

A Comparison of Batch Extracted Bio-oil and Continuous Hydrothermal Liquefaction Bio-oil using Spent Coffee Grounds as Biomass Feedstock

T. Jansen van Rensburg and C.J. Schabort

Abstract—This study focuses on bio-oil obtained from spent coffee grounds, either by reflux extraction or continuous hydrothermal liquefaction. Spent coffee grounds was chosen as feedstock for this study as it is available around the world and considered a second generation feedstock, as it is a food waste. The production of coffee in 2017 was more than 9.5 million tons, which translates into an increase of 2.3% from 2016. A large portion of coffee beans end up as spent coffee grounds during the production of instant coffee, making this waste product an ideal feedstock for the biofuel industry.

Spent coffee grounds was collected from a local coffee shop in Potchefstroom and used as feedstock in the production and extraction of bio-oil from the spent coffee grounds. Reflux extraction was done on the dried spent coffee grounds using hexane, ethanol and acetone as solvents. Different retention times were investigated for each solvent and the yield of the oil was reported. The maximum yield 11.7 wt% was obtained when hexane was used as a solvent. Continuous hydrothermal liquefaction was done using spent coffee grounds as a feedstock and a bio-crude yield of 28.5 wt% was obtained. The average higher heating value of the extracted oils was 39 MJ/kg, while the higher heating value for the hydrothermal liquefaction oil was a bit lower at 36 MJ/kg.

Index Terms—continuous hydrothermal liquefaction, reflux extraction, spent coffee grounds

I. INTRODUCTION

Since the industrial revolution in the 18th century the use of fossil fuels increased dramatically. Coal was the only source of fossil fuel until around 1860 when crude oil consumption began [8]. In 1960 the human population was only 3 billion. This number more than doubled in the past 58 years to around 7.2 billion people in 2018. At the current growth rate of 1.2% per year the world population will double again in less than 60 years. This increase in population has increased the daily need for energy and thus the need for fuel [1].

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Africa is one of the fastest growing continents and is expected to have the highest growth in population between 2015 and 2050 [3]. This will lead to an increase in energy demand, which will translate into a more rapid decrease in fossil fuel reserves [1].

Sustainable energy sources like solar, wind, nuclear and biomass are being researched around the world due to the large strain on limited and depleting fossil fuel sources. The most suitable and sustainable energy source to be used in the production of transportation fuel is biomass. Within biomass the lipids that are comprised mainly of fatty acids and triglycerides, pose a promising application for biofuel production [10].

One method by which biofuels can be produced is the upgrading of bio-oils. There are a lot of different biomass feedstock that can be used to produce bio-oil. These biomass feedstocks include forest product wastes, agricultural residues, organic fractions of municipal solid wastes, paper, cardboard, plastic, food waste, as well as green waste [12]. With an average oil content of 15 wt%, spent coffee grounds (SCG) are also a suitable feedstock to produce bio-oil. According to the coffee foundation the average worldwide coffee consumption is more than 9.5 million tons of coffee per year [7]. In 2014 it was reported that more than 9 million tons of coffee ends up on landfills. This means that a viable and available bio-energy source is being wasted. [5]

The production of liquid fuel from lipids is usually done by pyrolysis, transesterification and hydrotreatment. Pyrolysis is a process where lipids produce gasoline-range hydrocarbons, but this process has a very low yield because of excessive gas formation. Transesterification of lipids is used to produce fatty acid methyl ester (FAME). FAME is known as first generation biodiesel and it can be used as a substitute for petroleum diesel. The development of FAME was restricted because of the disadvantages that come with the product. The disadvantages include high oxygen content, high viscosity, high cloud point temperature and poor oxidation stability.

The third process is hydrotreatment where lipids are converted into diesel-range alkanes (C₁₅-C₁₈). This is known as renewable or green diesel. This is the only diesel that can be used to completely replace ordinary petroleum diesel, because both share identical energy content and composition. [10]

II. MATERIALS AND METHOD

A. Materials

The spent coffee grounds (SCG) was collected from Toro coffee shop in Potchefstroom. The solvents used for the reflux extraction was n-hexane, acetone and ethanol. For the continuous hydrothermal liquefaction (HTL), filtered water was needed as the chlorine in Potchefstroom's water supply would corrode the reactor. For the difference in pressure needed to operate the plant, nitrogen was used and the final bio-crude was dissolved using acetone and filtered by means of a Büchner funnel.

B. Apparatus

For the reflux extraction a ball flask, oil bath, heating plate and a condenser was needed. The ball flask used had a capacity of 5 litres. The oil bath was set up on the heating plate with the ball flask inside the oil. The condenser was set up on top of the ball flask to condense any solvent that boils off. This decreases the loss of solvent and ensures the mixture does not run dry.

The temperature in the oil bath was increased or decreased depending on the different solvents that were used. A hand-held thermometer was used to measure the temperature of the oil in the oil bath. The solids were separated from the liquid using a Büchner funnel and the solvents were finally recovered using a rotary evaporator. The experimental setup of the reflux extraction experiments is shown in Fig. 1.

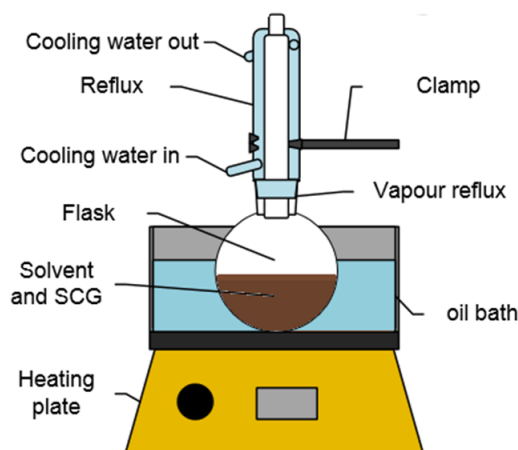


Fig. 1. Experimental setup of the reflux extraction.

The HTL experiments were performed using a continuous hydrothermal liquefaction pilot plant. The feed tanks to the reactor has a capacity of 100L and can be operated at a maximum flow rate of 150L/hr. The temperature of the reactor was increased using a hot oil plant that heated oil and pumped the oil through the reactor and the preheater. The maximum temperature of the hot oil plant is 340°C. The process flow diagram of the HTL pilot plant, is shown in Fig. 2.

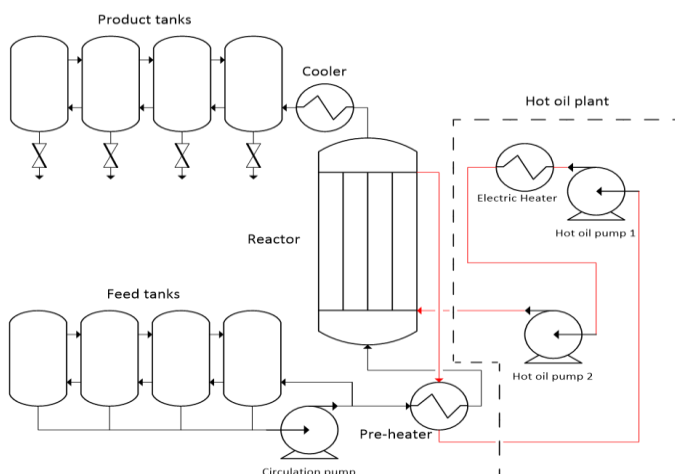


Fig. 2. PFD of the HTL pilot plant.

The heated oil flowed around the pipes of the reactor to ensure heat transfer to the feed. The slurry entering the reactor was preheated to a temperature of 130-140°C by means of the preheater. The slurry was further heated to a temperature of about 180°C inside the reactor before flowing through the cooler and into the product tanks. The liquid product was separated from the solid products using a pressure filter. The bio-crude was dissolved in acetone and separated using a Büchner funnel. Finally, the bio-oil was separated from the acetone using a rotary evaporator.

C. Procedure of reflux extraction

The SCG was dried in an oven at 110°C for 24 hours to remove all of the remaining moisture in the SCG. The dried SCG was then put into a 5L ball flask along with the solvent, either being hexane, acetone or ethanol. For each extraction, 500 grams of dried SCG was mixed with 1.5 litres of solvent. The volume-to-mass ratio of solvent to SCG remained 3:1 for each extraction. The mixture of SCG and solvent was brought to boil in an oil bath and boiled for either 1 or 2 hours, while the solvent was refluxed back into the ball flask.

After the ball flask was taken out of the oil bath, it was left to cool down for 15 minutes. A Büchner funnel was then used to separate the SCG solids from the bio-oil rich solvent. Once this separation was completed the solvent that was still mixed with the bio-oil was evaporated using a rotary evaporator. The bio-oil product was weighed and sent for analysis. All of these steps were followed using a different solvent in each case, namely hexane, acetone or ethanol.

D. Procedure for continuous HTL

1) Slurry preparation

The SCG used as feedstock for the continuous HTL runs was also obtained from Toro coffee shop in Potchefstroom. The SCG was dried prior to HTL, as the exact moisture of the SCG was not known. The SCG was spread out in oven pans and dried at 110°C for 24 hours. After the SCG was dried, exactly 2.2kg of dried SCG was weighed off and mixed with 20L of water and left overnight. This was done to ensure enough contact with the water so that the SCG could absorb the water and swell. The water functions as both a solvent and a catalyst in the continuous HTL process. This slurry was then mixed in the HTL feed tanks and additional water added to

make 60L total slurry. The feed to the reactor was thus 3 vol% solids.

2) Operation of the continuous HTL reactor

The circulation pump at the bottom of the feed tanks was used to ensure that the SCG was sufficiently mixed with the water. As soon as the SCG was sufficiently suspended in the slurry, the feed and product tanks were sealed. The feed through the reactor was induced by a difference in pressure between the feed and product tanks. Nitrogen was used to increase the pressure in the feed tanks and a bypass valve was used to equalize the pressure in the feed and product tanks.

Once the operating pressure of the process was reached the bypass valve was closed sealing the feed and product tanks from each other. As the pressure increased in the tanks the temperature was simultaneously increased using a heat transfer oil (HTO). The HTO temperature was set at the chosen set point. This was done to ensure that the temperature increased to the operating temperature inside the reactor.

Once the inlet stream of the HTO oil from the reactor had reached the desired temperature, the flow of the slurry to the reactor was started by opening the flow cut off valve located before the product tanks. The pressure difference was induced by venting the N₂ from the product tanks. By venting the N₂ from the product tanks a pressure difference was created between the feed and product tanks. The slurry flowed from the high pressure feed tanks through the reactor, to the low pressure product tanks. The flow through the reactor was controlled by the rate of depressurisation of the product tanks.

The slurry was then allowed to flow through the reactor. Once the liquid level in the feed tanks was low enough a liquid level controller switched off the circulation pump to keep the pump from running dry. The remaining slurry was sent through the reactor to ensure that all of the liquid and SCG were processed. The total time for the run was calculated by summing the time until the circulation pump switched off and the time that was still left to send the rest of the slurry through the reactor.

After each run the heaters that were used to heat the HTO were shut down and the reactor was left to cool down. As soon as the temperature of the HTO dropped below 100°C the circulation pumps in the hot oil plant were shut down. The HTL plant was then depressurised and the product was sampled into product drums and taken to the laboratory for analysis.

3) Product separation

After a successful HTL run the product was separated into the bio-crude or bio-oil, bio-char and aqueous phase. The product mixture was kept at room temperature where the bio-char and bio-crude was in a solid state, while the aqueous phase was a liquid. The separation of bio-crude and char from the aqueous phase was done by pressure filtration. Pressure filtration was used because of the large amount of separation that needed to be done and pressure filtration gave a bigger difference in pressure than Büchner vacuum filtration which was needed because the fatty bio-crude blocked the filter paper very fast.

When the separation between the bio-crude, bio-char and aqueous phase was complete the bio-char and bio-crude were separated from each other. This was done by using a solvent to dissolve the bio-crude phase while the bio-char phase remained a solid. The solvent that was used to dissolve the bio-crude was acetone. The solid bio-crude and bio-char were

soaked overnight in the acetone where after a Büchner funnel was used to separate the acetone rich crude from the char. Finally, the acetone was evaporated and recovered using a rotary evaporator.

E. Analysis

The yield of the HTL oil and the reflux extracted oil was determined using equation (1) and (2):

$$Yield = \frac{\text{mass of oil extracted}}{\text{mass of SCG used}} \times \frac{100}{1} \quad (1)$$

$$Yield(\%) = \frac{\text{mass of product}}{\text{mass (SCG loaded)} - \text{mass (SCG left in tanks)}} \times \frac{100}{1} \quad (2)$$

The same analysis was done on the HTL oil as well as the batch extracted oil so that both of the oils could be compared to each other.

- Gas Chromatograph - Mass Spectrometer (GC-MS) analysis was done to get the composition of the bio-oils. By knowing the composition of the oils it was possible to determine which pathway that was followed during the HTL process.
- Higher heating value (HHV) analysis was done in a bomb calorimeter to determine the energy density of the oils.
- Proximate analysis was done to determine the moisture, volatiles and ash content of the bio-oils. This was done in a vacuum oven.
- The oxidative stability analysis was done to determine the oxygen saturation of the bio-oils. This was used to indicate the longevity of the bio-oils.

III. RESULTS AND DISCUSSION

A. Fibre analysis of SCG

The composition of the SCG was needed to determine if the SCG could be used as a biomass feedstock for the extraction and the continuous HTL. Fibre analysis was done on the SCG to determine the composition thereof.

TABLE I: FIBRE ANALYSIS OF THE SCG

Component	wt%
Ash	1.34
Protein	12.71
Fat (ether extraction)	12.68
Carbohydrates	67.62
Hemicellulose	34.97
Cellulose	19.26
Lignin	10.54

From the Fibre analysis results reported in Table I it can be seen that the high protein, carbohydrate and fat content, as well as the low ash content, make the SCG ideal as feedstock for HTL and oil extraction. These values correspond to those obtained in other studies on SCG [9].

B. Yields obtained

1) Reflux extraction

The yield of the bio-oil by means of reflux extraction was determined using three different solvents. Each of the solvents were mixed in a ratio of 3:1. In each extraction 500 g of coffee was mixed with 1.5 litres of solvent. For each solvents two residence times were chosen and the yield was determined for both of the residence times. Five runs were done at each residence time for each solvent. Fig. 3 shows the yields obtained by each solvent for 1 hour of extraction *versus* 2 hours of extraction. The yields presented in Fig. 3 shows the yields in grams of oil per 100 grams of SCG.

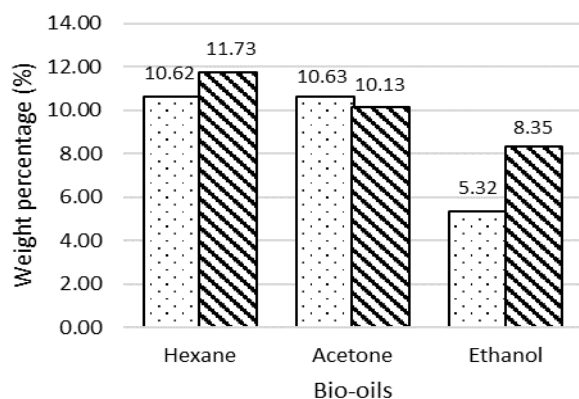


Fig. 3. Oil yield using different solvents and residence times.
□ 1 hr, ▨ 2 hr

In the case where 1 hour was used as residence time, the hexane and acetone resulted in almost similar yields of 10.62% and 10.63%, respectively. The oil yield obtained by means of ethanol extraction, 5.32%, was about 50% of that obtained by the other 2 solvents.

When the residence time was increased to 2 hours, the bio-oil yield increased slightly from 10.62% to 11.73%. An even greater increase in oil yield was observed in the case of ethanol as solvent, increasing from 5.32% to 8.35%. There was, however, a decrease in yield when acetone was used solvent. This could be due to a different batch of SCG that was collected.

An average yield of 11.73% for the hexane was obtained which is between the 10 – 15% average mentioned in the study done by Kondamudi *et al.* [6]. According to Kondamudi *et al.* [6], the average bio-oil yield has a large range because the bio-oil yield is affected by the different coffee grounds that is used when extracting the oil [6].

The yield of the acetone does correspond with the study done by Karmee [5] that obtained a yield of 12.92%. The difference in yield can be attributed to the difference in coffee grounds that was used. There can also be a yield difference because this experiment used reflux extraction while Karmee [5] used Soxhlet extraction.

It can be concluded that ethanol was the worst solvent to use in the tested batch extraction process and that the effect of time on the yield of the ethanol was the largest. Based on the results, it can also be concluded that either hexane or acetone could be used and that an economic evaluation is required to determine whether it is economically viable to extend the process time by 1 hour to obtain about 1% more oil.

2) Continuous HTL oil yield

The continuous HTL was done at a feed rate of 120 L/h, which translates into a residence time of 10 min for the HTL run. The yield obtained when using a residence time of 10 min was 28.5% and 15.2% for the bio-crude and the bio-char respectively

Yang *et al.* [11] did batch HTL on SCG in water to test the effect on the yield of the bio-oil at different reaction parameters. The study concluded that a shorter retention time gave the best bio-oil yield, which was 31.63%. This corresponds very well to the yield obtained in this study which is 28.5%. The small difference in yield can be because of the difference in the SCG and due to the reactor being continuous versus the batch the reactor used by Yang *et al.* [11].

C. Higher heating value

1) Reflux extracted oil

The HHV of each of the products was tested to see how much energy is stored in the product. Fig. 3 shows the HHV of the oils as well as the SCG used as the biomass.

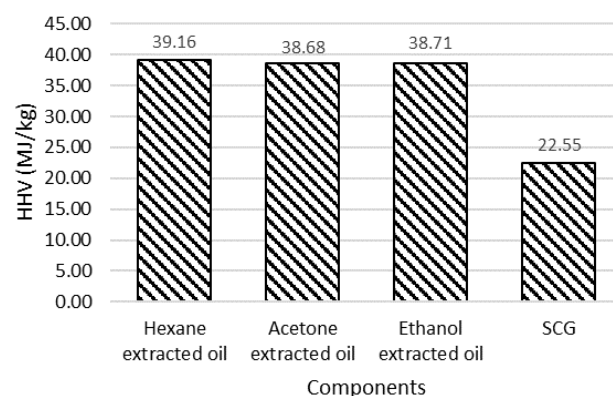


Fig 1. HHV of reflux extracted oils and the raw feedstock

With a maximum difference of 0.48 MJ/kg between the hexane extracted oil (39.16 MJ/kg) and the acetone extracted oil (38.68 MJ/kg), there is no real notable difference in the HHV when using different solvents for the extraction. This can be explained by the fact that the solvent is only used to extract the oil from the SCG without reacting physically with the solvent, resulting in a similar bio-oil, regardless of the solvent.

The HHV of the oils is around 39 MJ/kg, which corresponds very well with the HHV obtained by Deligiannis, *et al.* [2] who reported a HHV of 39.49 MJ/kg using coffee bio-oil extracted by soxhlet extraction and using SCG as biomass and hexane as solvent.

There was a large increase of the energy density from the raw feed to the bio-oils. The HHV of the hexane extracted oil increased with 16.61 MJ/kg which is a significant increase but still not in the transportation fuel range. According to Hore-Lacy [4], the HHV of petroleum crude is usually between 45-46 MJ/kg which means that the extracted oil will have to go through another process to further increase the HHV before being used as a transportation fuel. This can be done through a process like hydrotreatment [4].

2) HHV of continuous HTL products

A bomb calorimeter was used to measure the energy content of the bio-crude and the bio-char that were produced

during the HTL. The HHV of the oil produced using a residence time of 10 min was analysed. Table II gives the results obtained for the bio-char as well as the bio-oil.

TABLE I: HHV OF THE HTL PRODUCTS

HTL product	HHV (MJ/kg)
Bio-oil	36.124
Bio-char	29.816

It can be seen that both the bio-oil and bio-char has a higher HHV than the original SCG, which had a HHV of 22.55 MJ/kg. The HHV of the bio-oil of 36.12 MJ/kg compares well with the HHV of 31 MJ/kg for bio-oil produced in another study using a batch reactor [11]. The HHV of the HTL bio-oil is, however, lower than that of the bio-oil obtained by solvent extraction.

D. GC-MS results

To study the difference between the different bio-oils, a GC-MS was done on each of the bio-oils that were extracted. This analysis showed the free fatty acids in the oils as well as the chain length of the carbon atoms in the oils. Table III shows the results from the hexane extracted oil.

TABLE II: COMPOSITION OF OIL EXTRACTED USING N-HEXANE

Component	Retention time (min)	Area (%)
n-Hexadecanoic acid	32.607	18.88
9,12-Octadecanoic acid (Z, Z)	35.72	8.53
Octadecanoic acid (Stearic acid)	36.086	3.11
9,12-Octadecanoic acid-2,3-dihydroxypropyl ester	54.589	0.74
9-Octadecanoic acid (Oleic acid)	35.682	6.57

The extracted bio-oil comprises of mainly C_{16} and C_{18} fatty acids. The most common component in the bio-oil is the hexadecanoic acid which makes up about 18.88% of the bio-oil composition. The rest of the oil comprises of isomers of octadecanoic acid. The same was done for the ethanol extracted oil but Table IV shows that the free fatty acids were a lot less in the bio-oil although the oil still comprised of mostly C_{16} and C_{18} fatty acids. When using the ethanol, it was also noted that caffeine was present in the composition, but the GC-MS result did not pick up on any noticeable ester that formed.

TABLE III: COMPOSITION OF THE ETHANOL EXTRACTED OIL

Component	Retention time (min)	Area (%)
Caffeine	29.562	2.15
n-Hexadecanoic acid	32.195	6.13
9,12-Octadecanoic acid (Z, Z)	35.038	3.71
9-Octadecanoic acid (Oleic acid)	35.128	1.65

The GC-MS results of the continuous HTL oil is shown in Table V. The oil comprises mostly of C_{16} and C_{18} fatty acids which is the same as most of the oils extracted using the reflux extraction technique.

TABLE IV: COMPOSITION OF THE OIL PRODUCED THROUGH HTL

Component	Retention time (min)	Area (%)
n-Hexadecanoic acid	35.001	40.41
9,12-Octadecanoic acid (Z, Z)	38.607	28.33
Octadecanoic acid (Stearic acid)	39.415	7.58
9,12-Octadecanoic acid-2,3-dihydroxypropyl ester	55.636	6.03
9-Octadecanoic acid (Oleic acid)	38.75	4.34

The oil produced using a continuous HTL reactor consists mainly of fatty acids. These fatty acids make out a lot more than what was found in the reflux extraction process. Hexadecanoic acid is the most common component in the HTL oil. The rest of the oil is comprised of octadecanoic acid and isomers thereof. The ester in this oil is more than esters found in the other extracted oils. The fatty acids produced in the HTL process is less than the fatty acids produced in the batch HTL process that was researched by Yang *et al.* [11]. The reason for this is the fact that the batch process used by Yang *et al.* [11] reached a higher temperature than the continuous process used in this study.

E. Oxidative stability of the oils.

The oxidative stability of any transport fuel as well as bio-oils aiming to produce transportation fuels are important. This is used to describe the degradation tendency of the fuel/oil. When the oils are oxygenized the formation of low molecular weight acids, aldehydes, ketones and alcohols can be formed. This is not favourable for the oils because the low molecular weight acids will increase the activity in the bio-oils which can lead to corrosion and the alcohols decrease the HHV and flash point of the bio-oils. Because oxidative stability analysis was run on renewable diesel standards the oxidative stability is very low, but this data can still be used in comparison with each other. The time for the oxidative stability is shown in Table VI.

TABLE V: OXIDATIVE STABILITY RESULTS

Bio-oil	Time (hr)
Acetone extracted oil	0.03
Ethanol extracted oil	0.04
Hexane extracted oil	0.11
HTL bio-oil	0.06

The oxidative stability analyser is calibrated for diesel products. Because of this the bio-oils that were sent through the analyser has a very low oxidative stability time. It is expected of the bio-oil to have a shorter time because the free fatty acids will oxygenise easily. The hexane oil had the longest oxidising stability time but there is no real difference in the times between the reflux oils and the HTL oil.

F. Proximate analysis

A proximate analysis was done on all of the bio-oils. The results are shown in Table VII.

TABLE VI: PROXIMATE ANALYSIS OF ALL OILS

	Hexane oil	Ethanol oil	Acetone oil	HTL oil
Total moisture (% mass fraction)	6.41	3.08	2.59	12.21
Volatiles (% mass fraction)	93.01	96.03	96.88	86.63
Ash(% mass fraction)	0.42	0.50	0.36	0.68
Fixed Carbon(% mass fraction)	0.17	0.40	0.18	0.48

The ash and fixed carbon values of all the oils were expected to be zero percent because the oils consists of mostly fatty acids and no solids. The values obtained for these variables are very low and can be because of experimental error. As expected the volatiles for each of the oils are very high and the moisture content is low. The low moisture content is because of the hydrophobic properties of the chain hydrocarbons that make up most of the oils.

IV. CONCLUSION

The study was done to compare bio-oils extracted and produced from SCG with each other. The maximum bio-oil yield when using the batch reflux extractions was 11.7% using hexane as a solvent and boiling the mixture for 2 hours. While using the pilot HTL plant a yield of 28.5% was obtained. This is more than double the yield that was obtained using batch extractions. The increase in yield sets the oil obtained from the continuous HTL process apart from the batch extraction process.

The energy content of the oils is very important and was investigated in depth in this study. The HHV of the batch extracted oils were higher than the HTL bio-oil. The HHV of the continuous HTL oil was very high in comparison to batch HTL process, which means that the continuous HTL process for bio-oil production is a very viable option. Further investigation is recommended into the hydrotreatment of the bio-oils to investigate if the hydrotreatment process can increase the energy content of the bio-oils.

SCG is a viable feedstock in the continuous HTL process and can be effectively used in the reflux extraction process. Quantitatively the continuous process is a lot better than the batch extractions using any of the three tested solvents. Qualitatively the batch extracted oils did have a higher energy content than the HTL oil but the HTL still had a very high HHV. The rest of the analysis showed that the oils are almost the same which means that the yield is the most important factor to consider.

Looking at all of the factors it is concluded that the continuous HTL process makes more sense and will yield the better bio-oil product.

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