the quantity method development. The result was analyzed by GC-FID and presented in Table 5 and Figure 7.

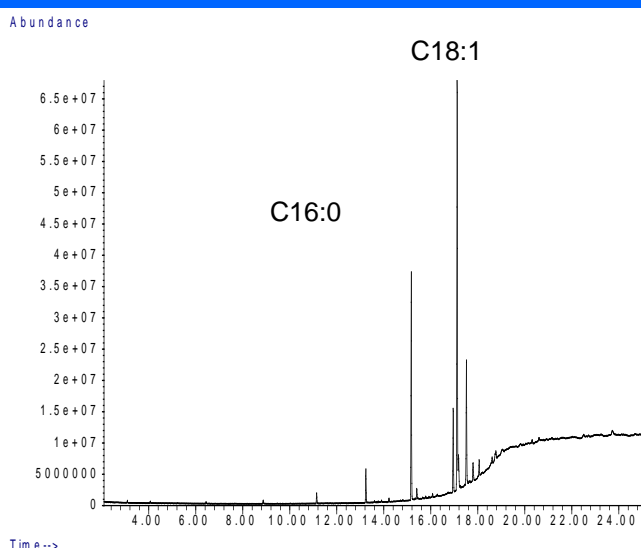


Figure 7. Chromatogram of ethyl esters analyzed from biodiesel sample

Table 5. Composition of biodiesel

| Peak # | Component Name | Time [min] | Area [$\mu\text{V}\cdot\text{s}$] | Concentration ug/ml |
|--------|----------------|------------|-------------------------------------|---------------------|
| - | C4:0 | 4.1 | 0 | 0.0 |
| - | C6:0 | 6.1 | 0 | 0.0 |
| - | C8:0 | 8.6 | 0 | 0.0 |
| - | C10:0 | 11.0 | 0 | 0.0 |
| - | C12:0 | 13.2 | 0 | 0.0 |
| 1 | C14:0 | 15.2 | 47318 | 53.4 |
| 2 | C16:0 | 17.1 | 278227 | 219.7 |
| 3 | C18:0 | 18.8 | 98026 | 78.9 |
| 4 | C18:1 | 19.0 | 635627 | 169.6 |
| 5 | C18:2 | 19.4 | 185739 | 126.8 |
| 6 | C19:0 | 19.7 | 154558 | ----- |
| 7 | | 20.0 | 29493 | 0.0 |
| - | C20:0 | 20.6 | 0 | 0.0 |
| - | C22:0 | 23.1 | 0 | 0.0 |
| - | C24:0 | 26.9 | 0 | 0.0 |
| 8 | | 28.5 | 31887 | 0.0 |
| | | | 1460875 | 648.5 |

It can be seen that C16 and C18 are the dominant components in the composition of the biodiesel that was synthesized from GTW. A yield of 96% FOG oil ethyl ester was obtained when the above described conditions were applied. This is higher than the product yield recently reported by Alhassan, Kumar [18]

IV. CONCLUSION

A simple process has been introduced to extract FOG from a partially dewatered GTW obtained from an environmental service in Adelaide, South Australia. Diethyl ether and hexane were utilized as the solvents for all of the extraction processes. A maximum extraction yield of 96% can be obtained when using diethyl ether as a solvent at the optimum conditions: 30°C temperature, 300rpm stirring speed, and the ratio between diethyl ether - GTW is 1:1 (vol/w). To reduce the forming of the emulsion in the extraction mixture, a mixture of hexane - diethyl ether with a ratio 1 to 1

(vol/vol) was introduced to the extraction process. All solvents were recovered by a rotary evaporator based on their boiling points to lower the operation cost. It was observed that up to 93% volume of solvents could be recycled and continuously used three times for the extraction process with an extraction yield of 88% obtained at the optimum condition. Finally, extracted FOG was examined as a potential feedstock for biodiesel production which can provide a very potential alternative fuel to substitute fossil fuels.

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