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Comparative Study on Bioethanol Production from Agro-Waste Feedstock via Acid Hydrolysis Method using Two Different Mineral Acids as Catalyst

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ABSTRACT

The huge demand for energy derived from non-renewable fossil feedstock is not only unsustainable but also have dire environmental consequences. Bioethanol produced from biomass is an environmental benign alternative to fossil fuels. This study evaluated the production of bioethanol from the acid catalysed hydrolysis of cassava and yam peels at different concentrations of HCl and H_2SO_4 respectively. *S. cerevisiae* (Baker's yeast) was employed for the fermentation of the hydrolysates to produce the bioethanol. Bioethanol produced from the hydrolysates of cassava and yam peels increased with increasing concentrations of the acids (HCl, H_2SO_4) respectively, with 3M acid concentrations giving the highest bioethanol yields. At 3M HCl, yields of 20.56±0.9 % and 24.11±0.44% were obtained for the hydrolysates of cassava and yams peels respectively while, 3M H_2SO_4 afforded 25.5±0.71 and 33.00±0.42% maximum bioethanol yield for cassava and yam peels respectively. Overall, the diprotic H_2SO_4 acid performed better than the monoprotic HCl in the bioethanol production resulting in higher yields under the conditions employed in this study. Generally, yam peels feedstock produced more bioethanol than cassava peels. Regardless of the acid employed for the hydrolysis, the boiling points of the bioethanol produced for cassava peels (81±1.00 - 83±2.00 ^oC) was found to be closer to that of pure ethanol (78 ^oC) than those obtained from yam peels (81±2.00 - 86±1.00^oC).

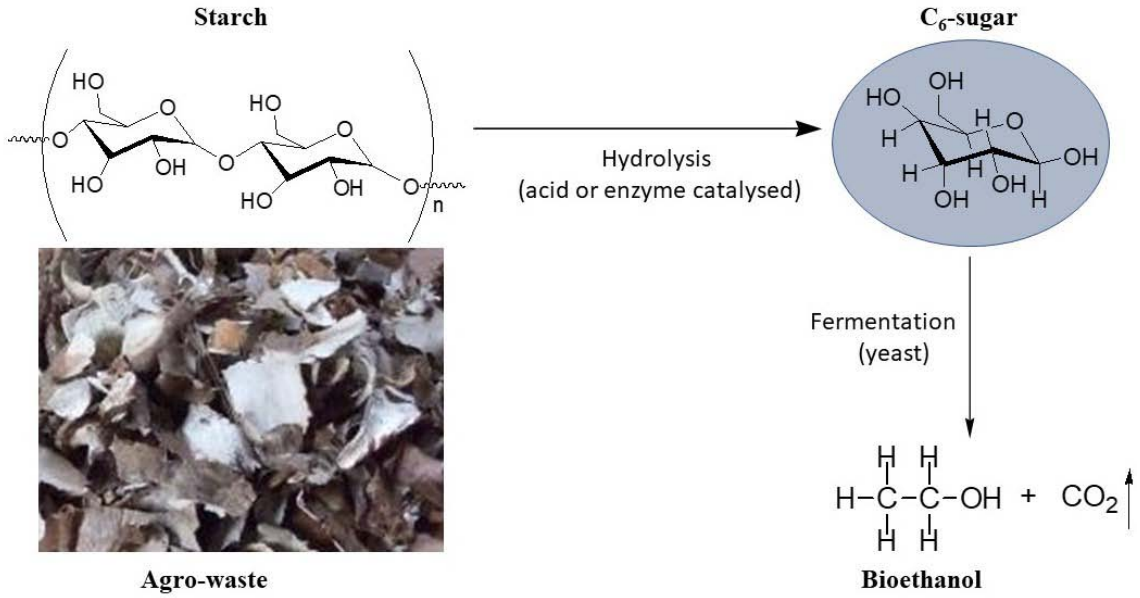
Keywords: Acid hydrolysis, Bioethanol, Cassava and Yam peels

INTRODUCTION

There is a continuous increase in the demand for fossil fuel to meet energy demand with increasing world population. The huge dependence on crude oil resources globally is by no means a sustainable way of meeting energy and chemical demands, because of its devastating effect on the environment and declining reserves (Galadima *et al*., 2011; Romera-castillo e*t al.*, 2018; Adeola *et al*., 2022). Of great concern is the emission of "greenhouse gases' (GHG) especially CO_2 into the environment from the exploration and consumption of crude oil products, leading to global warming (Isa, 2014;Hussain *et al*., 2022; Raza *et al*., 2022). There is a renewed call to action by the academic and industrial communities to provide sustainable alternatives that is void of fossil fuels to meet our energy, material, and chemical need to mitigate these concerns. In addition, the recent crisis in Eastern Europe has led to a worldwide disruption in the supply chain of crude oil resources. In the wake of this, there is now an intensified search for renewable and sustainable alternatives as crude oil price continuesto fluctuate. While there are technological advancements in the use of environmentally benign resources such as fuel cells, solar and wind for power generation (Omoruyi *et al*., 2016), the production of certain chemicals and liquid fuels from renewable resources is still evolving due to its limited alternatives. Biomass is still the most potent, renewable, and sustainable route of accessing some biochemicals/liquid fuels usually obtained from crude oil resources (Kumar *et al*., 2008; Saxena *et al*., 2009; Ahorsu *et al*., 2018).

Bioethanol is a very important renewable fuel obtained from biomass feedstock and it is biodegradable. Bioethanol is a component of the commercially available non-petroleum based P-series liquid fuels which is a blend of biomass derived ethanol, methyltetrahydrofuran and C_5 hydrocarbons (Demirbas, 2003; Omoruyi *et al*., 2016). It equally has the potential of replacing gasoline (petrol) in automobile engines owing to its high octane number emission (Balat *et al*., 2008; Nwufo *et al*., 2013; Patni *et al*., 2013). The high-octane rating of bioethanol also makes it a suitable blend with petrol thus improving the octane number of the blended fuel and eliminating the use of poisonous lead (Pb) previously employed for this purpose (Öhgren *et al*., 2006; Adetunji *et al*., 2015). Furthermore, the complete combustion and zero net CO_2 emission (Bušić *et al*., 2018; Oyegoke *et al*., 2022) associated with the use of bioethanol as biofuel due to the presence of oxygen in the molecule makes it a clean and sustainable form of

energy. Bioethanol is obtained from the fermentation of C_6-sugars derived from suitable biomass feedstock as shown in Scheme 1.



Scheme 1: Production of bioethanol from suitable biomass feedstock (agro-waste)

Energy crops such as cassava, sorghum, soya beans, sugar cane, wheat, guinea corn are some of the major biomass feedstock for bioethanol production (Kim and Dale, 2004; Adetunji *et al*., 2015). In Nigeria, the Automobile Biofuel Program (ABP), established by the Nigerian National Petroleum Commission (NNPC) in partnership with the Energy Commission of Nigeria (ECN) identifies sweet potato, maize, sugar cane, sorghum and cassava as the major feedstock for the production of bioethanol (Peter *et al*., 2009; Beniwo *et al*., 2016; Oyegoke *et al*., 2022). However, these are food crops and the continuous use of them in the production of biofuels is a danger to food security and the food industries. Alternatively, the use of agricultural solid waste feedstock is an attractive and sustainable route to produce biofuel with Nigeria being an agricultural nation, these raw materials are in relative abundance (Shaaban and Petinrin, 2014). Agricultural waste (agro-waste) such as yam peels, cassava peels rice husk, cocoyam peels etc have been employed as biomass feedstock for the production of biofuels such as bioethanol, biodiesel, and biogas (Binod *et al*., 2010; Akponah and Akpomie, 2011; Adetunji *et al*., 2015; Olayemi *et al*., 2019; Oyegoke *et al*., 2022). These feedstock have huge energy potential because they contain the much needed starch component which can undergo either an enzymatic hydrolysis or acid catalysed hydrolysis to produce glucose (C_6-sugar), which is thereafter converted to bioethanol through the fermentation process. Adetunji *et al.* (2015) successfully produced bioethanol from cassava peels obtained in Abeokuta (Ogun State, Nigeria) via enzymatic hydrolysis. Recently, bioethanol was produced from different agro-waste (yam, potato, watermelon and pineapple peels) via acid hydrolysis using different concentrations of monoprotic (HCl) acid (Ezejiofor *et al.*, 2018)While there are comparative studies on the type/nature of the biomass feedstock employed on the production of bioethanol via acid hydrolysis(Ezejiofor *et al.*, 2018), to the best of our knowledge, there is no comparative study on the effect of acids on the production of bioethanol. To this end, this research work evaluates the production of bioethanol from locally sourced agro-waste; cassava (*Manihot esculenta*) and yam (*Dioscorea cayenensis*) via acid hydrolysis using a monoprotic (HCl) and a diprotic (H_2SO_4) mineral acid respectively. The performance of both acid at different concentrations on the amount of bioethanol produced from the respective feedstock was evaluated.

MATERIALS AND METHODS

Collection/processing of samples

Cassava and yam peels (agro-waste) were collected from different household and market wastes in Benin city, Edo State, Nigeria. The samples were sorted and washed with water to remove sand and other dirt and then sun-dried for two weeks. After drying, they were milled using a standard milling machine and then sieved through a 425 µm sieve to obtain uniform particle size. Thereafter, they were stored in a clean and labeled containers for analyses. Acid hydrolysis of samples was performed following standard procedure adopted by Ezejiofor *et al*. (2018) with slight modifications.

Acid Hydrolysis

To a set of four different flasks assigned C_1, C_2,C_3 and C_4 respectively, 20 g of cassava sample was introduced to each flask. To another set of four flasks assigned Y_1, Y_2, Y_3, Y_4, 20 g of yam sample was introduced into each flask. Distilled water (50 mL) was added to the eight flasks and placed in the oven at 50 ^oC for 20 minutes as a pretreatment. Thereafter, a 100 mL solution of 0.5M HCl was added to each flask labeled C_1, C_2, Y_1, Y_2, while 100 mL of 0.5M H_2SO_4 was introduced into C_3, C_4, Y_3, Y_4 respectively. The samples were hydrolysed at 100 ^oC and the progress of hydrolysis was closely monitored with iodine solution until the starch content in the samples was completely hydrolysed. Following the complete hydrolysis, the pH of the samples was adjusted to 5 by the introduction of equal concentration of sodium hydroxide (0.5M) solutions to the respective samples. Thereafter, the samples were filtered through a No 1. Whatman filter paper and the presence of reducing sugar in the respective filtrate was established using Benedict solution, while the amount produced estimated using a refractometer. The experiment was repeated at different concentrations of 1.0M, 1.5M, 2.0M, 2.5M and 3.0M HCl and H_2SO_4 respectively.

Fermentation of reducing sugar to bioethanol

A solution containing 20 g of activated *S. cerevisiae* (Baker's yeast) was aseptically inoculated into each flask containing the agro-waste hydrolysates. Fermentation proceeded by mixing the resulting solutions with a glass rod at room temperature. Thereafter, each flask was covered with an aluminum foil to exclude air and then kept for seven days with constant agitation whilst maintaining the pH of systems to remain within the 4-5 which is the pH range of fermentation. The bioethanol produced were recovered from the reaction mixtures via simple distillation between 78 ^oC – 80 ^oC(Oyeleke and Jibrin, 2009; Iticha, 2016; Bakare *et al*., 2019).The quantity of bioethanol obtained from each distillate was measured using a measuring cylinder and values recorded. The boiling point, colour, and odour of the products were assessed to ascertain quality of the bioethanol produced. The specific gravity (SG) for each bioethanol obtained was evaluated from equation 1. Following reported methods, the alcohol percent by volume (%v/v) was estimated from its specific gravity using the equation 2 (Girish *et al*., 2014; Olayemi *et al*., 2019).

$$Specific\ gravity\ (SG) = \frac{weight\ of\ total\ volume\ ethanol\ distillate}{weight\ of\ equal\ volume\ of\ distilled\ water} \quad (1)$$

$$Percentage\ bioethanol\ (\%\ v/v) = [8610.6 - (16584 \times SG) + (7973.3 \times SG^2)] \quad (2)$$

RESULTS AND DISCUSSION

Bioethanol obtained from the agro-waste feedstock (cassava and yam peels) at different concentrations of HCl and H_2SO_4 respectively were colourless liquids and had the characteristic smell of alcohols. Tables 1 and 2 show the results obtained for bioethanol production from cassava and yam peels respectively using different concentrations of hydrochloric acid while Tables 4 and 5, displays the results obtained for H_2SO_4 from the respective agro-waste. It was observed that the boiling point for the bioethanol obtained from cassava agro-wastes were considerably lower than those obtained from yam peels regardless of the acid catalysts employed for the hydrolysis. The boiling point of bioethanol obtained from HCl hydrolysis of cassava peels were between 82±1.00 to 83±2.00 ^oC while that obtained for yam peels employing the same acid ranged from 81±2.00 to 86±1.00 ^oC. For H_2SO_4catalysed hydrolysis, the boiling point ranged between 81±1.00 to 82±1.00 ^oC and 80±1.00 to 85±1.00 ^oC for cassava and yam peels respectively. Although these values are within the range of those reported by Ezejiofor *et al*. (2018), it is important to state that the close boiling point range for bioethanol obtained from cassava peels relative to that of pure ethanol (78 ^oC) is indicative of the high purity of the product isolated. On the other hand, the elevated boing points for bioethanol obtained from yams peels suggests the presence of more particles in the distilled products relative to those obtained from cassava peels.

Table 1: Bioethanol produced from cassava peels via HCl acid hydrolysis

HCl Conc. (mol/L)	Vol. of ethanol distilled (mL)	Specific gravity	%Ethanol yield (%v/v)	Boiling pt. (^{o} C)
0.50	7.08±0.30	0.9970	2.10±0.21	82±1.00
1.00	10.59±0.76	0.9980	5.60±0.40	83±2.00
1.50	24.71±0.60	0.9940	9.70±0.63	82±2.00
2.00	31.61±1.60	0.9920	13.40±0.76	83±1.00
2.50	39.33±1.03	0.9570	17.30±0.83	82±1.00
3.00	45.79±1.53	0.9580	20.56±0.90	83±1.00

Table 2: Bioethanol produced from yam peels via HCl acid hydrolysis

HCl Conc. (mol/L)	Vol. of ethanol distilled (mL)	Specific gravity	%Ethanol yield (%v/v)	Boiling pt. (^{o} C)
0.50	9.00±1.00	0.9949	3.01±0.27	86±1.00
1.00	13.00±1.00	0.9914	6.01±0.35	84±2.00
1.50	30.50±0.50	0.9885	8.29±0.57	84±1.00
2.00	44.50±0.50	0.9838	12.51±1.17	83±2.00
2.50	45.00±1.00	0.9776	18.24±0.65	82±1.00
3.00	54.50±0.50	0.9719	24.11±0.44	81±2.00

Table 3: Bioethanol produced from cassava peels via H_2SO_4 acid hydrolysis

H_2SO_4 Conc. (mol/L)	Vol. of ethanol distilled (mL)	Specific gravity	% Ethanol yield (%v/v)	Boiling pt. (^{o} C)
0.50	9.08±0.70	0.9970	3.01±0.21	81±1.00
1.00	13.75±0.80	0.9950	6.85±0.37	81±1.00
1.50	22.20±1.40	0.9930	9.60±0.45	82.±1.00
2.00	33.90±1.80	0.9940	12.80±0.50	81±1.00
2.50	43.30±1.30	0.9770	18.40±0.62	82±1.00
3.00	54.70±2.00	0.9680	25.50±0.71	81±1.00

Table 4: Bioethanol produced from yam peels via H_2SO_4 acid hydrolysis

H_2SO_4 Conc. (mol/L)	Vol. of ethanol distilled (mL)	Specific gravity	%Ethanol yield (%v/v)	Boiling pt. (^{o} C)
0.50	11.50 ± 0.50	0.9922	5.40±0.27	85±1.00
1.00	18.50 ± 0.50	0.9875	9.27±0.66	84±1.00
1.50	42.50 ± 0.50	0.9835	12.59±0.72	83±2.00
2.00	55.00 ± 1.00	0.9781	17.74±0.45	82±1.00
2.50	60.50 ± 2.50	0.9749	20.38±035	81±2.00
3.00	67.50 ± 0.50	0.9672	33.00±0.42	80±1.00

The acid catalysed hydrolysis of 20 g of agro-wastes (cassava and yam peels) at different concentrations of a monoprotic (HCl) and diprotic (H_2SO_4) acids respectively resulted in different yields of bioethanol after fermentation of the hydrolysates with Baker's yeast. There was a steady increase in bioethanol obtained from the respective agro-waste feedstock with an increase in the concentrations of the acid employed. When HCl was employed, the highest percent by volume (%v/v) yield of 20.56±0.90 and 24.11±0.44 were achieved at 3.0 M concentration of the acid for cassava and yam peels respectively while 0.5MHCl gave the least amount of ethanol with values of 2.10±0.21 and 3.01±0.27% recorded for cassava and yam peels respectively. A similar trend was observed when H_2SO_4 was employed as the catalyst for the hydrolysis of the agro-wastes. 3.0M H_2SO_4 resulted in the highest ethanol yields of 25.50±0.71% for cassava peels feedstock and 33.00±0.42% for yam peels, while 0.5M afforded yields of 3.01±0.21 and 5.40±0.27 respectively for cassava and yam peels. These results are in accordance with those reported by Ezejiefor *et al.* (2018) who observed an increase in ethanol yield with increase in the concentration of the HCl acid employed.

Comparing the performance of HCl and H_2SO_4 in the production of bioethanol from cassava and yam peels, the diprotic H_2SO_4 acid hydrolysis of cassava peels resulted in better bioethanol yields of 25.50±0.71 and 3.01±0.21% at the highest (3M) and lowest (0.5M) concentrations of acid employed in this study (Figure 1). However, when 2.0 M concentration of acid was employed, the monoprotic HCl acid gave a slightly higher bioethanol yield of 13.40±0.76% relative to a yield of 12.80±0.50% obtained from H_2SO_4 acid hydrolysis (Figure 1). For yam peels, it was observed that H_2SO_4 gave better yields of bioethanol irrespective of the concentration of the acid (Figure 2). Overall, the diprotic H_2SO_4 acid catalysed hydrolysis performed better, giving more bioethanol yield upon fermentation with Baker's yeast. While both acids are very powerful mineral acid catalysts, H_2SO_4 is a diprotic acid hence, the number of protons ($2H^+$) produced which is needed to catalyse the hydrolysis of the agro-wastes is twice that produced from HCl (H^+). This may account for the general increase in the yields of bioethanol obtained from H_2SO_4 acid hydrolysis of the agro-wastes.

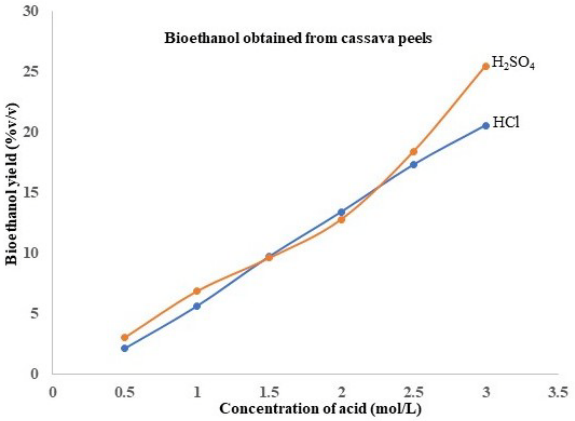


Figure 1: Chart showing the performance of HCl and H_2SO_4 in the production of bioethanol from cassava peels

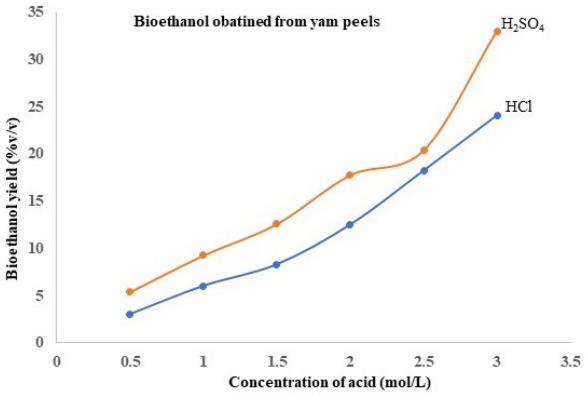


Figure 2: Chart showing the performance of HCl H_2SO_4 in the production of bioethanol yam peels

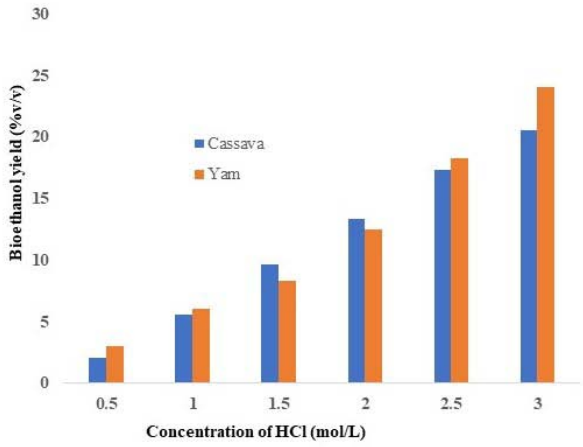


Figure 3: Bioethanol produced from HCl acid hydrolysis of cassava and yam peels respectively

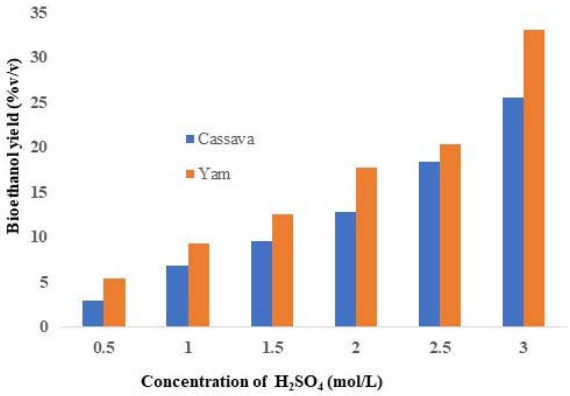


Figure 4: Bioethanol produced from H_2SO_4acid hydrolysis of cassava and yam peels respectively

A comparison on the amount of bioethanol produced from the respective agro-waste revealed that yam peels afforded higher bioethanol yields at the various concentrations of the acid except for 1.5M and 3.0M HCl where cassava peels gave higher yields (Figure 3 and 4). This is contrary to those reported by Oyeleke *et al.* (2012) who obtained a higher yield of bioethanol produced from enzymatic hydrolysis of cassava peels relative to potato peels. Furthermore, cassava is known to be very high in starch content which can be easily hydrolysed to the C_6-suagrs (Ruiz *et al.*, 2011; Chisenga *et al.*, 2019). For this reason, Nigerian National Petroleum Commission (NNPC) identified it as one the major feedstock for the production of bioethanol (Peter *et al.*, 2009; Ben-iwo *et al.*, 2016; Oyegoke *et al.*, 2022).Thus, it was expected that the bioethanol produced from cassava peels employed in this research ought to be higher due to its well-established high starch content. However, our findings revealed that yam peels performed better than cassava in the production of bioethanol under the conditions employed. Although higher bioethanol yields were observed for yam peels, the elevated boiling point of the products compared to those obtained from cassava peels signify a less pure product and indicates the presence of more impurities in the products. This may account for the higher yields observed.

CONCLUSION

The effect of a monoprotic acid (HCl) and a diprotic acid (H_2SO_4) on the hydrolysis of 20 g cassava peels and yam peels agro-waste respectively in the production of bioethanol was investigated. Employing different concentrations of the HCl and H_2SO_4 respectively for the hydrolysis of the agro-wastes afforded various

yields of bioethanol after fermentation with *S. cerevisiae* (Baker's yeast). The amount of bioethanol produced from the agro-waste employed increased as the concentration of the respective acids increased. Maximum bioethanol yields were obtained from the distillates of both agro waste at 3M concentrations of HCl and H_2SO_4 respectively. Overall, the H_2SO_4catalysed hydrolysis of the agro-wastes performed better than HCl under the condition employed as the distillates obtained from its hydrolysates gave better yields of bioethanol upon fermentation. Generally, yam peels produced more bioethanol than cassava peels. However, the boiling points for the bioethanol products obtained from cassava peels confirmed a higher purity of the products when compared to those obtained from yam and were on par with that of pure ethanol.

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