

PRODUCTION OF PYROLYZED OIL FROM CRUDE GLYCEROL USING A MICROWAVE HEATING TECHNIQUE

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(Received: October 2015 / Revised: December 2015 / Accepted: January 2016)

ABSTRACT

Crude glycerol, a by-product of biodiesel production created via transesterification was pyrolyzed using a microwave heating technique in an oxygen-deficient environment. Coconut shell-based activated carbon was used as a catalyst to assist in the heat transfer and the cracking of glycerol into gaseous and liquid products. Investigation into the product yield was conducted by varying the pyrolysis temperature between 300°C and 800°C. The result revealed that liquid and gaseous pyrolysis products yield fell in the range of 15–42% and 55–82% by mass, respectively. An analysis of the liquid product using gas chromatography mass spectrometry (GC-MS) shows that glycerin (C₃H₈O₃), methanamine (CH₅N), and cyclotrisiloxane (C₆H₁₈O₃Si₃) were among the highest derived compounds in the pyrolyzed liquid yield. The derived pyrolysis products can potentially be used as alternative fuels in combustion systems.

Keywords: Activated carbon; Crude glycerol; Microwave; Pyrolysis

1. INTRODUCTION

The global production of biodiesel has been steadily increasing over the past decade. This trend is projected to further rise upward in the near future as biodiesel is expected to remain an important alternative fuel (Da Silva et al., 2009; Hasheminejad et al., 2011). The wide acceptance of biodiesel is due to its effectiveness as a supplementary fuel for diesel and its environmental benefits. Biodiesel is mainly produced via the process of transesterification, where fat or oil derived from living organisms reacts with alcohol (in the presence of a catalyst) in order to produce methyl esters (biodiesel) and glycerol. For every ton of biodiesel produced, approximately 0.1 ton of glycerol will be produced. Thus, increased production of biodiesel will eventually lead to an excess production of glycerol, which could lead to the oversupply of glycerol and create environmental problems if it is not disposed of properly. One potential solution is to convert crude glycerol into a useful product.

Thermo-chemical conversion methods can be used to convert glycerol into useful secondary products. Common methods include combustion, gasification, and pyrolysis. Combustion is a

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Permalink/DOI: <http://dx.doi.org/10.14716/ijtech.v7i2.2979>

process which involves the complete oxidation of a product, gasification involves high temperatures but only partial oxidation, while pyrolysis is a thermal degradation process that occurs in the absence of oxygen, typically performed by using conventional furnace heating and microwave heating techniques. The latter method heats more effectively and with a lower energy consumption when compared to the former. Microwave pyrolysis utilizes electromagnetic waves to transfer heat, and by heating the material internally at the molecular level, heat loss is minimized when compared to conventional heating methods (Fernández, 2011). During the pyrolysis process, primary and secondary pyrolysis can occur. Primary pyrolysis involves the process of creating dehydration, dehydrogenation, decarboxylation or decarbonization reactions. Secondary pyrolysis involves thermal or catalytic cracking where heavy compounds are further broken down into gases.

The pyrolysis temperature is one of the main parameters that can affect product yields. A previous study has shown that a higher pyrolysis temperature increases char and gas yields (Domínguez et al., 2008). Pyrolyzed liquid shows a maximum yield at an intermediate temperature but it decreases at higher temperatures due to the thermal cracking of heavy compounds into small-chain products. Solid residue structures can be modified by pyrolyzing at a higher heating rate in order to increase the liquid and gaseous fraction (Menéndez et al., 2007).

A microwave pyrolysis method has been successfully used to pyrolyze a variety of feedstock including waste engine oil (Lam et al., 2010; Lam et al., 2016), wheat straw (Budarin et al., 2009), scrap tires (Appleton et al., 2005; Undri et al., 2013), oil palm biomass waste (Salema & Ani, 2011) and waste plastic (Russell et al., 2012). Valliyappan et al. (2008) performed a study on the pyrolysis of glycerol for the production of hydrogen or syngas by using a conventional heating method. Their findings show that glycerol was converted to a gaseous product containing 93.5% syngas with an H₂/CO ratio of 1.05 at 800°C under the carrier gas flow rate of 50 mL/min.

Fernández et al. (2009) pyrolyzed glycerol with activated carbon acting as a catalyst for the production of syngas. The yields of syngases created when using furnace heating and microwave heating methods were compared. Bituminous carbon and coconut shell-based activated carbon were used to study the effects of the catalyst on syngas production, separate from varying the heating temperature. The results reveal that a carbonaceous catalyst improved the yield of synthesis gas up to 81% by volume. The microwave heating method was more favorable for the production of synthesis gas. By heating up to 800°C, syngas yield was found to be the highest.

The present study focuses on an investigation into the pyrolyzed liquid product derived from employing the microwave-assisted pyrolysis method. In it, the effects of carrier gas flows, catalysts, and temperatures on the optimum operation conditions are determined. The derived liquid product is characterized by using gas chromatography-mass spectroscopy (GC-MS) and Fourier transform infrared spectroscopy (FT-IR).

2. EXPERIMENTAL SETUP

2.1. Material

The crude glycerol used in the present experiment was supplied by Caratino Malaysia Sdn Bhd. The specifications of the material are shown in Table 1. The coconut shell-based activated carbon supplied by Concept Ecotech Sdn. Bhd. was used as a catalyst. The surface area of the activated carbon is in the range of 1000–1150 BET-m²/g. The specifications of the activated carbon are shown in Table 2.

Table 1 The specifications of the crude glycerol that was provided by Carotino Malaysia Sdn Bhd.

| Parameters | Specification | Result |
|-------------|---------------|--------|
| Glycerol, % | 85 min | 86 |
| Moisture, % | 10 max | 6.3 |
| Ash, % | 5 max | 4.2 |
| MONG | 8 max | 3.5 |

MONG : Matter organic non-glycerol

Table 2 The specifications of the coconut shell-based activated carbon

| Properties | Value |
|--------------------------------------|-----------|
| Bulk density (g/ml) | 0.48-0.50 |
| CTC adsorption (%) | 50 |
| Iodine no (mg/g) | 1000 |
| Surface area (BET-m ² /g) | 1000-1150 |
| Pore volume (cc/g) | 0.44 |
| Moisture content (%) | <5% |

2.2. Apparatus

The pyrolysis test setup is shown in Figure 1. A quartz reactor with a diameter of 100 mm and a height of 180 mm was used to pyrolyze the glycerol. The quartz reactor is connected to a nitrogen line supply while the reactor outlet is connected to a condenser. The reactor is placed inside a single magnetron microwave (Samsung ME711k) that is able to provide a maximum power output of 800 watts. A thermocouple is placed inside the reactor to measure the feedstock temperature and to provide a feedback signal to control the heating. Nitrogen is supplied to the reactor to purge the reactor of air containing oxygen.

The pyrolysis experiment was performed at an elevated temperature of between 300°C and 800°C by using microwave heating. Glycerol was mixed with activated carbon in the reactor. Pyrolyzed gases flowed out of the outlet neck and passed through the condenser that used room temperature cooling water (25°C). The condensed liquid product was then collected using a conical flask.

2.3. Method of Analysis

A GC-MS was used to analyze the liquid products obtained from the experiment. The GC-MS analysis was conducted using an Agilent HP 6890 gas chromatograph and an HP 5973 mass selective detector. The liquid products were analyzed with the Agilent 19091s-433 30m×0.25µm×250µm, with an inlet pressure of 9.08 psi and split ratio of 20:1. The oven temperature was set to a constant 40°C for 2.0 minutes, then ramped to 290°C at 5°C min⁻¹. The peaks were identified by using the NIST 2008 mass spectral library. The liquid product was further analyzed using a Fourier transform infrared spectroscopy (FT-IR), where the possible functional groups existing in the liquid product were determined. Spectra were recorded between 400 cm⁻¹ and 4000 cm⁻¹ with a resolution of 4 cm⁻¹.

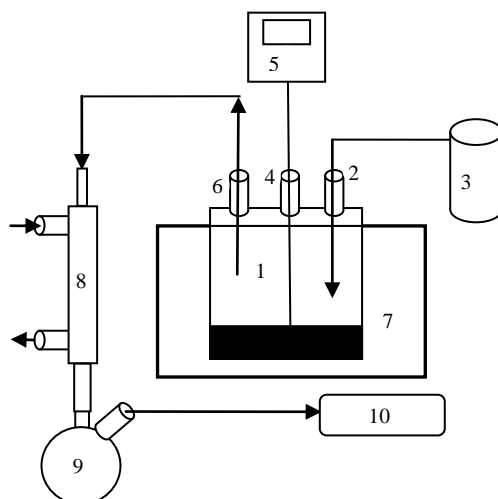


Figure 1 Experimental setup for glycerol pyrolysis

1. Quartz reactor; 2. Nitrogen inlet neck; 3. Nitrogen tank; 4. Temperature indicator neck; 5. Temperature indicator; 6. Outlet neck; 7. Microwave; 8. Condenser; 9. Conical flask (liquid collector); 10. Gas sampling bag

3. RESULTS AND DISCUSSION

3.1. Effect of the Catalyst

Coconut shell-based activated carbon was used as the catalyst to assist in the heat transfer and thermal cracking process during pyrolysis. A comparison of the pyrolysis process was performed at heating temperatures of 300°C and 400°C. Figure 2 shows the resulting product yields for the pyrolysis process both with and without the catalyst. Overall, the presence of the catalyst in crude glycerol pyrolysis resulted in an increase in liquid product and a decrease in gaseous product at both heating temperatures. At 300°C, the use of the catalyst decreased the gaseous product by 4.2%, but increased the liquid product by 9.2%. The difference was more significant when the heating temperature was raised to 400°C and the gaseous product was reduced by 19% while the liquid product increased by 22%. The solid product was reduced when the catalyst was used at both temperatures, albeit by less than 5%.

The function of a catalyst is to increase heat transfer and assist in the thermal cracking process since a more complete cracking of the molecules with fewer solid products can be achieved by using a catalyst (Domínguez et al., 2003; Dominguez et al., 2007). Previous studies have shown that adding a catalyst can increase the liquid yield and improve the quality of the liquid product (Bridgwater, 1996; Wang et al., 2006).

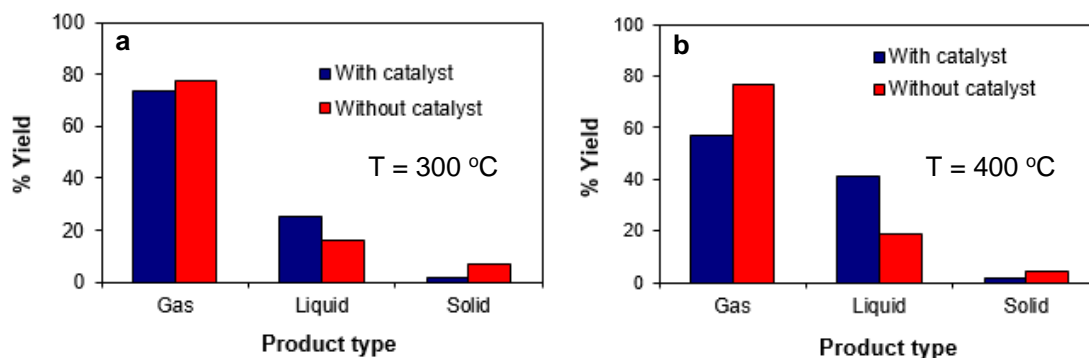


Figure 2 Product yield for pyrolysis with and without the presence of the catalyst at: (a) 300°C; and (b) 400°C

3.2. Effect of Temperature

The heating of the crude glycerol inside the quartz reactor was performed using microwaves. Coconut shell-based activated carbon was utilized as a catalyst to assist in elevating the pyrolysis temperature and facilitate thermal cracking. The ratio of catalyst to feedstock was fixed at 1:1. The pyrolysis process was tested in the temperature range of between 300°C and 800°C. The supply of nitrogen carrier gas flow was fixed at 33.3 g/s. Figure 3 shows the pyrolysis product yields (gaseous, liquid and solid) at a range of temperatures. At 300°C, the converted gaseous product was about 77%. The yield decreased to 67.5% when the heating temperature was raised to 400°C. The decrease in gaseous yield resulted in a corresponding increase of liquid product by 9%.

The highest liquid product yield was obtained at the heating temperature of 400°C. Further increase to the pyrolysis temperature resulted in a decrease of liquid product. At 700°C, the liquid product yield was 15.7% while the gaseous product yield was 83%. The results show that the yields for both liquid and gaseous products leveled off at 800°C with 16.2% and 82.1% respectively. The increase in pyrolyzed gaseous product at increasing temperatures is due to a higher degree of devolatilization and cracking. The higher amounts of energy imparted at elevated temperatures initiates a secondary cracking process, thereby leading to the differences in yield (Encinar et al., 2000; Maher & Bressler, 2007). Pyrolyzed solid products remain relatively insignificant at <2% of the total weight.

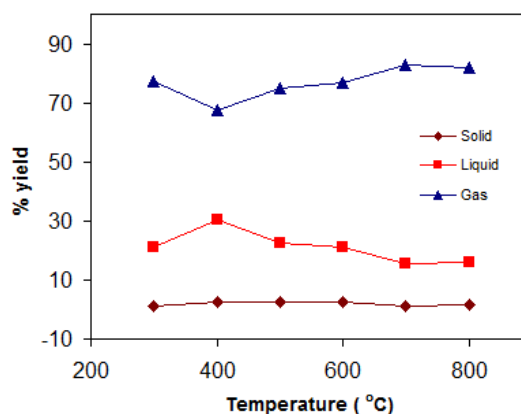


Figure 3 The pyrolysis product yield at a range of temperatures

3.3. Effect of Carrier Gas Flow Rate

Nitrogen as a carrier gas of was continuously supplied to the quartz reactor during the pyrolysis process to purge the gas products. The effect of the carrier gas flow rate on the product yield was investigated. Two nitrogen flow rates were used, i.e. 33.3 g/s (2 L/min) and 66.7 g/s (4 L/min), and these were supplied continuously and at constant rates throughout the pyrolysis process.

Figure 4 shows the pyrolysed product yield. For 33.3 g/s (2 L/min) of nitrogen flow rate, the highest liquid product yield was obtained at a heating temperature of 400°C. Increasing the pyrolysis temperature was found to result in a decrease in liquid product. The gaseous product reveals a reverse trend when compared to the liquid product. At 400°C, the gaseous product yield was lowest but it increased along with the rising temperature.

The increased N₂ supply to 66.7 g/s (4 L/min) resulted in different yield profiles. The liquid product yield was at its peak at 300°C, but it initially decreased as the temperature increased to 700°C, before slightly rising again at 800°C. Correspondingly, the gaseous product yield

increased along with the temperature and peaked at 700°C before slightly decreasing by 6% at 800°C.

A comparison of the product yield in Figure 4 shows that the effect of carrier gas flow rate on the product yield is significant. At 400°C, the difference in product yield is most evident between the two carrier gas flow rates that were compared. Gaseous product shows a difference of 9.5% whereas liquid product shows a difference of 9.9%. Liquid product yield is significantly lower at a higher N₂ flow rate. Varying the carrier gas flow rate changes the residence time of the products during the pyrolysis process, as the N₂ flow rate changes the distribution of carbon inside the reactor. A lower flow rate of carrier gases provides ample residence time for the product to undergo the chain-cracking process that leads to secondary reaction (Fernández et al., 2009; Khaghanikavkani & Farid, 2010). Furthermore, the higher N₂ flow rate reduces the time available for gaseous products to undergo the condensation process. As a result, the pyrolysis product remains largely as gaseous product (approximately >80%).

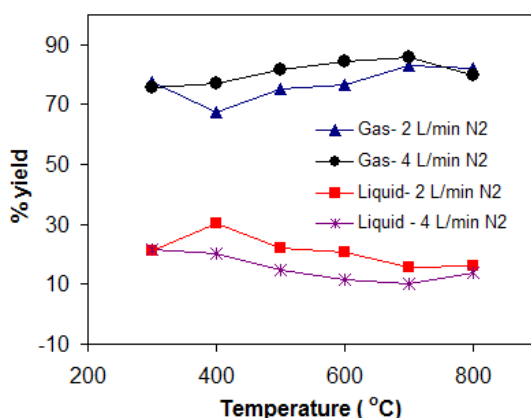


Figure 4 Pyrolyzed product yields using carrier gas flow rates of 2.0 L/min and 4.0 L/min

3.4. Characterization of Liquid Product

3.4.1. FT-IR analysis

The functional groups present in the liquid product are reflected by the characteristic absorption peaks revealed during the FT-IR analysis (Lambert et al., 1998). Figure 5 shows the FT-IR spectra of the liquid product obtained by undergoing the pyrolysis process. Each valley in the curve represents a functional group. The liquid product comprises a range of compounds from different groups. Table 3 shows the classes of compounds that exist in the liquid product. The significant peaks that present between the wave numbers of 3200–3600 cm⁻¹ could be that of O-H stretch. The stretch vibration indicates compounds such as water, phenol and polymeric compound impurities. The presence of alcohol and phenol are indicated in the range of wave numbers of 950–1300 cm⁻¹. This range includes the presence of ester and ethers.

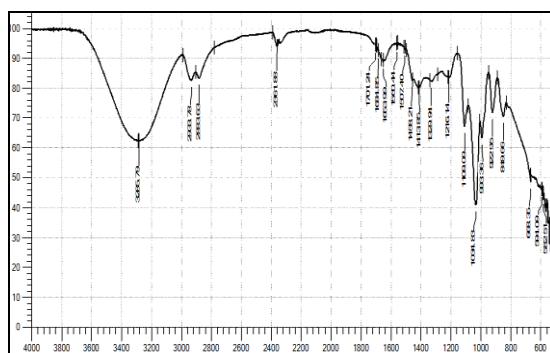


Figure 5 The FT-IR spectra for the liquid product

Table 3 The FT-IR analysis results for the pyrolysis process

| Wave number (cm ⁻¹) | Functional group | Compound class |
|---------------------------------|------------------|--------------------------------------|
| 3200-3600 | O-H | Polymeric, water impurities, phenols |
| 3000-2800 | C-H | Alkanes |
| 1780-1640 | C=O | Ketones, aldehydes, carboxylic acids |
| 1680-1580 | C=C | Alkenes |
| 1475-1350 | C-H | Alkanes |
| 950-1300 | C-O | Primary, secondary, and tertiary |
| | O-H | alcohols, phenol, ester, ethers |
| 900-650 | O-H | Aromatic |

3.4.2. GC-MS analysis

The pyrolyzed liquid products obtained were analyzed using a GC-MS to identify the derived compounds. Table 4 lists the chemical name, structural formula and percentage area covered in the GC graph. The percentage area covered by each compound is a qualitative indication of the concentration in the total liquid product (Bhattacharya et al. 2009). The glycerin concentration is the highest at approximately 42%, followed by methanamine at 10.2%. The high percentage of glycerin demonstrates that the conversion into gaseous or smaller-chain liquid product is incomplete. It is worth noting that the percentage of the glycerin in the final liquid product is relatively less, considering that most of the crude glycerol has already been pyrolyzed into a gaseous product (~80%) at an elevated temperature of 700°C. Cyclotrisiloxane and N-Methoxy-N-methylacetamide occupy the areas of 3.73% and 3.51%, respectively.

Table 4 Compounds in the liquid product derived at a pyrolysis temperature of 700°C

| Compound | Formula | % Area |
|--|---|--------|
| Glycerin, Methanamine | C ₃ H ₈ O ₃ , CH ₅ N | 41.92 |
| Methanamine | CH ₅ N | 10.16 |
| Cyclotrisiloxane | C ₆ H ₁₈ O ₃ Si ₃ | 3.73 |
| N-Methoxy-N-methylacetamide | C ₄ H ₉ NO ₂ | 3.51 |
| Octasiloxane | C ₁₈ H ₅₄ O ₇ Si ₈ | 1.95 |
| 1,2-Benzisothiazol-3-amine tbdms | C ₇ H ₅ NOS | 1.63 |
| Tetrasiloxane | H ₁₀ O ₃ Si ₄ | 1.41 |
| Silicic acid | H ₄ O ₄ Si | 1.19 |
| L-Arabinitol | C ₅ H ₁₂ O ₅ | 1.19 |
| Benzo[h]quinoline | C ₁₃ H ₉ N | 0.61 |
| Thiocyanic acid ethyl ester | C ₃ H ₅ NS | 0.50 |
| Sulfide sec-butyl cyclopentyl | C ₉ H ₁₈ S | 0.44 |
| 2(R),3(S)-1,2,3,4-Butanetetrol | C ₄ H ₁₀ O ₄ | 0.31 |
| 1,3-Dioxolane,2-ethyl-4-methyl-2-Oxazolidone | C ₆ H ₁₂ O ₂ | 0.30 |
| 2-Methyl-7-phenylindole | C ₁₅ H ₁₃ N | 0.30 |
| 2-Cyclopenten-1-one | C ₅ H ₆ O | 0.29 |
| 4-(3Dimethylaminopropoxy)benzaldehyde | C ₁₂ H ₁₇ NO ₂ | 0.29 |
| Silane | SiH ₄ | 0.25 |
| Phenol | C ₆ H ₆ O | 0.25 |
| Propylene Glycol | C ₃ H ₈ O ₂ | 0.24 |

4. CONCLUSION

In this study, crude glycerol was pyrolyzed into gaseous and liquid products using a microwave heating technique. The presence of a coconut shell-based catalyst assisted in the heat transfer and elevated the pyrolysis temperature to 800°C. Evidently, pyrolysis temperature affects the product yield. The liquid product yield was highest at the pyrolysis temperature of 400°C, whereas gaseous product peaked at 700°C. Inert carrier gas flow is another important factor that affects the product yield. Low carrier gas flow rate increases the residence time for pyrolysis, thus allowing for secondary cracking into smaller compounds to occur. The liquid product yield was found to increase when a lower carrier gas flow was used and this can be explained by an enhanced condensation rate. A variety of liquid compounds were derived from the pyrolysis process and the GC-MC analysis showed that glycerin, methanamine, and cyclotrisiloxane were among the most commonly derived components in the liquid product.

5. ACKNOWLEDGMENT

Financial support from the Malaysian Ministry of Education and Universiti Teknologi Malaysia (Research university grant Tier-1 vot no.: 09H79) is gratefully acknowledged.

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