EVALUATION OF UBI GAJAH FOR BIOETHANOL VIA HYDROLYSIS AND FERMENTATION

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ABSTRACT

This research was carried out to evaluate utilisation of Ubi Gajah (*Manihot esculenta*), a non-edible cassava species, as a feedstock for bioethanol production by acid hydrolysis. The Ubi Gajah peels and pulp substrates were hydrolysed for a max. of 48 hrs with H_2SO_4 acid concentration ranges 5% v/v to 15% v/v at high temperature and pressure before fermentation process. While the yield was noted at 24 hr of 15% v/v acid hydrolysis. From the experimental results yield of bioethanol for Ubi Gajah Peel and Pulp 156.65 g/L and 220.89 g/L were noted respectively when both the substrates were hydrolysed with 15% v/v of H_2SO_4 . The FTIR Spectra of the bioethanol, confirms –OH, C-O and C=C groups by absorption bands at 3251.98 cm⁻¹ to 3315.63 cm⁻¹; 1045.42 cm⁻¹ and 1085.92 cm⁻¹; and 1633.71 cm⁻¹ and 1645.28 cm⁻¹ respectively. The biofuel properties were tested according to ASTM standards and found to be complacent. The Bioethanol - diesel blends (BDB) of 5% to 20% v/v were prepared and fuel performance test was conducted on a diesel engine. The performance and emission results confirm suitability of the bioethanol as an alternative fuel.

Key words: Bioethanol, Ubi Gajah, acid hydrolysis

INTRODUCTION

Energy is one of the most fundamental essential needs of the life (Shafiee & Topal, 2009). It is reported that fossil fuel reserves continue to diminish while the global energy demand and consumption is growing, which is leading unto the global warming (Suranovic, 2013). Consequently, many efforts have been done to introduce alternatives of energy sources such as liquid biofuels which captured the attention of researchers, policymakers as well as the consumers (Orian-Research, 2018). The liquid biofuels such as bioethanol and biodiesel are derived from organic substances, which offers a better alternative to reduce the consumption of Petro fuels and environmental pollution. Since the level of oxygen in biofuels ranges from 10% to 45%, the fuel combustion becomes more efficient yet lower the hydrocarbons in exhaust emission (Demirbas, 2009). Table 1 shows some of the common feedstocks for bioethanol production (OECD/Food & Agriculture Organization of the United Nations, 2015).

The fuel blends of gasoline and ethanol are widely used in transportation sector. The most common ethanol-gasoline blend is known as "gasohol" or E10, a blend of 10% ethanol mixed with 90% gasoline. The blend of ethanol and gasoline will result in higher fuel consumption. This is because of the energy content in the blend. E10 has 3.3% less energy content per gallon than gasoline meanwhile, E85 has 24.7% less energy per gallon than gasoline (EIA, 2016). Ethanol-diesel blend, named as E-diesel which contains up to 15% v/v of ethanol and 0.2% v/v to 5.0% v/v of additive which is responsible to maintain the blend stability and certain fuel properties such as Cetane number, corrosion inhibition and lubricity. Such additive is very important because E-diesel blends is not stable and will separate after sometimes (Park et al., 2010). E-diesel fuels are still under testing and previous research has shown that the blends may reduce certain components of exhaust emission especially

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Feedstock properties	Examples
Direct sugar sources	Sugar beets, sugarcane, fruits, molasses
Starchy crops	Corn, wheat, cassava, potato, barley
Cellulosic or woody materials	Sawdust, straw, corncob, poplars

Table	1.	Feedstocks	for	bioethanol	production
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Source: (OECD/Food and Agriculture Organization of the United Nations, 2015).

when bioethanol and biodiesel are used in the blend (Orian-Research, 2018).

Ubi Gajah is a non-edible Cassava species scientifically known as *Manihot esculenta*. This is one of the abundantly available bioethanol feedstocks in the Malaysian region. According to our survey, very limited research has been conducted on Ubi Gajah as a bioethanol feedstock. This research focuses on production of bioethanol using Ubi Gajah following acid hydrolysis process. Also, the diesel engine performance and exhaust gas emissions are proposed to evaluate fuelled with bioethanol - diesel blends (BDB) of different bioethanol volumes. The biofuel properties will be tested according to ASTM standards.

MATERIALS AND METHODS

Naturally grown raw Ubi Gajah was obtained from a local village, Jalan Jambusan Bau, located in Sarawak, Malaysia. The Ubi Gajah pulp and peels were separated manually, chopped into smaller pieces and were blended using a heavy-duty professional electric blender (Omni Blend V, Imbaco, Australia). The samples were then divided equally and kept in different beakers for further experimentation. Chemicals that include H₂SO₄ (95-97%) and instant yeast (*Saccharomyces cerevisiae*) were drawn from the laboratory stocks while all the experiments were conducted at Energy lab, Faculty of Engineering, Universiti Malaysia Sarawak (UNIMAS), Malaysia.

Pre-treatment and acid hydrolysis

Acid pre-treatment was carried out by adding H_2SO_4 of 5% v/v, 10% v/v and 15% v/v into the substrate samples as prepared. The mixtures were autoclaved for 3 hr and the hydrolysed solutions were then filtered using filter paper and glass funnel, while, the glucose concentration in each sample solution was measured using pocket refractometers.

Fermentation and bioethanol production

The fermentation was carried out in a standard 250 mL shake flask. Firstly, the instant yeast (*Saccharomyces cerevisiae*) was cultured in Yeast Extract-Peptone-Dextrose (YPD) medium utilising

10 g of yeast extract, 20 g of peptone, 20 g of glucose and 1 L of water were mixed. The solution was autoclaved for 2 hr and refrigerated for further use. For restoring the yeast, rehydration process was carried out by adding a measured amount of 10 mL YPD medium. The rehydrated yeast mixture was then inoculated into measured amount of YPD medium for further incubation at room temperature in an orbital shaker for 20 hr at 150 r.p.m. Further, fermentation was carried out by mixing YPD and hydrolysed solution at 1:2 ratio. Then the fermented medium was thoroughly mixed employing an orbital shaker for 48 hrs with a rotational speed of 150 r.p.m at room temperature. Periodically, the solution sample were withdrawn to measure the glucose and bioethanol concentration utilising pocket refractometers.

Analysis of bioethanol fuel properties

The samples of bioethanol produced were tested to confirm their functional group following Fourier Transform Infrared Spectroscopy (FTIR) analysis (Perkin Elmer, 100 series) over wavelengths $4000 \text{ cm}^{-1} - 750 \text{ cm}^{-1}$. Also, the produced bioethanol fuel properties were tested according to the ASTM standards.

Engine performance and gas emission analysis test using bioethanol and diesel blends

Bioethanol - diesel blends (BDB) of different ethanol volume (5%, 10%, 15%, and 20% v/v) were prepared by using the produced bioethanol and Shell Malaysia diesel fuel. Pure diesel and BDB fueled TNM-TDE-700, Techno-mate, diesel engine was used to test the fuel performance. The engine testing was carried out three times successively for 20 mL fueling of diesel and each BDB while the output parameters were recorded to calculate the engine Torque, Fuel Consumption Rate, Engine Power, Specific Fuel Consumption, Brake Horsepower and Indicated Horsepower at 120 N and the speed of the throttle was set to 1/2. The exhaust gas emissions such as CO, NO, NO₂, NO_X and SO₂ were measured by using a Digital Portable Gas Analyzer (Measuring range 0-100 ppm, Precession 2% F.S, with response time of 10 Sec). Approximately 3 mL of fuel was poured in an evaporating dish. The end of a small piece paper was dipped in the fuel while the other



Fig. 1. Solutions of Ubi Gajah (a) Before hydrolysed (b): After Hydrolysed and filtered using 5%, 10% and 15% v/v H₂SO₄.

Hydrolysed SolutionsGlucose Concentration (g/L) $5\% \text{ v/v } \text{H}_2\text{SO}_4$ $10\% \text{ v/v } \text{H}_2\text{SO}_4$ $15\% \text{ v/v } \text{H}_2\text{SO}_4$ Ubi Gajah Peels110.28168.30212.80

236.00

175.00

Table 2. Glucose concentration after acid hydrolysis process

end was burned. Once the fuel started to burn, the analyser tip was pointed in the smoke from the burning fuel. The results generated by the device were recorded.

Ubi Gajah Pulp

RESULTS AND DISCUSSION

Analysis of Ubi Gajah pre-treatment and acid hydrolysis

The treated substrate solutions of three different concentrations after 3 hr of hydrolysis turned to reddish brown can be seen in Figure 1(a) and (b). It is observed that Higher the concentration of H_2SO_4 acid used to soak the substrate and pre-treatment, darker the colour of the solution obtained. The initial solutions obtained smells unpleasant and strong whereas, after hydrolysis and filtration, the solutions are odourless and free from impurities as shown in Figure 1(b).

Glucose concentration in the hydrolysed samples

The acid hydrolysis process converted all starch extracted from both Ubi Gajah Peels and Pulp into the glucose, but a little degradation in the product was observed. However, it is known that dilute acid hydrolysis of cellulose at high-temperature results in yields not exceeding 60-65% of the potential glucose. Table 2 shows the measured glucose concentration in each hydrolysed solution. It can be distinctly seen that higher the concentration of acid used to hydrolyse the substrate, higher the glucose produced. Also, from the tabulated results it can be deduced that more glucose was produced from the Ubi Gajah Pulp compared to Ubi Gajah Peels. Further, it can be inferred that Ubi Gajah pulp can produce higher glucose due to rich in fermentable sugars and starch compared to its peels which comprised of a large portion of lignocelluloses and a little amount of sugars. Also, the concentration of acid used in the hydrolysis process has affected the amount of glucose released by the substrates.

262.00

The results of glucose consumption in the fermentation of Ubi Gajah peels and pulp using 5%, 10% and 15% v/v H_2SO_4 were shown in Figure 2. In fermentation process, yeast consumes and converts the glucose to ethanol, and carbon dioxide as a side product. The highest glucose concentration of 212 g/L and 264 g/L was noted for Ubi Gajah peels and Ubi Gajah pulp respectively at the beginning of fermentation. Further it can be observed that glucose concentration in the samples is decreasing with increase in fermentation time. This indicates that the fermentation process is continuously happening but especially after 18 hr the consumption rate is slowing down.

Bioethanol production analysis

Figures 3 shows the graph of bioethanol production from Ubi Gajah peels and pulp using 5%, 10% and 15% v/v H_2SO_4 . From the results it is evident that at the bioethanol production rate was stagnant after 24 hr and at the end of the 48 hr, glucose was still present in all samples even though bioethanol produced was at the maximum level. Yeast is a living organism that requires optimum conditions to survive. High sugar concentrations encountered immediately after hydrolysis exert osmotic stress on yeast which may results in a longer lag phase at the beginning of fermentation. On the



Fig. 2. Glucose consumption in the fermentation of (a) Ubi Gajah peels (b)Ubi Gajah pulp using 5%, 10% and $15\% \text{ v/v} \text{ H}_2\text{SO}_4$.



Fig. 3. Bioethanol production during the fermentation of (a) Ubi Gajah peels (b) Ubi Gajah pulp using 5%, 10% and 15% v/v H₂SO₄.

contrary, ethanol is another inhibitory to yeast at high concentration by disrupting the integrity of the cell membrane (Alleman *et al.*, 2015; Muhaji & Sutjahjo, 2018) therefore, normal yeast strain may only be able to tolerate 12 - 15% of ethanol concentration. This explains the reason of inactivity in the fermentation process after passing 24 hr. No glucose can be converted to ethanol once the ethanol concentration exceeds yeast's ethanol tolerance (Muhaji & Sutjahjo, 2018). Also, the concentration of both glucose and bioethanol remained at the same level even though the fermentation period was extended.

Bioethanol characterisation using FTIR spectroscopy

The FTIR spectra of two Ubi Gajah samples (15% v/v peels and pulp solution) in which the ethanol concentrations had been diluted to 10%

shown in Figure 4. For the Ubi Gajah peels sample, Figure 4(a), the absorption band that showed a broad strong band range from 3251.98 cm⁻¹ to 3315.63 cm⁻¹ was assigned to stretching of -OH groups. In agreement with previous study by Doroshenko et al. (2013), the peels sample was confirmed to be in the alcohol functional group. However, the type of alcohol could not be clarified. since, in discriminable presence of C-O group from the spectral graph stretching in the range of 1260 cm⁻¹ to 1000 cm⁻¹. The medium narrow peak that could be noticed at 1633.71 cm⁻¹ was assigned to C=C stretching of an alkene group. While for Ubi Gajah pulp sample, Figure 4(b), the broad strong absorption band at 3304.06 cm⁻¹ was assigned to stretching of -OH groups, instantaneously confirmed the sample as an alcohol. The narrow bands of C-O stretch at 1045.42 cm⁻¹ and 1085.92 cm⁻¹ shows that the sample belongs to primary alcohol groups and





Fig. 4. FTIR spectra of bioethanol produced using (a) Ubi Gajah peels (b)Ubi Gajah pulp.

ethanol was a primary alcohol. Meanwhile, the presence of medium bands at 1633.71 cm⁻¹ and 1645.28 cm⁻¹ were set to C=C stretching of an alkene group. The readings were comparable with published literature (Doroshenko *et al.*, 2013).

Bioethanol fuel properties analysis

The fuel properties of Bioethanol produced from both Ubi Gajah peels and pulp samples were tabulated in Table 3. The fuel produced using peels sample is denser than the pulp sample, but both samples' temperatures are differing. Bioethanol

Table 3. Properties of U	oi Gajah pulp	and peels samples
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Bioethanol sample	Density (g/cm ³)	Temperature (°C)
Ubi Gajah peels	0.9941	28.1
Ubi Gajah pulp	0.9926	26.3

sample generally has a density that is easily affected by the surrounding temperature and pressure. Alterations in these factors will result in change in the volume of the Bioethanol, which in turn results

Engine Performance Indicator	Diesel	5% BDB	10% BDB	15% BDB	20% BDB
Torque (Nm)	36	36	36	36	36
Fuel Consumption Rate (mL/s)	0.1009	0.104	0.1067	0.1079	0.1089
Engine Power (kW)	0.585	0.5527	0.5053	0.4894	0.4739
Specific Fuel Consumption (mL/kW)	34.188	36.186	39.5804	40.8664	42.203
Brake Horsepower (kW)	4.2675	4.113	4.047	4.0282	4.0093
Horsepower (kW)	57.80	57.80	57.80	57.80	57.80
Mechanical Efficiency (%)	92.6268	92.8841	92.9983	93.0308	93.0635

Table 4. Diesel Engine Performance indicators

Table 5. Gas Emitted during the combustion of E-diesel fuel blends

			Types of gas emitted (ppm)				
Fuel	FT (°C)	CO	NO	NO ₂	NO _X	SO ₂	
Diesel	110	40	1	0.4	1.04	3	
5% BDB	107.8	45	1	0.3	1.03	1	
10% BDB	89.4	51	1	0.2	1.02	1	
15% BDB	79.7	62	1	0.2	1.02	1	
20% BDB	79	73	1	0	1	0	

in change of fuel density. Besides, the density of a distilled ethanol depends on the ratio of alcohol and water present. Therefore, even though the samples have the same ethanol concentration, their densities can be different from each other.

The density of a pure ethanol is 0.789 g/cm^3 at a temperature of 20°C. By referring to the published results data by John Aurie and Lange (1999), the peels sample's density indicates that the sample has about 4% v/v of ethanol while Ubi Gajah pulp sample ethanol content is about 6% v/v (John Aurie & Lange, 1999). The samples were diluted to have 10% v/v of ethanol, but there is a mismatch between the measured ethanol concentration and the ethanol content based on the densities determined. The possible reason is the accuracy of the 'pocket' refractometer in measuring ethanol concentration (Matuszewska *et al.*, 2013).

Diesel engine performance analysis

The properties determining the fuel performance on a diesel engine were presented in Table 4. For each testing condition, the volumetric fuel flow rate was measured to determine the fuel consumption rate. In terms of mechanical efficiency and Specific Fuel Consumption (SFC), Petro diesel is less efficient compared to the E-diesel fuels (BDB). The mechanical efficiency and SFC were increasing with percentage of Bioethanol added to the petro diesel fuel. A highest mechanical efficiency of 93.06% and SFC of 42.20 mL/kW were measured with 20% BDB respectively. On the contrary, Brake Horsepower (BHP) and Engine Power (EP) were declining with increase in Bioethanol volume in the fuel blends. Thus, the higher the ethanol content, the greater the fuel consumption will be. However, ethanol-diesel blends decreased the power of diesel engine without modification. This will lead to an increase in the specific fuel consumption (Matuszewska *et al.*, 2013). The engine performance indicators obtained were following the reported results (Park *et al.*, 2010, 2012; Gomasta & Mahla, 2012; Mehta *et al.*, 2013).

Analysis of exhaust gas emission results

The measured values of exhaust gases during the Combustion of E-diesel Fuel blends were tabulated in Table 5. Based on the results, E-diesel blends emitted lower SO₂ and NO_X gases. NO_X is the total concentration of both NO and NO₂ gases emitted. According to the results, the emission of NO_X blends decreased with percentage of ethanol content in the fuel. The increasing oxygen content from the ethanol can promote the formation of NOx but the maximum gas temperature is the most important factor of NOx formation. As can be seen from the tabulated results, the fuel temperature has been decreased with increase of bioethanol e volume in the blend. Further, the decreased gas temperature caused by higher latent heat of vaporisation of ethanol can reduce the NOx emission (Lei et al., 2011). Petro diesel has lower CO emission compared to the blended fuels. However, in BDB fueled

engines, the amount of CO gas emission was increased with the increase in volume of ethanol added to the blend. Ethanol-diesel blends would not deteriorate CO emissions except for certain engine load. The addition of ethanol causes the reduction of gas temperature, which restrains the oxidation of CO, and hence causing CO emission goes up when engine load is low. As the emission test was done by only burning the fuel, load is not certain and considered free and low which has resulted in greater CO emission. The experimental results comply with the published results (Park *et al.*, 2010; 2012; Gomasta & Mahla, 2012).

CONCLUSIONS

This experimental work concludes that Ubi Gajah is one of the promising alternative non-edible feedstocks for bioethanol production. The Acid hydrolysis process was tested on conversion of Ubi Gajah Peels and Pulp to Glucose which is adequate for fermentation. It is found that the acid concentration significantly affects the concentration of glucose produced. The higher the concentration of H₂SO₄ for hydrolysing the Ubi Gajah, the higher gain in the glucose concentration as well as yield of bioethanol from the fermentation process. This has enhanced the bioethanol production using Ubi Gajah Peels and Pulp from 83.22 g/L to 156.65 g/L and 72.42 g/L to 220.89 g/L respectively. The concentration of Bioethanol produced process depends on the fermenting yeast and also, yeast's low ethanol tolerance limits the bioethanol production. The diesel engine performance results indicate that, even though the blending has better mechanical efficiency and cleaner gas emission, fuel consumption rate is high which is ineffectual.

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