

UTILIZATION OF AGRO-RESIDUAL (SUGARCANE

BAGASSE) FOR ETHANOL FUEL PRODUCTION

BY

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REG. NO: 2001/11558EH

DEPARTMENT OF CHEMICAL ENGINEERING

SCHOOL OF ENGINEERING AND ENGINEERING

TECHNOLOGY

FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA,

NIGER STATE.

NOVEMBER, 2007.

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**A PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMICAL
ENGINEERING, SCHOOL OF ENGINEERING AND ENGINEERING
TECHNOLOGY,**

MINNA, NIGER STATE.

**IN PARTIAL FULFILMENT OF THE REQUIREMENTS FOR THE
AWARD OF BACHELOR OF ENGINEERING (B.ENG). DEGREE IN
CHEMICAL ENGINEERING.**

NOVEMBER, 2007.

DECLARATION

I hereby declare that this research project "UTILIZATION OF AGRO-RESIDUAL (SUGARCANE BAGASSE) FOR ETHANOL PRODUCTION" was carried out solely by me under the guidance of my supervisor, Engr Aisha Farouk of the department of Chemical Engineering, School of Engineering and Engineering Technology, Federal University of Technology, Minna, Niger State.

All literature cited in this project has been duly acknowledged in the reference.

.....
Aisha Farouk

OYEDEPO KEHINDE OLAWALE

(2001/11558EH)

..... 30/11/07

Date

CERTIFICATION

This is to certify that this project "UTILIZATION OF AGRO-RESIDUAL (SUGARCANE BAGASSE) FOR ETHANOL PRODUCTION" was carried out by Oyedepo Kehinde Olawale (2001/11558EH) and supervised, moderated and approved by the following under listed persons as meeting the requirement for the award of bachelor of Engineering, B. Engr. Honors of Chemical Engineering on behalf of chemical engineering department, School of Engineering and Engineering Technology, Federal University of Technology, Minna, Niger State.

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Head of Department

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Date

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External Examiner

DEDICATION

This research project is dedicated to Almighty God for His guidance, protection, and infinite mercies towards me throughout my academic programme. Also to my entire family members for their support, they have been there through thick and thin.

ACKNOWLEDGEMENT

have come thus far in the race of life not by any other virtue of existence but by His GRACE which has always been sufficient for me in all facets of my existence. He is indeed the PILLAR of my life. I would like to express my profound gratitude to my supervisor, Engr. Aisha, who gave me the privilege to embark on this research work as well as for her guidance, correction and making useful suggestions that saw to the successful completion of this research work, may the Almighty bless and reward you boundlessly.

My unreserved gratitude goes to my amiable family; my parents Engr.E.B.Oyedepo and Mrs.R.Oyedepo, my brothers and sisters Mrs.E.Adeniyi, Taiwo, Bose and Shola for their all-round support. God bless you all, grant your heart desires and fulfill your dreams.

To my lovely uncle and family, Dn and Dns Oyedepo and family, I am grateful for all the support you accorded me. God bless you and reward you all. To my special friend, Elizabeth Ogundade, I appreciate your unflinching love and prayers. God bless you abundantly.

I am highly indebted to the following for their spiritual, moral and financial support; Rev A. Ajayi and family, all members of the Faith Baptist Church, U/Romi, Kaduna.

To those who made my stay in Federal University of Technology, Minna worthwhile especially, Ayokanmi Oshagbemi, Gabriel Amakoha, Hamza Abdulahi, Rosenje Ismaila, Habiba Ismail, Francis Opanachi, Joshua Gana, Tony Attah, and to all my compatriots in chemical engineering 2006/2007 session, I am indeed grateful to you all. I wish above all things that we will all meet at the topmost top.

ABSTRACT

This research work was based on the production of ethanol from sugarcane bagasse. The three major process embarked on in the production includes hydrolysis, fermentation, and distillation.

Two stage acid hydrolysis using sulphuric acid against 183g of biomass (bagasse) produced 205g/L of sugar (xylose). The sugar produced was then fed into a fermentor and allowed to ferment for three days at room temperature and the pH was maintained at about 4.5. Ethanol yield recovered was about 44.05%. After the distillation process, the final ethanol concentration was found to be 90.3%. Ethanol analysis shows a close relation with the standard values. Viscosity obtained was 3.4cP, specific gravity was 0.79g/L and refractive index obtained was 1.329. The possibility for agro-based residue (bagasse) to be used for fuel production has proven to be viable and would serve as an alternative for gasoline.

TABLE OF CONTENTS

Title page	i
Declaration	ii
Certification	iii
Dedication	iv
Acknowledgement	v
Abstract	vi
Table of content	vii
List of figures	xi
List of tables	xi
CHAPTER ONE	
1.0 INTRODUCTION	1
1.1 Aims and Objectives	2
1.2 Scope and Limitation of study	2
1.3 Justification of Study	2
CHAPTER TWO	
2.0 LITERATURE REVIEW	4
2.1 Sugarcane	4
2.1.1 Nomenclature and description	4
2.2 Sugarcane bagasse	4
2.2.1 Historical perspective	4

2.2.2 Composition	5
2.3 Processing sugarcane bagasse	5
2.3.1 Hydrolysis of bagasse	6
2.3.1a First stage dilute acid hydrolysis	6
2.3.1b Two stage dilute acid hydrolysis	6
2.3.1.2 Concentrated acid hydrolysis	6
2.3.1.3 Enzymatic hydrolysis	7
2.3.2 A case study of acid hydrolysis of hemicelluloses from bagasse	7
2.3.3 Sugar and lignin determination	8
2.3.4 Hemicellulose hydrolysate characterisation	8
2.4 Fermentation	10
2.4.1a Batch fermentation	10
2.4.1b Continuous fermentation	11
2.4.1c Fed- batch fermentation	11
2.4.2 Fermentation process	11
2.4.3 Microorganism selection	11
2.4.4 Medium	12
2.4.5 Fermentation with free cell	12
2.4.6 Ethanol determination	12
2.4.6a Measuring weight decreases	12
2.4.6b Using pycnometer method	12

2.4.7	Determination of efficiency yield and productivity	13
2.5	Distillation	14
2.5.1	Distillation via differential solubility	15
2.6	Bioethanol	15
2.6.1	History of bioethanol	15
2.6.2	Current research on bioethanol	16
2.6.3	Applications	16
2.7	Environmental issues	17
2.7.1	Ethanol and environment	17
2.7.2	Environmental impact of bioethanol production technologies and their life cycles assessment	18
4.1	(LCA)	18
2.8	Limitation	18
2.9	Bioethanol Characterization	19
2.9.1	Viscosity	19
2.9.2	Cold weather start	19
2.9.3	Flash point	19
2.9.4	Miscibility	19
2.9.5	Oxygen content	19
2.9.6	Cetane rating	19
2.9.7	Pour point	19
2.9.8	Color	19

CHAPTER ONE

1.0 INTRODUCTION

Since the 20th century, our major energy demand has been supplied by fossil fuels such as oil, coal and natural gas. Fossil fuel originates from deceased organisms that lived several million years ago and by time have been embedded in the earth's crust. Incineration of this fossil remains result in a net-increase of today's carbon dioxide level (Chandel et al, 2006).

Environmental issues such as the threatening increase in temperature caused by the greenhouse effect and the fact that fossil fuels are non-renewable resources, has increased the interest in producing fuels from renewable resources e.g. biomass (biomass includes the full range of plant and plant derived materials which consist of cellulose, hemicelluloses and lignin) Hayne et al, 1993.

Ethanol as well as other biofuels produced from plant biomass is an alternative to fossil fuels. Ethanol does not add to a net CO₂ atmospheric increase, thus there is in theory no contribution to global warming (Lin and Tanaka, 2006). Combustion of ethanol results in relatively low emissions of volatile organic compounds, carbon monoxide and nitrogen oxide. Today, the production cost of ethanol from lignocelluloses is still too high, which is the major reason why it has not made its breakthrough yet. When producing ethanol from sugarcane, the raw material constitutes about 40-70% of the production cost. By using cheaper waste production like sugarcane bagasse, the cost may be lowered. Sugarcane bagasse is a complex material which is a by-product of the sugarcane industry. Due to its abundant availability, it can serve as an ideal substrate for microbial processes for the production of value added product (Dominguez et al, 1996).

In Brazil, more than 60,000,000 tons of bagasse containing 50% moisture can be produced annually during the ethanol production season (Orlando Filho et al, 1994; Molina Junior et al, 1995). This waste has been used as a raw material to produce hydroxymethyl, furfural, paper pulp, acoustic boards, pressed woods and agricultural mulch (Dominguez et al, 1996). About 70% of the dry mass in lignocelluloses biomass consists of cellulose and hemicelluloses. If these two carbohydrates were utilized in an efficient hydrolysis process, the

hemicelluloses would be completely hydrolyzed to D-xylose (50-70% w/w) and L-arabinose (5-15%w/w), and the cellulose would be converted to glucose (Ladish 1989; Cao et al, 1995).

Sugarcane bagasse can be hydrolyzed using dilute acid to obtain a mixture of sugar with xylose as the major component. However, in the hydrolysate some by-products generated in the hydrolysis, such as acetic acid, furfural, phenolic compounds can be present. These are potential inhibitors of a microbiological utilization of this hydrolyzate (Dominguez et al, 1996).

Processes such as two-stage acid hydrolysis can be employed to produce xylose and glucose (Beck 1986). Treatment with dilute sulphuric acid at moderate temperatures (the first stage of acid hydrolysis) has proven to be an efficient means of producing xylose from hemicelluloses (Roberto et al 2994; silva 1996). In the second stage more drastic reaction conditions are employed and glucose can be produced from cellulose hydrolysis (Gregg and Saddler 1995).

In general, acid treatment is effective in solubilizing the hemicellulosic component of the biomass. Proper combinations of pH, temperature and reaction time can result in high yields of sugar, primarily xylose from hemicelluloses.

1.1 AIMS AND OBJECTIVES

- Utilization of agro-residual (sugarcane bagasse) for ethanol fuel production.
- Determination of the properties of ethanol fuel that makes it suitable alternative to fossil fuel.
- To compare the quality of ethanol fuel with that of fossil fuel.

1.2 SCOPE

This project work entails the production of ethanol fuel from sugarcane bagasse and the determination of the properties that makes it a viable alternative to fossil fuel.

1.3 JUSTIFICATION

Research on the utilization of ethanol fuel from biomass materials will serve to provide information on the production on the production process of ethanol as alternative source of energy to the conventional fossil fuel.

Bagasse a waste obtained after sugar has been extracted from sugarcane undergoes three major production processes viz; hydrolysis, fermentation and distillation. The cost of production

involving these processes is relatively cheap compare to the cost of other material like corn, sorghum, cassava etc. Analysis on the cost of economics of ethanol production from bagasse using *saccharomyces cerevisae* as yeast suggested pre-concentrating the sucrose obtained after hydrolysis of bagasse is economically in getting high ethanol concentration in fermented broth. Membrane distillation process has the lowest operational cost, is flexible, simple to use and is easy to maintain.

There are great opportunities that can be exploited from this research work because it will help to highlight economic ways in which an alternative to fossil fuel (gasoline) can be produced economically in small scale even at home if necessary equipment is available.

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 SUGARCANE

2.1.1 NOMENCLATURE AND DESCRIPTION

The sugarcane plant is known botanically as *saccharum officinarum*. It is a member of the grass family. The root system is fibrous and relatively shallow lying. The plant produces tillers (branch stems) which arise close to the ground level; thus in a well established plant, several major erect stems can be seen. Generally, there are two types of sugarcane which include the thick cane (this has thick stems and a large quantity of juice) and the thin cane (this is thinner, harder and has less juice) (Onwueme, 1993).

Sugarcane supplies more of the world's sugar than all other crops combined. It may be consumed directly as household sugar, may be used in sweets, confectionery and syrups, or may be fermented to produce alcohol for various uses. (Skerman, 1990).

Sugar obtained from sugarcane contains about 15-20% sucrose which is processed for the production of ethanol by fermentation. For the purpose of this project, sugarcane bagasse is of special interest as it is the feedstock for bioethanol production process.

2.2 SUGARCANE BAGASSE

2.2.1 HISTORICAL PERSPECTIVE

Bagasse is a biomass remained after sugarcane stalks are crushed to extract the juice. Most sugar factory produces 30% of bagasse out of its total crushing (Nguyen and Saddler, 1991).

In the very early days of sugar manufacture (18th century), the cane was passed through a single mill, and the defecation and concentration of the saccharine juice took place in a series of vessels mounted over a common flue with a fire at one end, and a stack at the other (Jamaica train method). This method required an enormous amount of fuel, and it was frequently necessary to sacrifice the degree of extraction to obtain the required amount of bagasse than could be burned as fuel. In addition, the amount of labour involved in spreading and collecting was great (Yu and Zhang, 2004; Moiser et al, 2006).

2.2.2 COMPOSITION OF BAGASSE

Bagasse consists of lignocelluloses, insoluble inorganic matter (ash), water soluble material (brix) and water. The lignocelluloses comprises of cellulose, hemicelluloses and lignin.

The cellulose is a linear crystalline homopolymer with a repeat unit of glucose strung together by beta-glucosidic bond.

The hemicelluloses consist of short, linear and highly branched chain of sugars. It is a heteropolymer of D-xylose, D-glucose, D-galactose, D-mannose and L-arabinose (Saha et al, 2003).

2.3 PROCESSING SUGARCANE BAGASSE

Bagasse undergoes the following processes for the production of ethanol fuel viz;

- Hydrolysis
- Fermentation
- Distillation

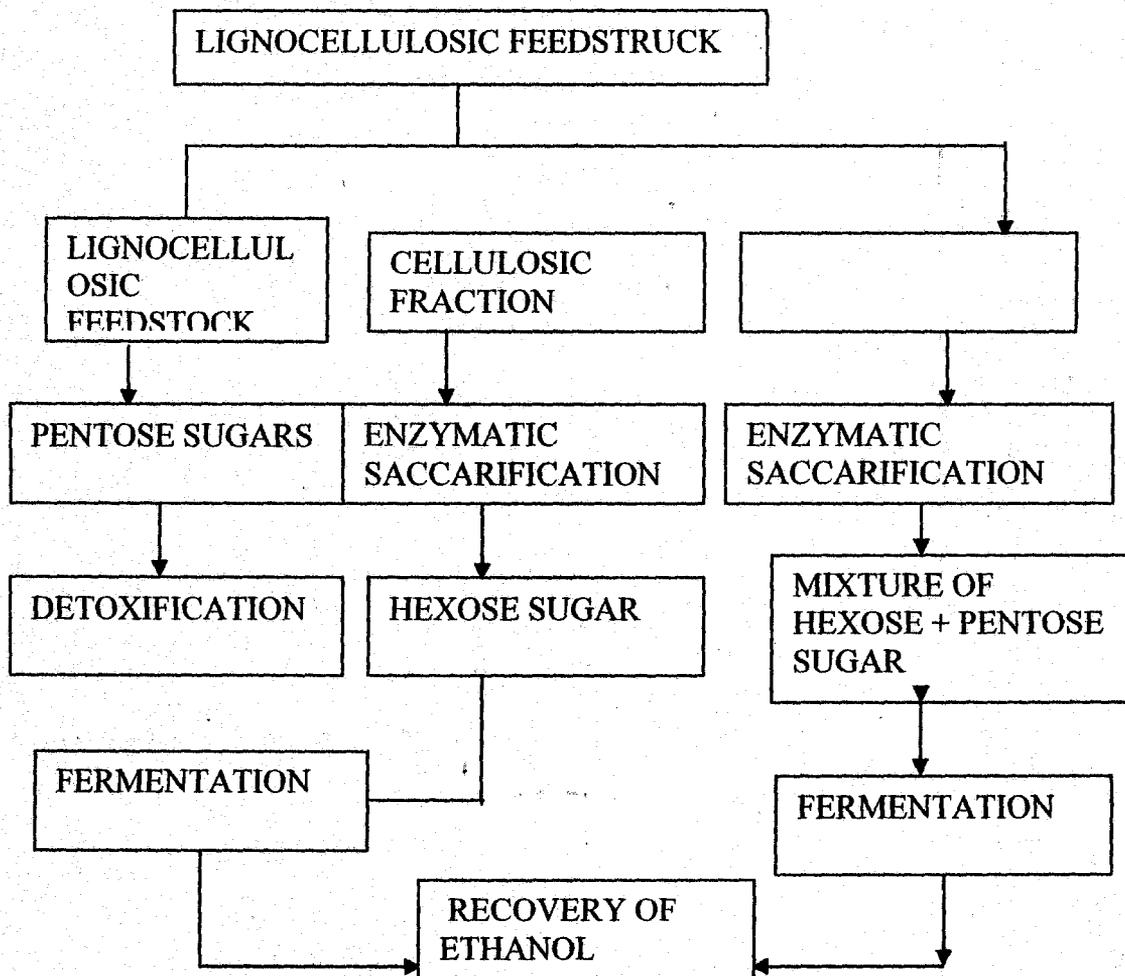


Fig 1: Dilute acid hydrolysis(first stage and two stage)

2.3.1 HYDROLYSIS OF BAGASSE

Hydrolysis is a chemical reaction that releases sugar which is normally linked together in complex chains (Keller et al, 2003). In early biomass conversion processes, acids were used to accomplish this. Recent research has focused on enzyme catalysts called "cellulose" that can attack these chains more efficiently leading to very high yields of fermentable sugars (Kim et al, 2003).

There two types of methods of hydrolysis viz;

- Acid (dilute and concentrated) hydrolysis
- Enzymatic hydrolysis

2.3.1a FIRST STAGE DILUTE ACID HYROLYSIS

The lignocellulosic material is first contacted with dilute sulfuric acid (0.75%) and heated to approximately 50 °c followed by transferring to the first stage acid impregnator where the temperature is raised to 190 ° c. Aaproximately 80% of the hemicelluloses and 29% of the cellulose are hydrolyzed in the first reactor. The hydrolysate is further incubated at a lower temperature for a residence time of 2hours to hydrolyze most of the oligosaccharides into monosacharides followed by the separation of solid and liquid fraction. The solid material again washed with plentiful of water to maximize sugar recovery. (Sanchez et al; 2004)

2.3.1b TWO STAGE DILUTE ACID HYDROLYSIS

In the two stage dilute hydrolysis process, first biomass is treated with dilute acid at relatively mild conditions during which the hemicellulose fraction is hydrolyzed and the second stage is normally carried out at higher temperature for development of cellulose to glucose. The liquid phase containing the monomeric sugar is removed between the treatments, thereby avoiding degradation of monosacharides formed. (Sanchez et al: 2004)

2.3.1.2 CONCENTRATED ACID HYDROLYSIS

This method uses concentrated sulfuric acid followed by a dilution with water to dissolve and hydrolyses the substrate into sugar constituents. This process provides complete and rapid conversion of cellulose to glucose and hemicelluloses to xylose with a little degradation. The concentrated acid process uses 70% sulfuric acid at 40-50°c for two to four hours. In a reactor,

the low temperature and pressure leads to minimizing the sugar degradation. The hydrolyzed material is then washed to recover the sugar. (Iranmahboob et al: 2002)

In the next step, the cellulose fraction has to be depolymerised. The solid residue from first stage is De-watered and soaked in 30 – 40% sulfuric acid for 50 minutes at 100^oc for further cellulose hydrolysis. The resulting slurry mixture is pressed to obtain second acid –sugar stream (approximately 18 % sugar and 30 % acid). Both the sugar stream from two hydrolysis steps is combined and may be used for subsequent ethanol production. (Iranmahboob et al: 2002) performed the concentrated acid hydrolysis of mixed wood chips found that maximum sugar recovery (28-78% of theoretical yields) was achieved at sulfuric acid concentration (26%) for two hours of residence time.

The primary advantage of the concentrated acid process is the potentials of high sugar recovery efficiency of about 90% of both hemicelluloses and cellulose fraction get depolymerized into their monomeric fractions. The acid and sugar syrup are separated via ion exchange and then acid is re-concentrated through multiple effect evaporation. The remaining lignin rich solid are collected and optionally palletized for fuel production.

2.3.1.3 ENZYMATIC HYDROLYSIS

The acid, alkaline or fungal pretreated lignocellulosics can be saccharified enzymatically to get fermented sugar (Ghose and Bisaria, 1979; Kuhad et al; 1997; Itoh et al 2003; Tucker et al; 2003). Bacteria and fungi are good source of cellulose, hemicellulase that could be used for hydrolysis of pretreated lignocelluloses. The enzymatic cocktails are usually mixture of several hydrolytic enzymes comprising of cellulose, hemicellulase and mannanases.

In the last decades, new cellulose and hemicellulases from bacterial and fungal sources have continued to be isolated and regular efforts have been made for improved production of enzymatic titres. (Aro, et al., 2005; Foreman, et al., 2003).

2.3.2 A CASE STUDY OF HYDROLYSIS OF HEMICELLULOSE FROM BAGASSE.

Sugar cane bagasses were obtained from Usina Nova America S/A (Taruma /sp Brazil). It was weighed, introduced into a 25ml laboratory reactor and indirectly heated with saturated steam to 140, 150, and 160^oc for 10-20 minutes. The final concentration of sulfuric acid in the hydrolysis suspension was 70 and 100 mg_{acid}/g_{dm} and the solid liquid ratio were 1:10. The steam

value was closed and the reactor was kept completely closed until cooled to room temperatures. Next the reactor was opened and the material inside was weighed. Three of the best conditions used for hydrolysis in the laboratory reactor (140⁰c for 10 minutes with 100mg_{acid}/g_{dm}, 140⁰c for 20 minutes with 100mg_{acid}/g_{dm} and 150⁰c for 20 minutes with 70mg_{acid}/g_{dm}) were also used for hydrolysis in a 25l semi pilot reactor utilizing 1000g of bagasse

2.3.3 SUGAR AND LIGNIN DETERMINATION.

Fermentable reducing sugars (FRS) and non fermentable reducing sugars (NRS) were determined by the method of Seaman et al 1945). Total carbohydrate content in the bagasse were evaluated after hydrolysis with 70% sulphuric acid using Dunings method (1949). FRS and NRS were closely related to the content of cellulose and hemicellulose respectively. Hemicelluloses recovery in water extract was measured by determining the content of NRS before and after hydrolysis with 4% H₂SO₄ (121⁰c for 15 minutes). The objective of this acid hydrolysis was to convert the oligosaccharides to monosacharies. The lignin was gravimetrically estimated from the insoluble residue by Moores methods (1967)

2.3.4 HEMICELLULOSE HYDROLYZATE CHARACTERIZATION

Total reducing sugars (TRS) were determined as glucose using the nelson method (1944). Glucose, Xylose and acetic acid concentration were determined by HPLC (an HPX-87H Bio Rad column with a RI16X detector). Aliquots of 20µl were analyzed as 45⁰c with 0.01N sulfuric acid as the eluent (flow rate of 0.6ml min⁻¹). Furfural hydroxymethy furfural were analyzed by HPLC (20µL of sample injected) under the following condition an RP18HP column an acetic acid: acetonitrile: water solution (1:10:80 volume ratio) as the eluent with flow rate of 0.8ml min⁻¹, a temperature of 25⁰c and a uv detector.

HPLC- (High pressure liquid chromatography)

2.3.5 TABLE SHOWING XYLOSE RECOVERED (%) AFTER SUGARCANE BAGASSE
ACID HYDROLYSIS

Hydrolysis conditions			Recovered
Temp (°c)	Time (min)	Acid concentration (mg acid/gdm)	xylose(%)
140	10	100	74.0
140	20	100	83.3
150	20	70	72.2

Adapted from (Dominquez, et al., 1996)

2.3.6 TABLE SHOWING PARTIAL COMPOSITION OF BAGASSE "IN NATURA" (%
W/W of the dry matter)

COMPOSITION	%(W/W)
TRS	70.9
Xylose	25.2
Glucose	41.0
NRS	26.3
Lignin	23.0
ASG	1.1
Moisture	47.8

Adapted from (Silva et al., 1995)

TRS- Total Reducing sugars

FRS- Fermentable reducing sugars

NRS- Non fermentable reducing sugars

%W/W of wet mater

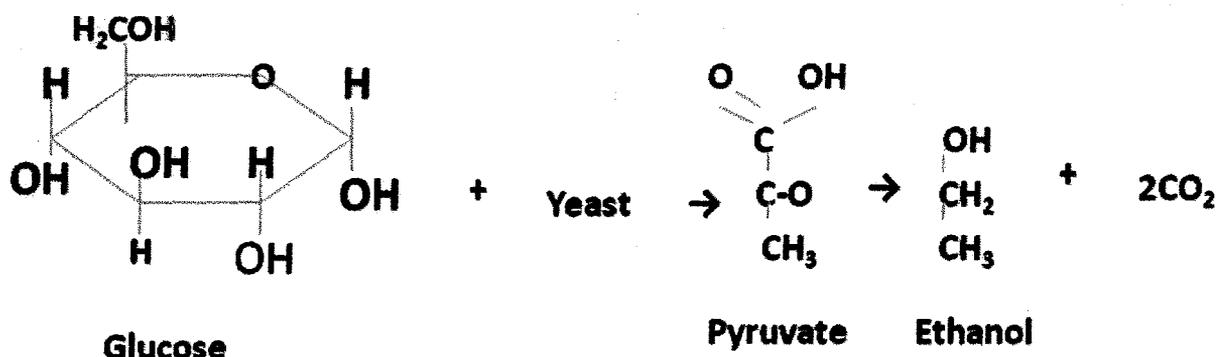
2.3.7 TABLE SHOWING CHEMICAL COMPOSITION OF ACID HYDROLYXATE.

CONSTITUENTS	%(W/W)
Xylose	18.5
Glucose	5.1
Acetic acid	3.7
TRS	27.7
Hydroxymethyl furfural	0.08
Furfural	2.0

Adapted from (Roberto et al., 1994).

2.4 FERMENTATION

Fermentation is mainly the breakdown of glucose into smaller molecules with the yield of ethanol and CO₂ as by-product using yeast. The chemical formula for fermentation is given as



Fermentation can be performed as batch, continuous and fed batch. The choice of most suitable process will depend upon the kinetic energy of the microorganism and type of lignocellulosic hydrolysate in addition to process economic aspects (Towolla, *et al.*, 1984).

2.4.1a BATCH FERMENTATION

In batch fermentation, substrates and yeast culture are charged into bioreactor together with nutrients. Most of the ethanol produced today are done by batch since the investment cost are low, donot require much control and can be establish with unskilled labor. Complete sterilization and management of feed stocks are easier than in other processes. It has greater flexibility that can be achieved by using a bioreactor for various product specifications. (Olsson and Hagerdal, 1996).

2.4.1b CONTINUOUS FERMENTATION

It can be performed in different kinds of bioreactor-stirred tank reactors (single or series) or plug flow reactors. Continuous fermentation often gives a high productivity than batch fermentation, but at low dilution rates which offers the highest productivities. (Alexander *et al.*, 1989). Studied the effect of shift in temperature and aeration in steady state continuous culture of *C. Shehatae* to determine the effect of ethanol exerted a delayed inhibitory effect on the specific rate of substrate utilization. The continuous process eliminate much of the unproductive time associated with clearings, recharging, adjustment of media and sterilization. (Alexander *et al.*, 1989)

2.4.1c FED BATCH FERMENTATION

It is also regarded as a combination of batch and continuous operation and very popular in the ethanol industry. In this operation the feed solution, which contains substrate, yeast culture and the required minerals and vitamins are fed at constant intervals while effluent is removed discontinuously (Schugeri, 2987; Taherzadah, 1999).

2.4.2 FERMENTATION PROCESSES.

Ethanol production from sugar is possible by using free or immobilized cells. Microorganism should be properly selected to provide the best possible combination of characteristics for the process and equipment used (Kohli, 1980). High volumetric productivities can also be obtaining with the combination of high cell concentration and high flow rates.

The process includes

- Microorganism selection
- Medium preparation

2.4.3 MICROORGANISM SELECTION

Saccharomyces cerevisiae remains the most exploited microorganism known to industries and is still the primary microorganism used for the production of virtually all portable and industrial ethanol (Lin and Tanaka, 2006).

2.4.4 MEDIUM

A litre of production medium should be prepared according to the requirements of *saccharomyces cerevisiae* containing about 22% sucrose, 0.3% dry yeast extract, 0.5% peptone, 0.15% $(\text{NH}_4)_2$ and 0.3% MgSO_4 in tap water (M.K.Hamdy; K.Kim, 1990).

2.4.5 FERMENTATION WITH FREE CELL

About 400ml fermentation medium should be inoculated with 50ml inoculums and the PH adjusted to about 5.0. It should be carried out in a rotary shaker at 200rpm and 28°C. The weight decrease should be measured every 2 hours in order to determine the amount of ethanol. Fermentation should be terminated after 96hours (R.W. Silman, 1992)

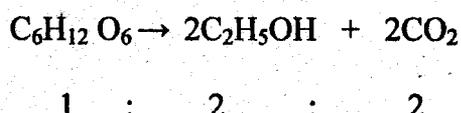
2.4.6 ETHANOL DETERMINATION

The amount of ethanol can be determined in two ways.

- Measuring the weight decreases of the system at intervals
- Using pycnometer method

2.4.6a MEASURING WEIGHT DECREASES.

According to the equation below, 1mole of glucose produce two moles of CO_2 which escapes from the reactor. The phenomenon is reflected as a weight decrease, which can be correlated to the amount of ethanol produced



In terms of weight, every grams of glucose can theoritically yield 0.51g of ethanol. It is assumed that 50% of glucose was used to produce ethanol and 50% of it to produce CO_2 ; thus there is a weight decrease due to the amount of CO_2 removed from the system and the amount of eethanol produced.

2.4.6b USING PYCNOMETER METHOD

A clean and dry pycnometer (50ml) was weighed (W). it was then filled with water up to a level predetermined for each pycnometer. Subsequently each was placed in a waterv bath at 20°C for 20 -30 min. at the end of this period the water above the pycometer was removed with blotting paper and weighed (W_{water}). This constant value was proportionalm to the volume of

the pycnometer. For ethanol determination, a 50ml sample was put into the distillation balloon. The pycnometer was rinsed with 25mlm of pure water and was added to thw distillation balloon. After that, the balooon was connected to the distilltion unit. The sample is distilled until alcohol is obtained up to just under the level of pycnometer. The pycnometer is filled with distill water up to level at 20⁰C. then it is dried and weighed (W_{sample}). The equation is used to find the density of the sample.

$$d (g/l) = \frac{(W_{\text{sample}} - W)}{(W_{\text{water}} - W)}$$

2.4.7 DETERMINATION OF EFFICIENCY, YIELD AND PRODUCTIVITY.

The efficiency, yield and volumetric productivities can be determined using the equations

$$\text{Efficiency (\%)} = \frac{(\text{gram ethanol produced})}{(\text{gram sucrose used})(0.51)} \times 100$$

$$\text{Yield (\%)} = \frac{(\text{gram ethanol produced})}{(\text{gram sucrose used})} \times 100$$

$$\text{Volumetric productivity} = \frac{\text{Ethanol formed (g/l)}}{\text{Volume of reactor (l)}}$$

2.4.8 The table below shows values of production processes in which microorganisms were used in free form.

	Batch	Continuous	Fed - batch
Fermentation time (i	96	262	240
Reactor Volume (l)	0.3	0.3	0.3
Initial concentration (g/l)	220	220	100
Added concentration (g/l)	-	480	540
Removed	-	-	-

concentration ($\frac{\text{g}}{\text{l}}$)				
Final	sul	26.59	164.48	22.71
concentration ($\frac{\text{g}}{\text{l}}$)				
Final	E	96.71	267.76	314.06
concentration ($\frac{\text{g}}{\text{l}}$)				
Efficiency (%)		86.19	75	96.22
Yield (%)		43.96	38.25	49.07
Volumetric	produ	1.01	1.022	1.309
($\frac{\text{g}}{\text{hr}}$)				
Amount of Alcohol(8.76	7.68	9.88

2.5 DISTILLATION

Distillation involves the separation of a liquid from other liquids or solids. Because each substance has a fixed rate of vapouration (which varies with heat) determined by the pressure, the vapours develop in a closed container to achieve equilibrium with fluid. One liquid can be separated from other matter by carefully controlling the heat applied to the mixture.

Alcohol's vapour pressure happens to be higher than water's, so ethanol's vapour pressure reaches an equilibrium with atmospheric pressure (the point at which a liquid boils) before water's vapour pressure does. But when water and alcohol are mixed, the boiling points of the separate constituents (water will boil at 100°C; alcohol boils at 78.3°C).

It is the rate of the water to alcohol which determines the actual temperature of boiling for the mixture. More alcohol lowers the boiling point and less raises it. This make the temperature of mash to raise throughout the distillation run as the alcohol is drawn off.

There are different methods of distillation which includes

- Simple pot distillation
- Distillation by solar energy

- Distillation by differential solubility
- Extractive distillation.

Solubility, by differential miscibility and by extractive distillation.

2.5.1 DISTILLATION VIA DIFFERENTIAL SOLUBILITY

Sulphur would be used to separate ethanol from water. It is mixed with the ethanol/water. The ethanol/water/sulphur mixture would be placed in a retort, where it could be heated and pressurized to a temperature above the critical temperature and pressure of ethanol [243°C and 63atm \approx (6.4Mpa) respectively] but below the critical temperature and pressure of water [374.1°C and 218.3atm (22.12Mpa), respectively]. The mixture would be retorted at a temperature slightly above 243°C and at a pressure slightly above 63atm (6.4Mpa), putting the ethanol in the superficial state, on which it should easily dissolve all three form of sulphur (including the form which is insoluble at ambient temperature and pressure). The water on the other hand would still be well below its critical state and still should not dissolve sulphur. The sulphur/ethanol mixture would settle to the bottom of the retort, which it could be piepled away under pressure and at high temperature. The sulphur/ethanol mixture would then be expanded to a lower temperature and pressure at which not as much sulphur could be dissolved in the ethanol and at which ethanol would partially separated from the mixture.

Further heating of the remaining mixture at a pressure of 1atm (0.1Mpa) would separate most of the remaining ethanol and sulphur.

2.6 BIOETHANOL

2.6.1 HISTORY OF BIOETHANOL

In 1925, Henry Ford had quoted ethyl alcohol (ethanol), as the fuel of the future. "He further stated that the fuel of the future is going to come from apples, weeds, sawdust-almost anything. There is fuel in every bit of vegetable matter that can be fermented". Today Henry Ford's futuristic vision significance can be easily understood. (Wyman, 1999: Lynd, 2004).

It is welcome to understand that the use of bioethanol as a source of energy would be more than just complementary for solar, wind, and other intermittent renewable energy sources in the long run (Yu and Zhang 2004). In the past, fossil fuel was considered more important because it was available and the vehicles then were designed to suit the petroleum fuel. Environmental considerations, energy and tax policies was limiting against the full extent of ethanol utilization (Moiser et al, 2006).

During the world ethanol production in 2004, it was estimated to produce 40giga liters which gave a higher yield than fossil fuel. This made companies like the U.S Department of Energy (DOE) to organize the bioethanol programme as a possible alternative to fossil fuel (Warren Gretz, 1990). Other countries like Brazil, India, and Australia to join in the programme using different sources as the feedstock for production.

Ethanol was discovered to have a much higher octane rating (116AK1, 129 RON) than ordinary gasoline (86/87AK1, 91/92 RON), allowing higher compression ratio and different spark timing for improved performance. A study conducted by Hu et al (2004) revealed that the E-85 fueled vehicle is better than the gasoline fueled car by balancing of all the 3E's in terms of the energy, environmental and economic aspects. (Fleming et al, 2006).

2.6.2 CURRENT RESEARCH ON BIOETHANOL

Recent research is on the development of genetically engineered microorganism that will ferment all possible sugars in biomass to ethanol at high productivity. Dr Lonnie Ingram at the University of Florida started an E.coli bacterium capable of metabolizing multiple sugars. Other bacterium like Zymomonas when cultured properly is capable of yielding sugars for producing ethanol (Min Zhang, 2002; Mike Himmel, 2004).

2.6.3 APPLICATION

The benefits of using ethanol fuel as an alternative for petrol fuel are enormous and are currently in use in some parts of the world. While it is worthy to note that ethanol fuel may not completely replace fossil fuel, it has its place both as an alternative to gasoline fuel and also as a blend with petrol. Its higher octane number compare to gasoline fuel is an advantage and can contribute to higher compression ratio and different spark timing for improved performance.

2.7 ENVIRONMENTAL ISSUES

2.7.1 ETHANOL AND THE ENVIRONMENT

Ethanol represents closed carbon dioxide cycle because after burning of ethanol, the released carbon dioxide is recycled back into plant material because plants use CO₂ to synthesize cellulose during photosynthesis cycle (Wyman, 1999; Chandel et al 2006). Ethanol production process only uses energy from renewable energy sources; no net carbon dioxide is added to the atmosphere, making ethanol an environmentally beneficial energy source. As energy demand increases, the global supply of fossil fuels cause harm to human health and contributes to the green house gas (GHG) emission. Hahn-Hagerdal (2006) alarmed to the society by seeing the security of oil supply and the negative impact of the fossil fuel on the environment, particularly on GHG emissions. The reduction of GHG pollution is the main advantage of utilizing biomass conversion into ethanol (Demirbas, 2007). Ethanol contains 35% oxygen that helps complete combustion of fuel and thus reduces particulate emission that pose health hazard to living beings. Generally, the advantage of ethanol fuel to the environment can be enumerated as follows:

- Ethanol is a clean-burning, renewable fuel.
- E85 is the cleanest burning fuel available to the market.
 - 10% ethanol enriched fuel reduces carbon monoxide better than
- any other gasoline by as much as 30%.
- Ethanol reduces tailpipe fine particulate matter emissions by 50%.
- Ethanol is biodegradable, meaning it would not harm ground water in the event of a spill.

2.7.2 ENVIRONMENTAL IMPACT OF BIOETHANOL PRODUCTION TECHNOLOGIES AND THEIR LIFE CYCLE ASSESSMENT (LCA)

Life cycle assessment (LCA) is a conceptual framework and methodology for the assessment of environment impacts of product systems on a cradle to grave basis (Graedel, 1999; Tan et al 2002). It encompasses the extraction of raw materials and energy resources from the environment, the conversion of these resources into the desired products, the utilization of the product by the consumer, and finally the disposal, reuse or recycle of the product after its service life (Tan et al, 2002). It is also an effective way to introduce environmental consideration in process and product design or selection (Azapagic, 1999).

In an extensive study by Kadam (2000), LCA of acid and enzymatic hydrolysis was compared. All environmental flows were examined from the product life cycle, its production and extraction from raw materials through intermediate conversion process, transportation, distribution and use. Dilute acid process was found better than the enzyme process in terms of greenhouse gas potential, natural resource depletion, acidification potential and eutrophication potential. The reason is dilute acid process sends a much higher proportion of biomass to the boiler for electricity production, which turn offsets large amounts of emissions.

2.8 LIMITATION

In spite of laboratory based bioethanol success stories, the production of fuel ethanol at plant scale still remains a challenging issue. A positive solution to this issue could bring economic advantage not only for fuel and power industry, but also benefit the environmental rehabilitation and balance issues and cause.

2.9 BIOETHANOL CHARACTERIZATION

Bioethanol characterization is based on the following characteristics;

2.9.1 VISCOSITY

The viscosity of a fuel is defined as the measure of the resistance of fuel to flow. Viscosity index is the measure of the constancy of the viscosity of a lubricant with changes in temperature with higher values indicating viscosity that changes little with temperature.

2.9.2 COLD WEATHER START

This is a factor which makes neat alcohol fueled engines difficult to cold start especially at ambient temperature below 10°C . This can be solved by adding additives.

2.9.3 FLASH POINT

The flash point of a fuel is the lowest temperature to which it must be heated in a specified instrument for the vapor given off to be sufficient enough to ignite when tested under specified conditions.

2.9.4 MISCIBILITY

This is the ability of a fuel system of mixing fluids in any ratio without separation.

2.9.5 OXYGEN CONTENT

This is a factor that contributes to the corrosion and wear problems as well as chemical degradation of material in a vehicle fuel system.

2.9.6 CETANE RATING

This is the measure of the ignition value of a fuel. The cetane number for ethanol fuel is always 8.

2.9.7 POUR POINT

This can be defined simply as the lowest temperature at which movement of fuel sample can be determined when the sample container is tilted under the condition of standard test method.

2.9.8 COLOR

Color is not a critical property; a rare change from the usual may indicate possible change in quality or possibly, contamination with another product. Color is the visual appearance of fuel.

CHAPTER THREE

3.0 RESEARCH METHODOLOGY

3.1 SOURCE: Sugar cane was obtained in Minna at mobil market which was later pressured to remove the sugar content. The waste being the bagasse used for the production of ethanol. other material used like the baker's yeast (*Saccharomyces cerevisiae*) was obtained from a local bakery (King's bakery) in Bosso, Niger state.

3.2 EQUIPMENT USED

Table 3.2a Showing list of equipment used

S/N	EQUIPMENT	SIZE	USES
1	Beaker	250ml	
2	Measuring cylinder	500ml	To measure the volu the solution.
3	Conical flask	100ml	To prepare st solution.
4	Thermometer	-	To measure temperature of the sample heated.
5	Stirrer	-	For proper agitation mixture.
6	Weighing Balance	-	Taking weight samples.
7	Heating mantle	-	To heat the sam specified temperatu

Table 3.2b Showing list of reagent use

S/N	REAGENT
1	Sulphuric acid (H ₂ SO ₄)
2	Sodium hydroxide (NaOH)
3	Yeast (<i>Saccharomyces cerevisiae</i>)
4	Distilled Water

3.3 PRODUCTION PROCEDURE

The bagasse was rinsed in water to remove all particles and then pressed to remove any considerable part of water left, then the bagasse was aired, milled and screened to a particle size of 250 μ (0.25mm).

183grams of bagasse (dry mass) was loaded into a conical flask and mixed with 500ml of distilled water. 0.75% of sulphuric acid was added to the mixture and heated with a heating mantle at a temperature of 70°C for 10 minutes with a heating proper stirring. The solid-liquid mixture was separated to obtain hydrolysate. The solid obtained was dried in oven for 5 minutes, and then 1.0% of sulphuric acid was added and heated at a temperature of 140°C for 5 minutes. The hydrolysate obtained was mixed with the first step. The acid-sugar mixture was separated using a column chromatography. NaOH was added to neutralize the acid effect and pH level adjusted to 0.5.

8.20g of baker's yeast, *saccharomyces cerevisiae* was added to the product obtained after hydrolysis for fermentation to take place. The temperature was maintained between 25-32°C for 3 days.

The ethanol was distilled using simple distillation apparatus where it was distilled at a temperature of 78.5°C to about 89°C, having a lower boiling point than that of water. It was redistilled to remove more of the water content.

3.4 ETHANOL FUEL ANALYSIS

Analysis was carried out for the purpose of comparing the properties with conventional fuel.

3.4.1 RELATIVE DENSITY (SPECIFIC GRAVITY)

The relative density of the fuel sample was measured using a density bottle plus water was noted as $(x + y)$. Also weight of bottle plus fuel sample was noted as $(x + v)$. The relative density was then calculated using the formular below

$$\text{R.d} = \frac{(x+v)-x}{(x+y)-x} = \frac{v}{y}$$

3.4.2 VISCOSITY

The cannon Fenske Viscometer was used to determine the viscosity of the sample. Some quantity of the sample was put in the viscometer up to the level of the upper mark. The time taken for the meniscus of the sample to fall from the upper meniscus of the bulb was noted and the viscosity.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION OF RESULTS

4.1 RESULTS

Table 4.1 showing sugar (glucose) recovered in % after a 2 stage dilute acid hydrolysis of bagasse.

Hydrolysis cor (stage 1&2)				Recovered
Mass of biomass	Temp (°C)	Time (min)	Volume of acid (mol %)	Sugar (%)
183	70	10	0.75	117.6
120	140	5	1.0	87.4

Table 4.2 Showing the readings obtained for the fermentation process.

Fermentation stage (Days)	Temp (°C)	pH	Initial substrate (g/L)	Final sul (g/L)	Final E conc (%)	Efficiency (%)	Yield (%)
1	26	4.5	205	26.86	90.3	86.4	44.05
2	25	4.5	205	26.86	90.3	86.4	44.05
3	26	4.5	205	26.86	90.3	86.4	44.05

Tables 4.3 Properties of Ethanol produced.

Analysis	Unit	Standard Values		Result
		Gasoline	Ethanol	
Specific Gravity	g/L	0.8	0.78	0.79
Flash Point	$^{\circ}\text{C}$	-6.7	12.8	10.5
Viscosity	cP	1.2		3.4
Refractive Index		1.36		1.329
Cetane Number		5-20	8	
Octane Number		86-94	100	98
Ignition	$^{\circ}\text{C}$	280	365	320
Boiling point	$^{\circ}\text{C}$	30-180	78.6	78.4

4.2 DISCUSSION OF RESULT

Sugarcane bagasse was used as the feedstock to produce ethanol using three major processes viz; hydrolysis, fermentation and distillation.

The result as stated in table 4.3 shows that the ethanol produced has a specific gravity of 0.79 which falls within the range of standard specification for ethanol fuel. This indicates a better lubricity index for flex fuel engines and could contribute to longer injector fuel life. The flash point obtained was 10.5°C which is higher than that of gasoline. It makes it easier to handle in case of fire outbreak. The octane number of ethanol was much higher which gives ethanol a higher octane rating allowing higher compression ratio and different spark timing for improved performance. The refractive index obtained was 1.329 which falls within the standard value. It indicates that the strength of the ethanol is high. Also the value of viscosity obtained was also high. The high value leads to complete burning of ethanol and less emission to reduce the ozone precursors by 20-30%.

The overall observation from the fuel analysis of ethanol produced shows that when compared with petro fuel (gasoline) has a better blending, relatively cheaper and environmental friendly.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

Developing alternative natural sources to replace traditional fuel (fossil fuel) has been of interest because of the increasing world wide concern for environmental protection.

Based on investigation, sugarcane bagasse was found to be relatively cheap and promising feedstock for ethanol production. Bagasse was used to carry out this research work by three major processes viz: dilute acid hydrolysis, fermentation and distillation. The result obtained shows a close relation with the standard result and when compared with gasoline has higher advantage like high octane rating which serves as a better antiknock agent than gasoline. The highest average percentage composition of ethanol recovered was 90.3%

The benefits of ethanol fuel are enormous. It is a clean fuel hence environmental friendly, it is cheap to produce and has an alternative advantage to gasoline.

5.2 RECOMMENDATION

1. Optimization and scale-up, which are outside the scope of this work, could then follow.
2. It is also recommended that the procurement of zeolite 3A molecular sieve for the dehydration process to produce fuel grade ethanol be done.
3. Advance in pretreatment by acid catalyzed hemicelluloses hydrolysis or employing an integrated approach in the form of consolidated bioprocessing with application of novel, tailored cocktails of enzymes for cellulose breakdown.
4. It is also recommended that genetically engineered microorganism that could ferment all possible sugar in biomass to ethanol at high productivity be used.
5. Since ethanol and water forms an azeotrope mixture at 89.43mol%, higher composition of ethanol could be achieved if an azeotropic distillation is employed with benzene molecular is

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APPENDIX

CHARACTERIZATION OF RESULTS

(1) Efficiency(%) = $\frac{\text{gram of ethanol produced} \times 100}{\text{Sucrose used} \times 0.51}$

$$= \frac{90.3}{205 \times 0.51} \times 100$$

$$= 86.4\%$$

(2) Yield (%) = $\frac{\text{gram ethanol produced}}{\text{Sucrose used}} \times 100$

$$= \frac{90.3}{205} = 44.05\%$$

(3) Specific gravity = $\frac{(x + v) - x}{(x + y) - x} = \frac{v}{y}$

Where

X = weight of empty bottle = 35.5g

V = weight of sample

Y = weight of water

$$= \frac{V}{Y}$$

$$= \frac{395.5}{500}$$

$$= 0.793\text{g/L}$$

(4) Viscosity = time taken \times c

$$= 20.2 \text{ sec} \times 0.17$$

$$= 3.4 \text{ cP}$$

(5) Refractive index (n_d) = $1.327338 + (3.93470 \times 10^{-4} \times \text{zeiss}) - (20.4467 \times 10^{-8} \times \text{zeiss}^2)$

Where zeiss = 18 = 1.329