**Production Of Bio-Ethanol From Sugarcane: A Pilot Scale Study** 

In Nigeria

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## ABSTRACTS

The search for alternative and renewable source of fuels especially for transportation has taking a centre stage across the globe due to its ecofriendliness and environmental sustainability. The use of bio-ethanol in internal combustion engines is an alternative to fossil fuels which are erstwhile non renewable and contributes to global warming due to the emission of green house gases (GHGs). This study presents a pilot scale of bioproduction from ethanol bio-resources (sugarcane) through the process of fermentation. The designed pilot plant consists of sugar cane crusher, fermenter, strainer, distillation column, adsorption column (with Zeolite A3 as an adsorbent), heat exchangers and steam generator. The remaining accessories such as pump and compressors were selected. All equipment were fabricated locally in Nigeria, installed and successfully commissioned and test run. Fuel grade ethanol was successfully produced. A qualitative analysis of both fermenter product and the distillate using an infra-red spectroscope revealed the presence of ethanol and a quantitative analysis by refractor-metric method indicated the ethanol percentage in both samples to be 4.3% and 67% respectively. However, the product from the adsorption column was not qualitatively analyzed but its solubility in gasoline at different volumetric concentrations (E10, E20, E30, etc) was observed to be 100% soluble. The blend in gasoline and the product were used to start a new gasoline engine (generator) which started well without any difficulty. The pilot plant has proven the availability of local technology for bio-ethanol production in Nigeria.

Keywords: pilot plant, design and construction, fuel grade ethanol, production, sugarcane.

# **1.0 INTRODUCTION**

# 1.1 Ethanol as Fuel

The search for alternative fuels especially for transport has been refocused on the use of

ethanol as internal combustion engines. The product of fuel ethanol from bio-resources by fermentation process is taking centre stage across the globe.

Fermentation is a bio-chemical process wherein commercial products are obtained through the activity of micro-organism. The chemical changes are brought about in an organic substrate through the action of enzymes by micro-organism. Some of the products of this process include: ethanol, lattice, wine, vinegar, antibiotics, vitamins, acetone, butyl alcohol, etc [1-2]. The modern definition of industrial fermentation is any microbial process controlled by humans that produces useful products [2-4]. Fermentation is directly caused by the life processes of minute organisms. Micro-organisms, which include; bacteria, yeast and moulds, feeds on the organic materials, if they are supplied with the necessary energy, along with other needed nutrients, this process is of utmost interest to the manufacturer. These micro vegetative organisms will not only grow and multiply, but will change the substrate into other chemical substances. Fermentation has been used since ancient time, people who lived along the River Nile in North Africa brewed beer around 300BC [2,5]. Fermentation generally produces a water-ethanol mixture which needs to be separated. Fractional distillation is used to separate mixtures of liquids with different boiling points, such as alcohol and water [6-8]. Pure ethyl alcohol boils at approximately 78.33° C, while water boils at 100° C. A mixture of the two liquids will boil at all temperatures between 78.33° and 100°, depending on the ratio of alcohol to water [9Sloley, 2001]. The previous relationships of alcohol-water mixtures hold true up to alcohol concentrations of about 95.6 percent (w/w) [1,8-9]. At this concentration, the two substances quit boiling separately (i.e., the alcohol in the vapour phase is no longer more concentrated than in the liquid phase), and fractional distillation no longer works. A mixture of this composition is called an "Azeotropic mixture". Process flow diagram for the pilot plant is shown in Figure 1.



Figure 1: Process flow diagram of the pilot plant

#### 1.2 Process description

Production of ethanol from sugarcane involves the following four (4) major stages:

- 1. Crushing the sugarcane to extract the juice.
- 2. Fermentation of the sucrose-rich juice obtained.
- 3. Distillation to separate the ethanol-water to a composition close to the azeotropic composition.
- 4. Adsorption to produce fuel grade ethanol by removal of remaining water.

Ethanol can be blended with gasoline at varying proportions to reduce the consumption of gasoline, and to reduce air pollution [10-11]. It is increasingly used as an oxygenate additive for standard gasoline, as a replacement for toxic octane enhancers such as benzene, toluene, xylene and methyl t-butyl ether (MBTE), these chemicals (MBTE) are responsible

for considerable ground water and soil contamination [12-14].

Table 1: Characteristics of Ethanol and Gasoline.					
Characteristic	Ethanol	Gasoline			
Flash point	12.8°C	-6.7oC			
Ignition	365°C	280°C			
Temperature					
Vapor	3.3-19	1.4-76			
Flammability					
limit (vol%)	0.78	0.8			
Specific gravity					
(water equals 1)	1.6	3-4			
Vapor density	77.8	30-180°C			
(vapor equals 1)					
Boiling point					
Source: (John, 2002)					

bouree. (Joini, 2002)

The Nigerian National Petroleum in Corporation (NNPC) has joined the developed countries by its desire to produce fuel grade ethanol from sugar cane and cassava that will be blended Table2:The physical properties of ethanol

with petrol in proportions not exceeding 10% ethanol by volume. The resulting blended fuel will be called 'Green fuel'. Table 2 shows the physical properties of ethanol. A lot of bench scale research has been done all over the world on bio-ethanol and their resultant data are readily available and accessible. This research focuses on the operation of a pilot plant for bio-ethanol production in Nigeria. This has become necessary for the following reasons:

1. Cost effectiveness and renewability of the source materials compared to fossil fuels

2. Bio-ethanol burns evenly and is environmentally friendly when compared with gasoline.

3. Design and implementation of bio-ethanol pilot plant will lead to the development of small scale industries, thus employment opportunities would be increased.

Property	Ethyl alcohol
Molecular formula	C <sub>2</sub> H <sub>6</sub> O
Boiling point	78.5°C
Melting point	-117°C
Density	0.789g/ml
Solubility in water	Completely soluble in all proportion
Action on metal sodium	Vigorous reaction, hydrogen evolved
Structural formula	нн
	H - C - C - O - H
	нн
Condensed structural formula	CH <sub>3</sub> CH <sub>2</sub> OH

Source: (Clausen & Glady, 1989)

Nigeria has since signed a memorandum of understanding with two companies in Brazil-PETROBRAS and COIMEX to leverage their experience in Ethanol production and marketing with machineries put in place to commence importation of fuel Ethanol since first quarter of 2006. Nigeria also has capacity to locally develop its bio-fuels industry, even though the Nigerian government has approved the importation of ethanol to test the market in the production of "green petrol" (E10) by the NNPC, there is the need for backward integration to produce all the bio-ethanol needs of Nigeria locally using our vast agricultural resources.

#### 2.0 Methodology

After successful commissioning of the pilot plant, a careful test run was carried out using water and later with compressed air to check any possible leakages along the pipe lines which were

subsequently addressed. Sugarcane was obtained from the market and charged into the crusher to squeeze out the juice. The juice was pumped into the

fermenter and allowed to ferment applying suitable yeast (*Saccharamysis cerevesae*). This was later charged into the fermenter product tank. The product from the tank was later charged into the distillation column through the pre-heater for separation into overhead distillate and bottom product. The overhead product was heated and passed in vapor state through the adsorption column which contained zeolite 3A (adsorbent), the product later condensed and collected in a tank as fuel grade ethanol. The collected product was then mixed with gasoline in the ratio E10, E20, E50, E60, E80 and E100. All proportions of the blend were tested in a new gasoline powered generator. Samples from the four Table 3: Values of operational temperatures points namely; sugarcane juice, fermenter product, overhead distillate and bottom product were qualitatively analyzed using infra-red spectroscopy.

# 3.0 Results and Discussion

After the pilot plant was successfully commissioned, some operational data were collected. These included the various temperatures at the fermenter, distillation column and heat exchangers. The results are shown in Table 3.

Unit	Inlet temperature	Exit temperature
Fermenter cooler	initial 22°C	Initial-22°C
	after 24hrs-28°C	after 24hrs-24°C
	after 48hrs- 32°C	after 48hrs- 24°C
Feed pre-heater	22°C	75°C
Re-boiler	88°C	90°C
Condenser	64°C	40°C
Volume of distillate collecte	ed =5.0 litres	

#### Qualitative analysis

Samples were collected at both raw material/feed end as well as the distillate and bottom products and these were analysed. Fourier Transform Infra-Red Spectroscope [12] was used to ascertain the quality

Table 4: FTIR spectra of sugarcane juice

of these products. Tables 4-8 show the results obtained for sugar cane juice, fermenter product, distillate, and bottom products. The spectra are also shown in Figures 2-6.

rable 4. I The spectra of su	garcane juice	
Wave number (cm <sup>-1</sup> )	IR finger print	Remarks
3401.55	Water	Presence of -OH functional group and the IR
		match it with the –OH from water.
2360.24	Butyramide	Presence of C-C or C-N triple bond and the IR
		match it with butyramide.
2340.32	Lanthanum sulphate	Presence of S-H in the sample and the IR match it
	Nona hydrate	with lanthanum sulphate Nona hydrate.
2070.06	Heptadien-4-ol	Presence of a secondary alcohol and C-C double
		bond which the IR matched with hepta-di-ene-4-
		ol.
1636.12	(+)-beta-D-lactose	Presence of sucrose which the IR matches with
		(+)-beta-D-lactose.

#### Table 5: FTIR Spectra of Distillate

Wave number	IR finger Print	Remarks
3367.24	Ethanol	Presence of –OH from alcohols or carboxylic acid and was matched by the IR as ethanol.
2976.60	D-(-)-fructose	C-H stretching and the IR matched it with D-(-)-fructose.
2538.36	Water	Presence of O-H bond and was matched by the IR as water.
2360.78	1,3-Dichlorobutane	Presence of halogenated compound and the IR matches it with 1,3- Dichlorobutane.
2129.87	(+)-Beta-D-lactose	Presence of C-N triple bond but was matched by IR as (+)-Beta-D-



Figure 2: Infra-red spectrum of sugar cane juice

Figure 3: Infra-Red Spectrum of fermented Juice



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eak finding	result table	):							
Peak#	1	2	3	4	5	6	7	8	9
Position	3367.24	2976.60	2538.36	2360.78	2129.87	1921.45	1647.45	1384.14	1326.85
Height	0.133	0.373	54.875	43.390	40.867	49.719	1.328	3.714	8.172
Peak#	10	11							
Position	1047.31	879.76				7,			
Height	0.252	0.771							

Peak finding results for: distillate Frequency: 400.00 - 4000.00, threshold: 60.559, sensitivity: 50.00 Peak finding result table:





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#	Index	Match	Compound Name	Library Name
1	15	65.61	Water	Example Library
2	19	32.64	BUTYRAMIDE	Aldrich Condensed Phase Sample Library
3	59	20.14	BENZAMIDE, 99%	Aldrich Condensed Phase Sample Library
4	97	18,16	NYLON 6/10	Aldrich Condensed Phase Sample Library
5	1	12.84	Acetaldehyde	Example Library
	50-	Acetaidei Match: 17	Pite w	man
	50-	MYLON 6 Match: 1	8:16	Manna
	50	BENZAM Match: 2	0.14 WW	Marina
	50	BUTYRA Match: 3	2.64	Man
	80 60	Materic	961	
	50.0	Dotton		235697386 1041.665
			3500 3000	2500 2000 1500 1000 500 Wavenumbers (cm-1)

Figure 5: Infra-red mach (finger print) of bottom product

# Quantitative analysis

A refractor metric method for the approximate measurement of the alcoholic strength at room temperature [15] was conducted on the product samples and the results obtained are shown in Table 8.

Table 8: Densities and Refractive Indices

Product	Density (kg/m <sup>3</sup> )	Zeiss number	Refractive index (n <sub>d</sub> )
Fermented juice	1003	5	1.3293
Distillate	879	18	1.33435
Bottom product	1012	3.8	1.32883
Sugar cane juice	1077	-	18.2

The formula for calculating the refractive index for ethanol strength is given by;

 $n_D = 1.327338 + (3.93470 * E-04 * zeiss) - (20.4467 * E-08 * zeiss<sup>2</sup>)$ 

From the value of refractive index  $(n_D)$  and density of the sample, D and R were obtained from Tables A and B respectively. Hence ethanol strength  $(P_{EtOH})$  is given by the summation of these values, i.e. R and D.

Thus for fermented juice, R = 6.2 and D = -1.92 Therefore,  $P_{EtOH} = 6.2 + (-1.92)$ 

= 4.28%

For the Distillate, R = 6.2 from Table B while D was obtained by extrapolation from Table A as 60.8. Therefore,  $P_{EtOH} = 6.2 + 60.8$ 

= 67.0%

For the Bottom product, R = 6.2 and D = -7.68Therefore,  $P_{EtOH} = 6.2 + (-7.68)$ 

= -1.48% which shows complete absence of ethanol.

# **3.2 Discussion of results**

A pilot plant with capacity to produce about one hundred litre/day of bio-ethanol pilot plant was successfully test-run. All units, except adsorption column, were operated and bio-ethanol was produced. Experimental analyses of the feed, distillate, and bottoms of the distillation column using infra-red spectroscope revealed the presence of ethanol in the fermented juice and the distillate but none in the bottom product. However, a fuel grade ethanol has 99.9% (weight basis) purity as was designed in this work. Further analyses using refractor-metric method revealed that the distillate contains 67% ethanol (wt %), the feed contains 4.28% ethanol (wt %), and the bottom contains no ethanol. The ethanol content of 67% in the distillate was below the design value (about 95%). This was due to inadequate packing material in the distillation column. The packing height above the feed plate was only 0.4m as against the designed height of 1.1m. This brought about the ineffective separation. Lack of appropriate parking materials (Raschig rings), was responsible for this. Improvisation with broken bottles had to be done to set the distillation column on stream. Infact to achieve 67% in the distillate in spite of the inadequate packing shows the distillation column was effective. The ethanol strength of the column feed was contrary to the theoretical and designed value of 7-10%. The difference can be attributed to the volatility of

ethanol. The fermented juice stayed for about a month before distillation was conducted and hence the ethanol volatilized and its smell was perceived all around the premises and this reduced the quantity of the product. This could also have contributed to the low value (67%) of ethanol in the distillate. The infra-red match of the bottom product clearly showed that it contains no ethanol at all. This may be as a result of conversion to other products as the analysis conducted was not immediate. Theoretically and by the design, it is supposed to contain at least 0.02% of ethanol as confirmed by the infra-red match of the bottom product (figure 5). Zeolite 3A, which was imported from China and applied as an adsorbent in the adsorption column yielded and fuel grade ethanol was produced. The product was used to test a new generator and their mixtures in different proportion with gasoline were prepared. The prepared mixtures did not show any separation hence, fuel grade ethanol was achieved [6]. Besides its use as fuel, ethanol and alcohols generally are in wide use in industry and laboratory as reagents, and solvents. Ethanol and methanol can be made to burn more cleanly than gasoline. Because of its low toxicity and ability to dissolve non-polar substances, ethanol is often used as a solvent in medical drugs, perfumes, and vegetable essences such as vanilla. Thus, the product of the pilot plant will have other economic uses in Nigeria besides its use as fuel.

## **5.0 Conclusion**

A hundred litres per day bio-ethanol pilot plant was operated. Qualitative analysis using infrared spectroscope showed that both the fermented juice and the distillate contain ethanol but the bottom product had none. The quantification of the ethanol strength using refractor-metric method showed that the fermented juice contains 4.28% ethanol while the distillate has 67%. The distillate was further dehydrated in the adsorption column. Mixture of the product from the adsorption column with gasoline showed that the product was nearly 100%. The ethanol-gasoline mixtures were used to start a new gasoline generator which started well without any difficulty. This work has demonstrated that fuel grade ethanol can be produced from sugarcane using indigenous technology in Nigeria.

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