Process Optimization and Characterization of Bioethanol from Yam (*Dioscorea rotundata*) Peels

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Abstract- Today, fossil fuels are been used for both domestic and commercial energy needs in the World. Environmental effects, coupled with the depletion issue of these fossil fuels has led to the search for alternative energy sources. Bioenergy is one of these alternative energy resources. It represents the production of sustainable chemicals and fuels by utilizing biomass as starting raw material. Bioethanol and biogas are the common fuels that are obtained from biomass. The present study is aimed at optimizing the production process and characterizing bioethanol produced from yam peels. Different hydrolysis temperatures and times were used to determine the temperature and time that could give an optimal yield of glucose. The results revealed that hydrolysis temperature of 110 ^{0}C and hydrolysis time 180 minutes gave the optimal yield of glucose. This temperature and time were then used for the production of bioethanol. Results obtained revealed that two hundred grams of the sample gave yields of 180 mL after fermentation for 5 days. The bioethanol produced from yam peel was characterized to determine its density, specific gravity, refractive index, sulphur content, flash point and cloud point. Results obtained revealed that the properties of the bioethanol produced correspond to the properties of bioethanol recommended by the American society for testing and materials (ASTM).

Index Terms- bioethanol, biomass waste, hydrolysis, fermentation.

I. INTRODUCTION

Today, fossil fuels are been used for both domestic and commercial energy needs in the World[1]. Environmental effects, coupled with depletion issue of these fossil fuels has led to the search of alternative energy sources[2]. There has been changes in the sources of energy in the globe for more than two decades. The utilization of wood as biomass resources dated back to when mankind used wood to either keep themselves warm or for cooking food. It was reported by Demirbas (2006)[3] that transition from nuclear energy led to oil, from oil to natural gas and from wood biomass led to coal. Frauke and Mitchell (2011)[4] reported that due to the continuous utilization of fossil-based resources, there is increase in the daily amount of carbon dioxide that is produced and emitted into the environment[5]. Over a lengthy period of time, carbon dioxide that is emitted to the earth's atmosphere accumulates and increase on the warmth of the planet and global warming effect is because of the increase in carbon dioxide deposition. Renewable energy technologies that does not emit carbon dioxide and other gases, that is comparable or that perform better than the current fossil-based energy technologies were proposed as a solution to the current global warming. Abdulkareem *et al.* (2012)[5] made it clear that in other to minimized emission level, maximized recyclability, and increased functionality to get rid of hazardous emissions in our environment there is need for the search for an alternative energy resource. Bioenergy is one of this alternative energy resources. It represents the production of sustainable chemicals and fuels by utilizing biomass as starting raw material. Bioethanol and biogas are the common fuels that are obtained from biomass.

The conversion of huge organic wastes of biomass generated by food processing sector and other human endeavours into useful products such as ethanol is now growing interest in both Nigeria and many developing countries.

In the present years, considerable attention has been given to bioethanol produced from renewable biomass. The development of an eco-friendly and economical ethanol production process was the focus of most research in the last decade. Bioethanol produced from forestry and agricultural residues and other forms of lignocellulosic biomass were given more attention[6]. The disposal and burning of agricultural products residues in the past were an accepted practice, however, due to concern over health effects from burning fields, it is now being challenged. This waste and residue contain a reasonable amount of cellulose which is a good source of fermentable sugars. Production of bioethanol is made environmentally friendly and also renewable through bioconversion of cellulosic biomass into fermentable sugar using microorganisms[7].

II.   Materials and Methods

Biomass Sample Pretreatment

In the present study, yam peels (*Dioscorea rotundata*) as residue from agricultural crops were used as biomass sources. The Yam peels samples was sources locally in Kaduna State. In the conversion of lignocellulosic biomass such as sugarcane bagasse into bioethanol; four major unit operations are employed. These include: pre-treatment, hydrolysis, fermentation and product recovery/ distillation. Pre-treatment is the first stage in the production of bioethanol. The peels was washed under running tap to remove sand and other impurities, and was sun-dried for three days before milling into a powder (flour) and it was then sieved to standardize the particle size. The sieved biomass sample was then stored in plastic jars at ambient temperature. Figure 1 shows the processed waste biomass used in this study.

Figure 1. Biomass Sample

Acid Hydrolysis of Yam Peels

The acid hydrolysis was carried out as described by Oyeleke and Manga (2009)[7]. The effect of temperature and time of hydrolysis process was investigated to ascertain the optimal experimental conditions for the production of sugar from yam peels (cellulosic feedstock) for bioethanol production. The effect of temperature on the hydrolysis process was investigated by varying the temperature between 60.00 - 110.00 ⁰C with step increment of 10.00 ⁰C. Hydrolysis time was also varied between 30.00 - 180.00 min with step increment of 30.00 min. A quantity (30.00 gram) each of Yam peels sample was weighed into 6 separate conical flasks, and 50.00 mL of 1.00 M of HCl was added to each conical flask respectively. The flask was covered with cotton wool, wrapped in aluminum foil and was heated in a water bath. The hydrolysed sample was autoclaved for 15.00 min at 121.00 °C. The flask was allowed to cool, then followed by filtration using No 1 Whatman filter paper and the sugar yield measured and recorded.

The optimum hydrolysis temperature and time was then used to hydrolyse 200.00 grams of yam peels in 1000.00 mL of 1.00 M HCl, the sugar of the filtrate was measured and the pH of the filtrate sample was adjusted to pH of 5.00 using 20.00 % NaOH and was subjected to fermentation process.

Determination of Sugar Content of the Filtrate

The Abbe 60 direct reading refractometer was used to determine the sugar content of the filtrate. The sugar was determined using the standard procedure of the American Society for Testing and Materials (ASTM D1218). Few (3 – 4) drops of the sample were transferred into the glass slide of the refractometer. Water at 30.00 °C was circulated round the glass slide to keep its temperature uniform. Through the eyepiece of the refractometer, the dark portion viewed was adjusted to be in line with the intersection of the cross. At no parallax error, the pointer on the scale pointed to the refractive index above and sugar level below. This was repeated and the mean value noted and recorded as the sugar level.

Fermentation of Hydrolysed Samples

The fermentation was carried out as described by Kroumov *et al.* (2006)[8] and Oghgren *et al.* (2006)[9]. After adjusting the pH of the hydrolysed sample, 20.00 grams of *Saccharomyces cerevisiae* (yeast) was added and was shaken for about 5 min to activate the yeast. The sample was then allowed to undergo fermentation for 5 days.

Distillation of Fermented Samples

After fermentation for 5 days, the sample was then removed. The fermented broth was then dispensed into round-bottom flasks fixed to a distillation. A conical flask was fixed to the other end of the distillation column to collect the distillate. A heating mantle with the temperature adjusted to 78.00 °C was used to heat the round-bottomed flask containing the fermented broth[7]. The volume of the distillate was measured using measuring cylinder.

Confirmatory Test for Bioethanol Produced

The sample of bioethanol produced was analyzed using Shimadzu FTIR-8400S Fourier Transform Infrared Spectrophotometer, at NARICT Zaria, Kaduna State, Nigeria, to confirm or otherwise if ethanol was actually produced.

Characterization of Bioethanol Produced

Based on reported method Ademiluyi and Mepba (2013)[10], the bioethanol produced from yam peels was characterized to determine its properties and was compared with standard values of bioethanol.

Density

As described by Ademiluyi and Mepba (2013). The density was measured using pycnometer. Empty pycnometer was weighed. The pycnometer was then filled with the sample (ethanol), the excess was wiped off, the weight was recorded, and the density was calculated using the formula:

$$\text{Density (g/mL)} = \frac{\text{mass}}{volume} \qquad [1]$$

Specific gravity

As described by Ademiluyi and Mepba (2013). The specific gravity was measured using pycnometer. Distilled water was filled into the pycnometer, weighed and recorded. The specific gravity was then calculated using the formula:

$$\text{Specific gravity (spg)} = \frac{\text{Density of Ethanol}}{\text{Density of Water}} \qquad [2]$$

Refractive index

In accordance with the standard procedure of the American Society for Testing and Materials (ASTM D1218), Abbe 60 direct reading refractometer was used to determine the refractive index. Few drops of the sample were transferred into the glass slide of the refractometer. Water at 30.00 °C was circulated round the glass slide to keep its temperature uniform. Through the eyepiece of the refractometer, the dark portion viewed was adjusted to be in line with the intersection of the cross. At no parallax error, the pointer on the scale pointed to the refractive index above and brix level below. This was repeated and the mean value noted and recorded as the refractive index.

Flash point

The American Society for Testing and Materials (ASTMD92) method was used to determine the flash point of the bioethanol. A quantity (10.00 mL) of the sample was introduced into a (50.00 mL) beaker and then a thermometer was inserted. The beaker was placed on a bunsen burner. A flame source was brought at intervals to determine the temperature at which a flash appeared on the surface of the sample while the sample in the beaker was heated.

III.   RESULT AND DISCUSSIONS

Optimization of Hydrolysis Temperature

Yam peels was utilized as a feedstock for the conversion of bioethanol. The hydrolysis time and temperature were first optimized and the bioethanol was produced using the optimum hydrolysis temperature and time.

As reported in literature, increase in hydrolysis temperature positively favoured the sugar yield[11]. In this present study, hydrolysis temperature was varied between 60.00 °C to 110.00 °C, while hydrolysis time was fixed at 20.00 min with acid concentration of 1.00 M. The results obtained as presented in Table 1.0 indicated that, increase in hydrolysis temperature positively favoured the sugar yield with optimum yield of 12.00 % at 110.00 °C. When compared to the work of Egbosiuba et al. (2014)[12] in Table 2.0, the optimum sugar yield of 12.00 % at 110.00 °C obtained in this study contradicted the result which reported a decreased in sugar yield when temperature was increased to 110.00 °C. The variation in the temperatures could be attributed to the variation in the variety of feedstock utilized and other operating parameters.

Table 1.0: Effect of Hydrolysis Temperature on the Yield of Sugar

Temperature (°C)	Sugar Yield (%)
60.00	2.50
70.00	3.00
80.00	4.00
90.00	5.00
100.00	11.00
110.00	12.00

Table 2.0: Comparison between the Effect of Hydrolysis Temperature on the Yield of Sugar on Yam Peels and Cassava Peels

Temperature (°C)	Present Work Sugar Yield (%)	[1]Reported Sugar Yield of Cassava Peels (%)
60.00	2.50	3.50
70.00	3.00	4.00
80.00	4.00	4.50
90.00	5.00	5.00
100.00	11.00	6.00
110.00	12.00	5.50

[1] (Egbosiuba et al., 2014)[12].

Optimization of Hydrolysis Time

Also investigated in this study was the influence of time on hydrolysis. For this purpose, hydrolysis time was varied between 30.00 min to 180.00 min, while hydrolysis temperature was fixed at 50.00 °C with acid concentration of 1.00 M.
Results obtained as depicted in Table 3.0 shows that hydrolysis time of 120.00 min gave the maximum sugar yield of 8.60 %. The sugar yield from hydrolysis of the yam peels increases with increase in hydrolysis time from 30.00 min to 120.00 min. Whereas, further increment of hydrolysis time resulted in decrease sugar yield. Compared to literature, this pattern of results closely agrees to the result reported by Egbosiuba et al. (2014)[12] (Table 4.0), who work on the "Production and Characterization of Bioethanol from Cassava Peel: Alternative Energy Source". The decrease in sugar yield when the hydrolysis time was increased may be due to degradation of sugar.

Table 3.0: Effect of Hydrolysis Time on the Yield of Sugar

Time (min)	Sugar Yield (%)
30.00	2.00
60.00	3.00
90.00	5.50
120.00	8.60
150.00	3.40
180.00	3.40

Table 4.0: Comparison between the Effect of Hydrolysis Time on the Yield of Sugar on Yam Peels and Cassava Peels

Time (min)	Present Work Sugar Yield (%)	Reported Hydrolysis Time (min) Used for Cassava Peels	[1]Reported Sugar Yield of Cassava Peels (%)
30.00	2.00	30.00	6.00
60.00	3.00	35.00	7.00
90.00	5.50	40.00	7.50
120.00	8.60	45.00	7.80
150.00	3.40	50.00	7.30
180.00	3.40		

[1] (Egbosiuba et al., 2014)[12].

Volume of Bioethanol Produced

The optimum hydrolysis temperature and time was used to produce bioethanol. The result as shown in Table 5.0 revealed that two hundred grams (200.00 grams) of the yam peels gave bioethanol yield of 180.00 mL after fermentation for 5 days. The results revealed the possibilities of producing bioethanol from yam peels which may serve as cheap alternative source of fuel and energy generation.

Table 5.0: Volume of Bioethanol Produced

Yam Peels	Hydrolysis Temperature	Hydrolysis Time	Fermentation Time	Volume of Bioethanol
200.00 grams	110.00 ºC	120.00 min	5 days	180 mL

Fourier Transform Infrared (FTIR) Results for Bioethanol

Figure 2.0 shows the FTIR result of the sample. The peak around 3419.90 cm^{-1} represents O–H stretching[13]. The peaks at 2924.18 cm^{-1} and 2853.78 cm^{-1} is ascribed to C–H stretch [14], while that observed around 1640.51 cm^{-1} as is between 1680 cm^{-1} – 1600 cm^{-1} represent C=C[15]. Also, absorption bands at 1300 – 1600 cm^{-1} related to lignin has also being reported earlier by Kumar *et al.* (2009)[16]. The peak at 1440 – 1220 cm^{-1} represent C–O–H bending as it appears as broad and weak[15], while the peak around 1153.47 cm^{-1} as is between 1260 – 1000 cm^{-1} represent C–O stretching which can be used to assigned primary, secondary, or tertiary structure to an alcohol[15].
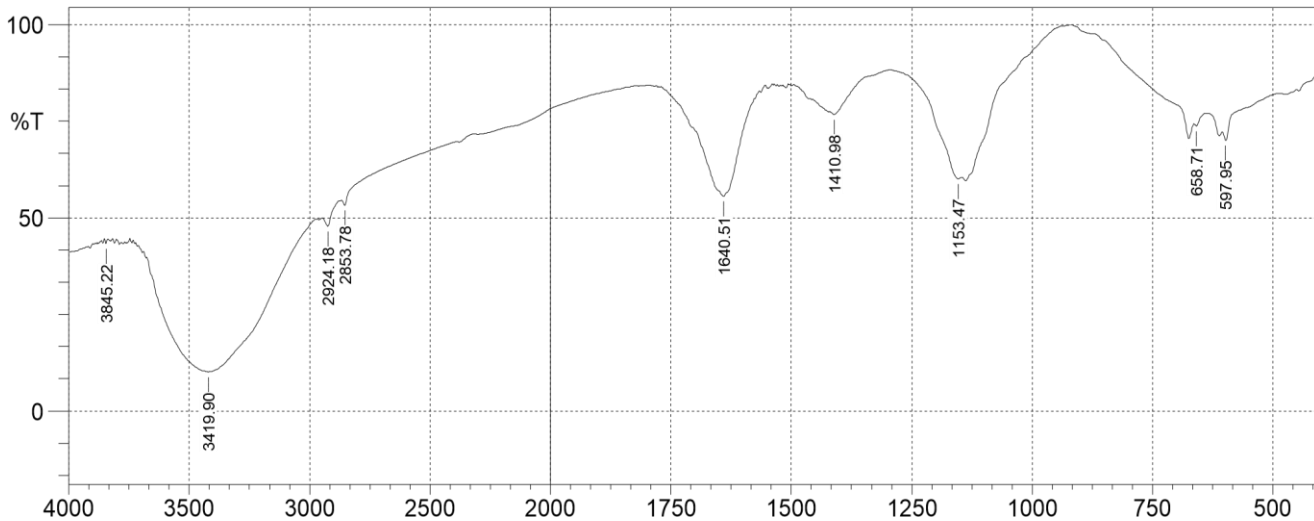

Figure 2.0: FTIR Result of the Bioethanol

Characterization of Bioethanol

The results of the properties of produced bioethanol is presented in Table 6.0. Density is an important parameter for ethanol fuel injection systems. The value of density must be maintained within the tolerable limits to allow optimal air to fuel ratios for complete combustion. High density bioethanol can lead to incomplete combustion and particulate matter emission[17,18]. The density of bioethanol produced was determined to be 0.784 gcm^{-3} which is less than ASTM standard value 0.790 gcm^{-3}. This value shows slight deviation from 0.789 gcm^{-3} reported in the work of Bilyaminu *et al.* (2016)[19] who worked on the characterization of bioethanol produced from sugarcane juice. The slight deviation in density observed in the result can be strongly attributed to differences in feedstock used, fermentation process employed and presence of impurities.

Also tested was the specific gravity of the produced bioethanol. The results as presented in Table 6.0 also indicated that the specific gravity of the produced bioethanol was 0.802 conform to the recommended ASTM standard value. Specific gravity is the ratio of the density of an observed substance to the density of a reference substance (mostly water) at the same conditions[20]. Compared to reported literature, the specific gravity in the present study is higher than that reported by Egbosiuba *et al.* (2014)[12] and Bilyaminu *et al.* (2016) [19], the deviation in the specific gravity might be the distillation process employed. It is a very important property of bioethanol which has relevance in blending with gasoline. This property also impacts positively on the efficient performance of engine.

Refractive index is another fuel property tested for which is used for the identification and the determination of the purity of bioethanol. Refractive index of the produced bioethanol in this study is 1.329 as presented in Table 6.0 which is lighter than the set ASTM value. Compared to the refractive index of 1.362 reported by Egbosiuba *et al.* (2014)[12], the refeactive index in this study is higher. It can be deduced from the results obtained that the bioethanol produced is pure.

Flash point described is a physical parameter that measures the potential of fuels to catch fire and explosion hazards in liquids and is also utilized for the classification and labelling of dangerous liquids[21]. Liquids with flash points which are less than 37.80 ºC are referred to as flammable and combustible liquids (ASTM, 2011). The flashpoint of the bioethanol produced was 17.5 ºC which in close proximity to 16.60 ºC reported in literature[19], but lower than the ASTM minimum value of 18.60 ºC. The result obtained showed that the bioethanol produced do not pose a hazard at low temperature during storage. The higher the flash point the safer the fuel in terms of handling, storage, and transportation[22].

It can be inferred from the various analysis conducted on the bioethanol produced that the properties of the bioethanol compared favourably with some of the properties of bioethanol. The variation in some of the properties can be attributed to the nature of the feedstock (yam peel) used in this study and on the experimental error.

Table 6.0: Comparison between the Properties of the Produced Bioethanol and Cassava Peels and Sugar Cane

S/N	Properties	Units	Present Results	[1]Cassava Peels	[2]Sugar Cane	ASTM Standard
1.	Density	gcm^{-3}	0.784	-	0.789	0.790
2.	Specific Gravity	-	0.802	0.785	0.789	0.750-0.850
3.	Refractive Index	-	1.329	1.362	-	1.360-1.364
4.	Flash Point	ºC	17.500	14.2	17	10.000-18.500

[1](Egbosiuba *et al.,* 2014); [2](Bilyaminu *et al.,* 2016) [19].

IV.   CONCLUSION

The production of bioethanol from yam peels was reported in this study through an optimized process of hydrolysis. Also, it can be concluded based on the results obtained that hydrolysis temperature and hydrolysis time affects the optimal yield of glucose during hydrolysis of yam peel. The results revealed that hydrolysis temperature of 110 ºC and hydrolysis time 180 minutes gave the optimal yield of glucose and two hundred grams of the sample gave bioethanol yield of 180 mL after fermentation for 5 days. The properties of the bioethanol produced from the yam peels also conform to the set limit by ASTM. Despite the ability to use yam peels for bioethanol production, the yield can be influenced by several factors especially temperature, time and fermentation time. If the bioethanol is produced under the stipulated experiment conditions with *Saccaharomyces cerevisiae* (baker's yeast), a substantial amount of bioethanol, which is used as a chemical feedstock, will be produced. Thus, the importation of ethanol can be reduced if substantial energy is devoted to the production of bioethanol from biomass.

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