PRODUCTION OF ETHANOL FROM MILLET

BY

AWELEWA KOLAWOLE MOBOLAJI

(2006/24171EH)

A PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMICAL ENGINEERING,

FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA

NIGER STATE

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

NOVEMBER, 2011.

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DECLARATION

I declare that this project report entitled "Production of Ethanol from Millet has been performed by me under the supervision of Engr A.D. Mohammed. No part of this report was presented for any other degree or diploma elsewhere at any institution to the best of my knowledge.

AWELEWA.M. KOLAWOLE
Name of Student

____
Signature

____
Date

CERTIFICATION

This is to certify that this project report titled "Production of Ethanol from Millet by Awelewa Kolawole Mobolaji meets the requirement for the partial fulfillment of the award of Bachelor of Engineering (B.Eng.) Degree in Chemical Engineering, Federal University of Technology Minna.

Project Supervisor
Engr. A.D Mohammed

Date

Dr. M.O Edoga
Head of Department

Date

External Examiner

23/02/2012
Date

DEDICATION

This work is dedicated to God Almighty for his relentless mercy upon my life.

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My profound gratitude goes to Almighty God for his divine grace, inspiration, favour, courage and strength given to me in the course of my studies.

I am grateful to my parents MR. J.B Awelewa and Princess C.A Awelewa, my uncle Mr. E.S Awelewa, Pastor M.O Adeleye, Mr Aderemi Adeleleye my siblings MJ, FM and Topsy. My cousins Emmanuel, Michael, Precious, Esther and Gabriel. Thank you all for your support.

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ABSTRACT

Bio-ethanol production using millet as substrate was carried out with Alkaline Hydrolysis (KOH) before inoculating Fermenter organism (Saccharimyces Cereviseae) which took 14 days to complete the fermentation. Ethanol was recovered from the fermented product by distillation, dehydration and further distillation process at 78.3 ^{0}C. From the results of ethanol yield per volume of hydrolyzed sample, the yield of ethanol increases with increase in volume of hydrolyzed sample. This implies that, to obtain a higher yield, then a large quantity of hydrolysate has to be used. The sample characteristics for density, boiling point, flash point and moisture content are 0.787 g/cm^3, 78.3 ^{0}C, 13.5 ^{0}C and 4.2 % respectively. These results show the validity of the experiment.

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Chapter One

1.0 INTRODUCTION

ioethanol is a renewable energy source produced mainly by the sugar fermentation process, lthough it can also be synthesized by chemical processes such as reacting ethylene with steam. thanol fuel blends are widely sold in the United States of America. The most common blend is 0 % ethanol and 90 % petrol. Vehicle engine required no modification to run on and vehicle arranties are not affected. Only flexible fuel vehicle can run on up to 85 % ethanol and 15 % etrol blends (Oyeleke and Jibrin, 2009).

he natural energy resources such as fossil fuel petroleum and coat are being utilized at a rapid te and these resources have been estimated to last only a few years. Therefore, alternative ergy sources such as ethanol, methane and hydrogen are being considered some biological rocesses have rendered possible routes for producing ethanol and methane in large quantities.

worldwide interest in the utilization of bioethanol as an energy source has stimulated studies n the cost and efficiency of industrial processes for ethanol production (Badgers, 2002).

uman activities generate large amounts of waste such as crop residue, solid waste from mines and municipal waste. They may become a nuisance and sources of pollution. It is therefore important to handle them judiciously to avoid health problems, since these wastes may habour pathogenic micro-organism Agricultural wastes including wood, herbaceous plant crops and forest residues, as well as animal wastes are potentially huge source of energy. In Nigeria, large quantities of these wastes are generated annually and are vastly under utilized. The practice is usual to burn them or leave them to decompose. However, studies have shown that these residues could be processed into liquid fuel such as biogas and bio-ethanol or combusted to produce electricity and heat. Ethanol production processes only use energy from renewable sources and there is not net Co_2 emission to the atmosphere, thus making ethanol and environmentally beneficial is the only liquid transportation fuel that does not contribute to the green house gas effect. This reduction of green house has emission is the main advantage of utilizing biomass conversion into ethanol (Sun, Y. and cheng, 2002).

Traditionally, ethanol has been produced in batch fermentation with fungal strains such as aspergillus niger mucor mucedo and saccharomyces cervisiae, which can not tolerate high concentration of ethanol. Therefore improvement programmes are required in order to obtain alcohol-telerant strains for fermentation

Zymomonas mobilis, a gram-negative bacterium, is considered an alternative organism in large scale ethanol production. Its advantages over yeast include higher sugar uptake and ethanol yield, lower biomass production and higher ethanol tolerance. The only limitation of Z mobilis, compared to the yeasts, is that its utilizable substrate range is restricted to glucose, fructose and sucrose. The organism can be isolated from palmwine or rotten oranges. Several agricultural wastes have been tested for their bioethanol – production, however the present study will utilize waste agricultural residues (Millet) for the production of bioethanol.

1.1 Aim of the research work

The aim of this research work is to produce ethanol from a millet

1.2 Objectives of Research work

The objectives of the research work are as follows:

- Sample collection
- Extraction, hydrolysis and fermentation
- Characterization of ethanol

1.3 Scope of the Research Work

The scope of the research work is limited to the physio-chemical production and characterization of ethanol.

1.4 Justification of the Research work

The completion of this research will encourage the use of biofuel from ethanol as a substituent to fossil fuel and other fuel additives. This will also boost the economy of millet production in large quantity in some area of the nation (Nigeria). However, it could also eliminate pollution effect posed by the use of fossil fuel with high sulphur content.

1.5 Problem statement of the Research work

The study will concentrate on the barrier of Bioethanol Production with availability of raw material and food security.The promotion of Bioethanol requires huge amount of arable crops needed for traditional purposes such as food production. The establishment of the land area that is available for the production of energy crops are very limited and restricted to some regions.also The project work is to develop a pollutant free environment and generate ethanol as a fuel energy and feedstock for our Automobile,Machineries,Industries and also Utilization of waste millet as a feedstock for the production of ethanol.

Chapter Two

2.0 LITERATURE REVIEW

2.1 Ethanol

Ethanol, also called ethyl alcohol, pure alcohol, grain alcohol, or drinking alcohol, is a volatile, flammable, colourless liquid. It is a powerful psychoactive drug, best known as the type of alcohol found in alcoholic beverages and in modern thermometers. Ethanol is one of the oldest recreational drugs. In common usage, it is often referred to simply as alcohol or spirits.

Ethanol is a straight-chain alcohol, and its molecular formular is C_2H_5OH. It's empirical formular is C_2H_6O. an alternative notation is CH_3-CH_2-OH_2 which indicates that the carbon of a methyl group (CH_3-) is attached to the carbon of a methylene group (-CH_2-), which is attached to he oxygen of a hydroxyl group (-OH) it is a constitutional isomer of dimethyl ether. Ethanol is often abbreviated as EtOH, using the common organic chemistry notation of representing the ethyl group (C_2H_5) with ethanol (Oyeleke and Jibrin, 2009).

The fermentation of sugar into ethanol is one of the earliest organic reactions employed by humanity. The intoxicating effects of ethanol consumption have been known since ancient times. In modern times, ethanol intended for industrial use is also produced from by-products of petroleum refining.

Ethanol has wide spread use as a solvent of substances intended for human contact or consumption, including scents, flavorings, colourings, and medicines. In chemistry, it is both an essential solvent and a feedstock for the synthesis of other products. It has a long history as a fuel for internal combustion engines (Akande and Mudi, 2008).

2.1.1 History of Ethanol

Ethanol has been used by humans since prehistory as the intoxicating ingredient of alcoholic beverages. Dried residues on 9,000-year-old pottery found in china imply that alcoholic beverages were used even among Neolithic people. Its isolation as a relatively pure compound was first achieved by the Arab chemist, Al-kindi (Al-kindus, 801-873),who unambiguously described the distillation of wine. Jabir Ibn Hayyan (721-815) also contributed to the development of distillation techniques, mentioning the flammable vapors of boiled wine. In 1796, John Tobias Lowitz obtained pure ethanol by filtering distilled ethanol through activated charcoal. Antoine Lavoisier described ethanol as a compound of carbon, hydrogen, and oxygen, and in 1808 Nicolas Theodore de sans sure determined ethanol's chemical formula. Fifty years later, Archibald Scott Couper published the structural formular of ethanol, which placed ethanol among the first compounds whose chemical stricture had been determined (Wang *et al*, 2004).

Ethanol was first prepared synthetically in 1826 through the independent efforts of Henry Hennel in Great Britain and S.G serullas in France. In 1828, Michael faraday prepared ethanol by acid-catalyzed hydration of ethylene, a process similar to that which is used today for industrial ethanol synthesis. Ethanol was used as lamp fuel in the united state as early as 1840, but a tax levied on industrial alcohol during the civil war made this use uneconomical. This tax was repeated in 1906, and from 1908 onward ford model automobiles could be adapted sellers of ethanol fuel were accused of being allied with moonshiners, and ethanol fuel again fell into disuse until late in 20th century (Boulton, 1996).

2.1.2 Physical Properties of Ethanol

Some of the physical properties of ethanol are as follows; miscibility, hygroscope, surface tension reduction and flammability .

2.1.2.1 *Miscibility*

Ethanol is a versatile solvent, miscible with water and with many organic solvents, including acetic acid, acetone, benzene, carbon tetrachloride, chloroform, diethyl ether, ethylene,glycol, glycerol, nitromethane, pyridine, and toluene. It is also miscible with light aliphatic hydrocarbons, such as pentane and hexane, and with aliphatic chlorides such as trichloroethane and tetrachloroethylene (Kirk Orthmer, 2008).

Ethanol's miscibility with water contents and that of longer-chain alcohols (five or more carbon atoms), which water miscibility decrease sharply as the number of carbon increases. The miscibility of ethanol with alkanes is limited to alkanes up to undecane, mixtures with dodecane and higher alkanes show a miscibility gap below a certain temperature (about 13 ^{0}C for dodecane). The miscibility gap tens to get wider with higher alkanes and the temperature for complete miscibility increases.

Ethanol-water mixtures have less volume than the sum of their individual components at the given fractions. Mixture of equal volumes of ethanol and water results in only 1.92 volumes of mixture. Mixing ethanol and water is exothermic. At 298 K up to 777 J/mol are set free. (Kirk Orthmer, 2008).

2.1.2.2 *Hygroscopy*

Hydrogen bonding causes pure ethanol to be hygroscopic to the extent that it readily absorbs water from the air. The polar nature of the hydroxyl group causes ethanol to dissolve many ionic compounds, notably sodium and potassium hydroxides, magnesium chloride, calcium chloride, ammonium chloride, ammonium bromide, and sodium bromide. Sodium and potassium chlorides are slightly soluble in ethanol. Because the ethanol molecule also has a non polar end, it will also dissolve non polar substances, including most essential oils and numerous flavoring, coloring, and medicinal agents (Kirk Orthmer, 2008).

2.1.2.3 *Surface Tension Reduction*

The addition of even a few percent of ethanol to water sharply reduces the surface tension of water. This property explains the "tears of wine" phenomenon. When wine is swirled in a glass, ethanol evaporation quickly form the thin film of wine on the wall of the glass. As the wine's ethanol content decreases, its surface tension increases and the thin film "beads up" and runs down the glass in channels rather than as a smooth sheet (Akande and Mudi, 2005).

2.1.2.4 *Flammability*

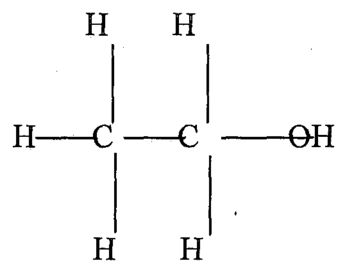
Mixtures of ethanol and water that contain more than about 50 % ethanol are flammable and easily ignited. Alcoholic proof is a widely used measure of how much ethanol (i.e. alcohol) such a mixture contains. In the 18^{th} century, proof was determined by adding liquor (such as rum) to gunpowder. If the gunpowder still just exploded, that was considered to be "100 degrees proof" that it was "good" liquor, hence it was called "100 degrees proof".

Ethanol-water solutions that contain less than 50 % ethanol may also be flammable if the solution is first heated. Some cooking methods call for wine to be added to a hot Pam, causing it to flash boil into a vapour, which is then ignited to burn off excess alcohol (BOULTON, 1996).

Other physical properties

- It has no action on litmus
- Ethanol is a colourless, volatile liquid with a characteristic taste and smell.
- It has a boiling point of 78.15 ^{0}C and a freezing point of -114.3 ^{0}C
- It has a density of 0.791 g/cm^3 viscosity at 20 ^{0}C is 1.2 Pas.

Table 2.1: Physical Properties of Ethanol

Property	Ethanol
Molecular formula	C_2H_5OH
Empirical point	79.4 ^{0}C, 352 k, 173 ^{0}F
Melting point	-114 ^{0}C
Density	0.789 g/ml
Solubility in water	Miscible
Refractive index (nD)	1.36 (25 ^{0}C)
Appearance	Colourless liquid
Molar mass	46.07 gmol-1
Condensed structural formula	CH_3CH_20H
Structural	

Source: (Badgers, 2002)

2.1.3 Chemical Properties of Ethanol

Ethanol is classified as a primary alcohol meaning that the carbon to which its hydroxyl group is attached has at least to hydrogen atoms attached to it as well. Many of the reaction of ethanol occur at its hydroxyl group.

2.1.3.1 *Ester Formation*

In the presence of acid catalysts, ethanol reacts with carboxylic acids to produce ethyl esters and water;

$RCOOH + HOCH_2CH_3 \longrightarrow RCOOCH_2CH_3 + H_2O$ 2.1

This reaction, which is conducted on large scale industrially, requires the removal of the water from the reaction mixture as it is formed. Esters react in the presence of an acid or base to give back the alcohol and carboxylic acid. This reaction is known as Saponification because it is used in the preparation of soap. Ethanol can also form esters with inorganic acids. Diethyl sulphate and phosphorus pent oxide respectively. Diethyl sulphate is a useful ethylating agent in organic synthesis. Ethylnitrite, prepared from the reaction of ethanol with sodium nitrite and sulphuric acid, was formerly a widely used diuretic (Oyeleke and Jidrin, 2009).

2.1.3.3 *Halogenation*

Ethanol is not used industrially as a precursor to ethyl halides, but the reactions are illustrative. Ethanol reacts with hydrogen halides to produce ethyl halides such as ethyl chloride and ethyl bromide.

$CH_3CH_2OH + HCl \longrightarrow CH_3CH_2Cl + H_2O$ 2.2

These reactions require a catalyst such as zinc chloride. HBr requires refluxing with a sulphuric acid catalyst. Ethylhalides can, in principle, also be produced by treating ethanol with more specialized halogenating agents, such as thionyl chloride or phosphorous tribromide.

$CH_3CH_2OH + SOCl_2 \longrightarrow CH_3CH_2Cl + SO_2 + HCl$ 2.3

Upon treatment with halogens in the presence of base, ethanol gives the corresponding haloform (CHX_3, where X = Cl, Br I). This conversion is called the haloform reaction. An intermediate in the reaction with chlorine is the aldehyde called chloral.

$4Cl + CH_3CH_2OH \longrightarrow CCl_3CHO + 5HCl$ 2.4

2.1.3.3 *Acid- Base Chemistry*

Ethanol is a neutral molecule and the PH of a solution of ethanol in water is nearly 7.00. Ethanol can be quantitatively converted to its conjugate base, the ethoxide ion ($CH_3CH_2O^-$), by reaction with an alkali metal such as sodium;

$2CH_3CH_2OH + 2Na \longrightarrow 2CH_3CH_2ONa + H_2$ 2.5

Or a very strong base such as sodium hydride;

$CH_3CH_2OH + NaH \longrightarrow CH_3CH_2ONa + H_2O$ 2.6

2.1.3.4 *Dehydration*

In the presence of excess concentrated tetraoxosulphate (vi) acid at a temperature above 170^0c, ethanol reacts to form ethyl hydrogen tetraoxosulphate (vi), $C_2H_5HSO_4$, which then decompose to yield ethane.

$C_2H_5OH_{(aq)} + H_2SO_{4(aq)} \longrightarrow C_2H_5HSO_{4(aq)} + H_2O_{(l)}$............ 2.7

$C_2H_5HSO_{4(aq)} \longrightarrow C_2H_4(g) + H_2SO_{4(aq)}$ 2.8

On the other hand, if the alkanol is in excess, and the temperature is lower, it will react with the acid to yield ethoxyethane (diethyl ether)

$C_2H_5OH_{(aq)} + H_2SO_{4(aq)} \longrightarrow C_2H_5HSO_{4(aq)} + H_2O_{(l)}$ 2.9

$C_2H_5HSO_{4(aq)} + C_2H_5OH_{(aq)} \longrightarrow C_2H_5OC_2H_5(g) + H_2SO_{4(aq)}$ 2.1

The formation of ethane and ethoxyethane from ethanol can be considered as dehydration reactions because the overall reactions are the loss of one molecule of water from one molecule and two molecules of ethanol respectively.

$$C_2H_5OH \xrightarrow{-H_2O} C_2H_4 \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 2.11$$

$$2C_2H_5OH \xrightarrow{-H_2O} C_2H_5OC_2H \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 2.12$$

(Osei, 1990)

2.1.3.5 *Oxidation*

Ethanol is readily oxidized to Ethanal by warming with potassium heptaoxodichromate (vi) solution which has been acidified with dilute tetraoxosulphate (vi) acid. Ethanal is given off as a pungent vapour, leaving behind a green liquid.

$$C_2H_5OH_{(aq)} \xrightarrow[\text{from potassium heptaoxodichromate (vi)}]{[O]} CH_3CHO_{(g)} + H_2O_{(l)} \ldots\ldots\ldots\ldots 2.13$$

On further oxidation in the presence of excess tetraoxosulphate (vi) acid, the ethanal is converted to ethanoic acid.

$$CH_3CHO_{(g)} \xrightarrow[\text{from potassium heptaoxodichromate}]{[o]} CH_3COOH_{(g)} \ldots\ldots\ldots\ldots 2.14$$

These oxidations can also be brought about catalytically by passing the ethanal vapour finely divided copper at 300 ^{0}C, and the ethanal vapour over manganese (ii) ethanoate respectively. Wine sometimes becomes sour on prolonged exposure to air because of the bacterial oxidation of ethanol to ethanoic acid.

Generally, primary alkanols are oxidized to alkanals and carboxylic acids, and secondary alkanols to alkanones, while tertiary alkanols are not oxidized (Osei, 1990).

2.1.3.6 *Combustion*

Ethanol readily burns in air or oxygen with a pale blur flame, yielding water and carbon (w) oxide

$$C_2H_5OH + 3O_{2(g)} \longrightarrow 3H_2O_{(l)} + 2CO_{2(g)} \quad \ldots\ldots\ldots\ldots \quad 2.15$$

(Osei,1990).

2.1.4 Types of Alcohol and Alcoholic Beverages

There are different types of alcohol. Some are used in chemistry laboratories and industries e.g isopropyl and methyl alcohol. Isopropanol or isopropyl alcohol is also used in industrial processes as well as in home cleaning products and skin lotions. It is also commonly known as "rubbing alcohol" methanol, or methyl alcohol or wood alcohol has been used as a methylated spirits. It is found in cleaning solvents, paint removes, photocopier developer and anti-freeze solutions. As such, it is often available in large quantities inexpensively. It is similar to ethanol but the end product after it is digested by the body is formaldehyde, which is poisonous. This is responsible for " alcohol poisoning . Methanol poisoning leading to blindness has been known to occur on consuming even small amounts. Another type of alcohol is ethayl alcohol, also known as ethanol. This has been consumed by human beings for its intoxicating and mind-altering effects. The term alcohol, unless specified otherwise, refers to ethanol or ethyl alcohol. It is a thin, clear liquid with harsh burning taste and high volatility. It is usually consumed in diluted concentrations of absolute (i.e. 100 percent) ethyl alcohol. Ethyl alcohol is also used as a reagent in some industrial applications. For such use, ethyl alcohol is combined with small quantities of methanol with the mixture being called "denatured ethanol" to prevent theft for human consumption (Akande and Mudi, 2005).

2.1.5 Brief Description of Alcoholic Beverage

2.1.5.1 *Wines*

Wines are made from a variety of fruits, such as grapes, peaches, plums or apricots. The most common wines are produced from grates. The soil in which the grapes are grown and the weather conditions in the growing season deter mine the quality and taste of the grapes which in turn affects the taste and quality of wines. When ripe, the grapes are crushed and fermented in large vats to produce wine (Oyeleke and Jibrin, 2009)

2.1.5.2 *Beers*

Beer is also made by the process of fermentation. A liquid mix, called wort, is prepared by combing yeast and malted cereal, such as corn, rye, wheat or barley. Fermentation of this liquid mix produces alcohol and carbon dioxide. The process of fermentation is stopped before it is completed to limit the alcohol content. The alcohol so produced is called beer. It contains 4 to 8 percent of alcohol . (Oyeleke and Jibrin, 2009)

2.1.5.3 *Whisky*

It is made by distilling the fermented juice or cereal grains such as corn, rye or barley. Scotch whisky was originally made in Scotland. The word "scotch"has become almost synonyms with whisky of good quality ((Oyeleke and Jibrin, 2009)

2.1.5.4 *Rum*

It is a distilled beverage made form fermented molasses or sugarcane juice and is aged for at least three years. Caramel is sometimes used for colouring.

2.1.5.5 *Brandy*

It is distilled from fermented fruit juices,. Brandy is usually aged in oak casks. The colour of brandy comes either form the casks or from caramel that is added.

2.1.5.6 *Gin*

It is a distilled beverage. It is a combination of alcohol, water and various flavours. Gin does not improve with age, so it is not stored in wooden casks.

2.1.5.7 *Liquors*

They are made by adding sugar and flavouring such as fruits, herbs or flowers to brandy or to a combination of alcohol and water, most liquors contain 20-65 percent alcohol. They are usually consumed in small quantities after dinner,

Table 2.2: Types of Alcoholic Beverages

Beverage	Source	Alcoholic Content (percentage)
Brandy	Fruit juices	40-50
Whisky	Cereal grains	40-55
Run	Molasses/sugarcane	40-55
Wines (sherry etc)	Grapes (also other fruits)	10-22
Beer cereals	Cereals	4-8

Source: (Oyeleke and Jibrin, 2009)

2.1.6 Types OF Ethanol Feed Stock

There are three types of ethanol feed stocks, namely; grain ethanol, sugar ethanol and cellulosic ethanol.

2.1.6.1 *Grain Ethanol*

Grain ethanol is made from corn, sorghum , millet etc. with the continued growth of the ethanol industry, the demand fir corn has increased. This heightened heed has resulted in the question of whether corn growers can meet the needs for both ethanol production and its traditional food uses recently, the ethanol industries have been changed as the reason for a rise in consumer food prices. Much of the debate has centered on the notion that the increased demand for corn for ethanol production will reduce the amount of food that is available for human consumption. However, ethanol is produced from field corn which human cannot digest its raw form. Ethanol production utilizes only the starch portion of the kernel. Therefore, the kernel is broken down with the starch being converted to ethanol and protein, vitamins, minerals and fiber being sold as high-value livestock feed. (Badgers, 2002)

2.1.6.2 *Cellulosic Ethanol*

Cellulosic ethanol is made from agricultural wastes such as; millet, wood, plant fiver, switch grass, crop residues etc. It is a fully renewable advanced biofuel that can be use in today's cars. It is one of the most effective ways to reduce green house gas emissions and gasoline consumption use in road transport, and can deliver CO_2 reduction benefits similar to improved vehicle efficiency. (Badgers, 2002)

Cellulose ethanol can significantly:

- Lower overall green house gas emission
- Reduce reliance on improved oil and increase energy security.
- Help build rural economics and improve farm income

2.1.6.3 *Sugar Ethanol*

Sugar ethanol is made from sugar cane. The production of ethanol from sugar cone requires lesser energy and compared to cellulosic ethanol. The feed stock is crushed to remove the juice. The wet juice contains sugar that can be digested by micro organisms to ethanol, carbon dioxide and water in a fermenter. The product from the fermenter contains a mixture of ethanol and water. For ethanol to be used as fuel in the internal combustion engines, the water has to be removed (almost 100 % ethanol is required). The product of digestion is store in storage tank from where a pump with drew the solution and fed to a distillation column.

Distillation is the primary step in purifying the ethanol form the mixture (and other residual solids) after fermentation. Distillation can produce an ethanol water solution containing 95 % ethanol the ethanol is "boiled off" form the rest of the solution in a distillation column.

Upon leaving the distillation process, the ethanol is at 89.1 % purity. At this point, the alcohol and water form an azeotrope, which means further separation using heat cannot occur. In order to blend with gasoline, the last 10.9 % of water must be removed, producing ethanol at 98.6 to 99.0 proof (Oyeleke and Jibrin, 2009).

2.1.7 Production of Ethanol

Ethanol can be produced generally be fermentation of sugars, also by hydration of ethylene form petroleum (Awafo *et al*, 1998).

2.1.7.1 *Fermentation of Sugar*

Fermentation has been used before 1930 and has remained the most commonly, and the process involves the conversion of carbohydrate (sugar) by the reaction of an enzyme called zymase which is found in yeast cells (saccharomyces cerevisae).The main raw material for this process includes corn, barley, wheat , cassava, rice, it can also be produced from biomass such as grass and trees, other raw materials are feed stock containing sugar. The starch is broken down on hydrolyzed into sugar by the action of some organic catalyst or enzymes called alph-amylase. Dilute acid or alkaline may be used instead of enzymes. Ethanol produced by fermentation or synthesis is obtained as dilute aqueous solution with carbon dioxide evolved.

Fermentation which is a slow decomposition of large molecules (organic molecules) into small molecules by micro organism is a very important process in the conversion of starch sugar to ethanol. A common micro organisms used is yeast it contains a variety of enzyme which decompose starch, it is first treated with malt (partly germinated barley) (Ababio, 2000).

$$2(C_6H_{10}O_5)_{n(s)} + n\ H_2O_{(l)} \xrightarrow[\text{from malt}]{\text{diastase}} nC_{12}H_{22}O_{11(aq)} \quad .. ..\ 2.1.6$$

$$C_{12}H_{22}O_{11(aq)} + H_2O \xrightarrow[\text{from malt}]{\text{diastase}} 2C_6H_{12}O_{(aq)} \quad .. ..2.1.7$$

$$C_6H_{12}O_6 \xrightarrow[\text{from yeast}]{\text{diastase}} 2C_2H_5OH_{(aq)} + 2CO_{2(g)} \quad .. ..2.1.8$$

Ethanol obtained from fermentation has a maximum concentration of 18 % as yeast cells die above the concentration. Further concentration and purification is done by fractional distillation ethanol which is generally sold called rectified spirit is 95 % pure and is further purified by distilling over quick lime (CaO) to obtain absolute ethanol which contains 99.5 % ethanol. It is very hygroscopic and must be kept away from atmospheric moisture. Various alcoholic drinks contain different concentration of ethanol (Ababio, 2000).

2.1.7.2. ***Hydration of Ethylene***

Hydration of ethylene to produce ethanol involves passing the mixture and a large quantity of steam at temperature of 300 ^{0}C (570 ^{0}C) and a pressure of several thousand pound per inch square (PSI) over solid catalyst. The ethylene used as raw material in this process is obtained during cracking of petroleum oil for the production of gasoline.

Ethylene can be hydrated directly by passing a mixture of ethylene and steam over tetraoxosulplhate (vi) acid, the catalyst at 500 to 600 ^{0}C and 80 to 100 atm.

$$C_2H_{4(aq)} + H_2O_{(g)} \xrightarrow[500\text{–}600^0]{H_2SO_4} C_2H_5OH$$

Or ethylene is hydrated indirectly; in this case, ethane is first absorbed in 95 % tetraoxosulhpate (iv) at 80 ^{0}C and 30 atm to from ethylhydrogen tetraoxosulphate (iv).

This is then hydrolyzed by bonding with water

$C_2H_{4(g)} + H_2SO_4 \longrightarrow C_2H_5HSO_{4(aq)}$ 2.1.9

$C_2H_5HSO_{4(aq)} + H_2O_{(l)} \longrightarrow C_2H_5OH_{(aq)} + H_2SO_{4(aq)}$ 2.2.0

Ethanol can also be produced in the laboratory by;

(i) Hydrolyzing Iodo ethane with an alkali

$CH_3CH_2I + NaOH_{(aq)} \longrightarrow C_2H_5OH_{(aq)} + NAI$ 2.2.1

(ii) Hydrolyzing Ethylester with hot alkali

$C_2H_5COC_2H_{5(l)} + NaOH_{(aq)} \longrightarrow C2H_5OH_{(l)} + C_2C_5COONa_{(aq)}$ .. 2.2.2

$C_3H_7COOH_{5(l)} + NaOH_{(aq)} \longrightarrow C_2H_5OH_{(l)} + C_3H_7COOHNa_{(aq)}$ ……2.2.3

2.1.8 Uses of Ethanol

Some benefits that could be derived from ethanol by the nation and to individuals are as follows;

- Industrial uses: As a fuel, the largest single uses of ethanol are as a motor fuel and fuel additive.
- Used as a cooking and lighting fuel.
- Ethanol fuel cells: Ethanol may be used as a fuel to power direct ethanol fuel cells (DEFC) in order to produce electricity and the by-products of water and carbon dioxide.
- Rocket fuel: Ethanol was commonly used as fuel in early bi-propellant rocket vehicles.
- Alcohol beverages: Ethanol is an important industrial ingredient and has widespread use as a base chemical for other organic compounds.
- Feed stock: Ethanol is an important industrial ingredient and has widespread use as a base chemical for other organic compounds.

- Antiseptic use: Ethanol is used in medical wipes and in most common antibacterial hand sanitizer gets at a concentration of about 62 % (percentage by volume, not weight)
- Antidote use: Ethanol can be used as an antidote for poisoning by other, more toxic alcohol, in particular methanol and ethylene glycol.

Other Uses

- Ethanol is easily miscible in water and is a good solvent. Ethanol is less polar than water and is used in perfumes, paints and tinctures.
- Ethanol is also used in design and sketch art makers, such as Copic, and Tria.
- Ethanol is also found in certain kinds of deodorants. (Ababio, 2000).

2.2 Brief History of Millet

Millets are some of the oldest of cultivated crops. The term millet is applied to various grass crops whose seeds are harvested for food or feed. The five millet species of commercial importance are proso, foxtail, barnyard, brown top and pearl. In China, records of culture for foxtail and proso millet extend back to 2000 to 1000 BC Foxtail millet (Setaria italica L.) probably originated in southern Asia and is the oldest of the cultivated millets. It is also known as Italian or German Millet. Its culture slowly spread westward towards Europe. Foxtail millet was rarely grown in the U.S. during colonial times, but its acreage increased dramatically in the Great Plains after 1850. However, with the introduction of Sudan grass, acreage planted to foxtail millet decreased.

Proso millet (Panicum miliaceum L.) was introduced into the U.S. from Europe during the 18th century. It was first grow along the eastern seaboard and was later introduced into the Dakotas where it later was grown on considerable acreage. In North Dakota acreage has ranged from 50,000 to 100,000 acres while in Minnesota only a few thousand acres have been grown. (Oyeleke and Jibrin, 2009)

Today, foxtail millet is grown primarily in eastern Asia. Proso millet is grown in the Soviet Union, mainland China, India and Western Europe. In the United States, both millets are grown principally in the Dakotas, Colorado and Nebraska.

Barnyard or Japanese millet (Echinochloa frumentaceae L.), is a domesticated relative of the seed, barnyard grass. It is grown for grain in Australia, Japan and other Asian countries. In the United States, it is grown primarily as aforage.

Brown top millet (Panicum ramosum) is a native of India and was introduced into the United States in 1915. It is grow in southeastern United States for hay or pasture and bird and quail feed plantings on game preserves. It is sometimes sold to Minnesota sportsmen for this purpose. Seed and forage yields of brown top millet have been low in Minnesota tests and it did to compete well with weeds.

Pearl or cattail millet (Pennisetum glaucum) originated in the African savannah and grown since prehistoric time. It is grown extensively in Africa, Asia, India and Near East as a food grain. It was introduced into the United States at an early date but was seldom grown until 1875. It is primarily grown in southern United States as a temporary pasture. It is preferred over sudan grass as a forage crop in the south. Varieties planted at Rosemount, Minnesota produced very little seed, and their forage yield was low compared to foxtail varieties.

The most commonly grown millets in the midwestern states are proso and foxtail with a limited acreage of barnyard . (Oyeleke and Jibrin, 2009).

2.2.1 Uses

The major uses of proso millet are as a component of grain mixes for parakeets, canaries, finches, lovebirds, cockatiels and wild birds and as feed for cattle, sheep, hogs and poultry. Millet for birdfeed purposes is often grown under contract. Large bright or red seed is preferred, and premiums are sometimes paid for superior quality. Two types of bird feed mixes are marketed. One type is for wild birds and the other for caged birds. The cage bird mixes require the better quality proso for which premiums are paid

Proso millet as livestock feed is similar to oats and barley in feeding, value. It is commonly fed in ground form to cattle, sheep and hogs. Whole seed can be fed to poultry. The protein values compare favorably with sorghum and wheat and are higher than corn. Porso also has considerably higher levels, due to attached hulls. The average composition of proso gain is shown in Table 1. Proso performs best in livestock rations when fed in mixture with other grains. If the amino acid levels are balanced in the feeding value to hogs is nearly equal to corn. Proso can be cut for hay, but it is not as suit as foxtail for this process.

Table 2.3: Showing the Composition of Millet.

S/No	Composition	Range (Percentage)
1	Protein	12
2	Fiber	8
3	Fat	4
4	Vitamin	B

Source: (Oyeleke and Jibrin, 2009).

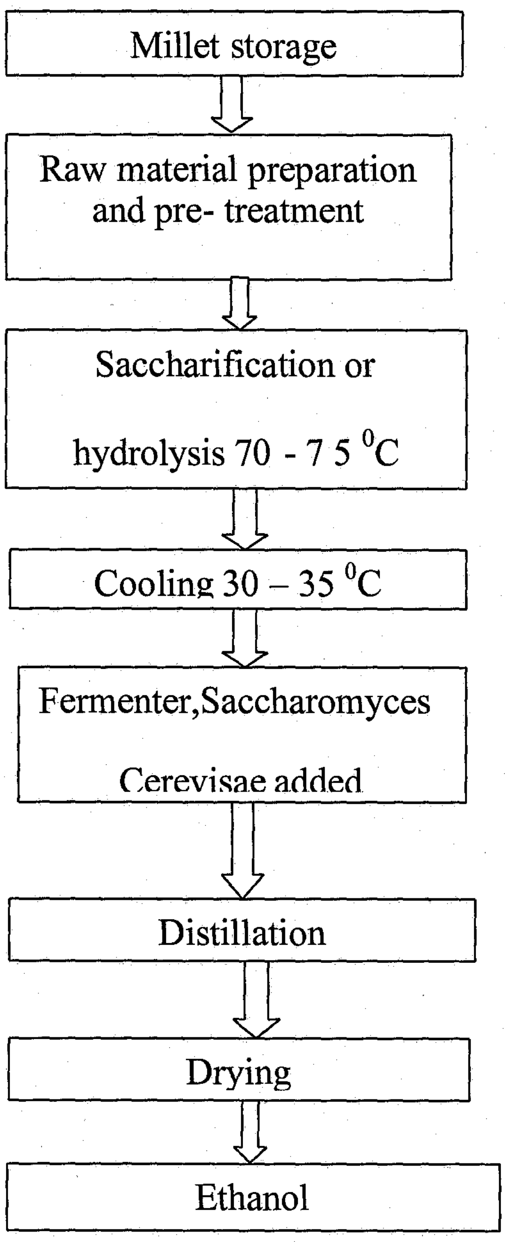


Figure 2.1: Block Diagram for Ethanol Production from Millet

Chapter Three

3.0 METHODOLOGY

3.1 Equipment and Materials Used

3.1.1 List of Equipment Used

- Heat source
- Water bath
- pH meter
- Conical flask
- Thermometer
- Measuring cylinder
- Test tube
- Stirring rod
- Alcohol distillation apparatus

3.1.2 List of Materials Used

- Millet
- Distilled water
- Potassium hydroxide (KOH)
- Ethanoic acid (CH_3COOH)
- Filter cloth
- Fehling's solution
- Iodide (I)
- Saccharomyces cerevisiae
- Foil paper
- Cotton wool

3.2 Experimental Methodology Procedure

3.2.1 Pre-treating and Milling Procedure

- The sample was washed, dried and milled.
- 300 g of the sample was collected and soaked in 750 cm^3 of water and for a period of 24 hours, after which it was filtered with the aid of a filter cloth.
- 600 cm^3 of the filtrate was collected and made up to 1000 cm^3 with boiled water.

  The mixture was stirred continuously to avoid formation of lumps it was then allowed to cool. On cooking, a thick jelly mass was formed.
- The gelatinized mixture was then poured into a 2000 cm^3 flask for hydrolysis.
- To verify the presence of starch in the sample, 2 cm^3 of the sample was measured in the test tube from the bulk sample, iodide reagent was then added dropwise and stirred, the colour change was observed and recorded.

  Observation: sample changes from brown to blue black.

  Inference: starch is present in sample.

3.2.2 Hydrolysis Procedure

- 100 cm^3 of 0.5m potassium hydroxide was added to the sample and immersed in the water both for hydrolysis and the temperature was maintained at 75 ^0C for an hour
- 50 % ethanol acid was then added to serve as a terminator of the hydrolysis reaction after which the mixture was set aside to cool.

3.2.3 Test For Reducing Sugars Procedure

- To 3 cm^3 to the hydrolyzed sample, few drops of Fehling's solution was added in a conical flask and heated, the colour change was observed and recorded.

Observation: Sample changes to brick red precipitate

Inference: Simple sugar is present.

3.2.4. Fermentation Reaction Procedure

- 10 g, 20 g, 30 g and 40 g of active yeast (saccharomyces cerevisiae) were separately added to 100 ml of warm water (37 ^{0}C) and allowed to stand for a period of 2 hours while the yeast was being monitored for growth. The yeast was then added to the sample at growth stge.
- Each of the mixture was transferred into the fermenter and allowed to ferment for a period of 14 days. At this stage, the enzyme inverts and zymase contained in the yeast acted on simple sugars degrading then to ethanol and carbon dioxide

3.2.5 Procedure for Filtration of Fermentation Broth

After fermentation, the fermentation broth was filtered with a filter cloth and a clear ıid was obtained for distillation.

3.2.6. Distillation Process Procedure

The filtrates were then distilled at 78.3 ^{0}C using alcohol distillation apparatus. The set up included the round bottom flask containing the filtrate placed in the heating mantle and the mouth fixed to the end of the set up. Hoses were connected to the condenser to supply water from the tap for cooling the condenser and letting water out of the condenser simultaneously.

The temperature on the heating mantle was set to the standard temperature for the product of ethanol which is 78.3 ^{0}C. As the filtrate was heated, the vapour rose and entered into the condenser tap water was passed into and out of the condenser using the rubber pipes and this condenser vapour was collected into the beaker at the other end of the distillation set up as the distillate (ethanol). The process was repeated for the other samples.

3.2.7 Further Ethanol (Distillate) Purification

The distillate was further purified by the use of lime (calcium oxide). Lime, a basic oxide, when added to the ethanol, absorbed the water to form calcium hydroxide, ($Ca(OH)_2$), an alkaline solution. The calcium hydroxide formed was separated from ethanol by further distillations which leave pure ethanol.

3.2.8 Test for Ethanol Procedure

2 cm^3 of alcohol was treated with iodine and sodium hydroxide, the colour change was erved and recorded.

Observation: A yellow precipitate was formed.

Inference: Ethanol is present.

3.2.9 Specific Gravity

Introduction: Specific gravity or relative density is the ratio of the density (mass of a unit volume) of a substance to the density of a given reference material. It usually means relative density with respect to water. The term "relative density" is often preferred in modern scientific usage. If a substance's specific density is less than one (1), then it is less dense than the reference; if greater than 1 then it is denser than the reference. If the specific density is exactly 1 then the densities are equal; that is, equal volumes of the two substances have the same mass. If the reference material is water then a substance with a specific density (or relative density) less than 1 will float in water. A substance with a specific gravity greater than 1 will sink.

Apparatus: Weighing balance, Empty bottle

Procedure: Weight of an empty bottle was determined by a weighing balance as W_1

The percentage alcohol yield at varying volume of filtrate is calculated as follows;

Volume of sample fermented = Vs

Volume of alcohol formed = Va

Percentage alcohol converted = Va/ Vs

Weight of bottle and sample was determined by the weighing balance as W_2, also weight of bottle and distilled water was determined as W_3.

Equal volume of sample and distilled water were used.

Hence, specific gravity = $\frac{W_2 - W_1}{W_3 - W_1}$ 3.0

Applications: specific gravity is commonly used in industry as a simple means of obtaining information about the concentration of solutions (juices, honeys etc) and acids.

3.3 Density

Introduction: the density of a material is defined as its mass per unit volume. The symbol most often used for density is g (the Greek letter rho). In some cases (for instance in the United States oil and gas industry), density is also defined as its weight per unit volume; although, this property is more properly called specific weight. Different materials have different densities, therefore density is an important concept regarding purity and packaging.

Apparatus: 100 ml graduated cylinder, digital weighing balance.

Procedure: the mass of the empty graduated cylinder was found by using the weighing balance after which 25 ml of ethanol was poured into the cylinder. The mass of the cylinder with 25 ml ethanol was determined using the weighing balance.

The density was then calculated using the formula

$$\text{Density} = \frac{Mass\ of\ Ethanol}{Volume\ of\ Ethanol} \qquad \text{........ 3.1}$$

Application: it is used to measure the ratio (specific gravity) of the density of a liquid grape to that of pure water.

3.3.1. Boiling Point

Introduction: the boiling point of a liquid is the temperature at which the vapour pressure of the liquid equals the atmospheric pressure surrounding the liquid.

A liquid in a vacuum environment has a lower boiling point than when the liquid is at atmospheric pressure. A liquid in a high pressure environment has a higher boiling point than when the liquid is at atmospheric pressure. Therefore, the boiling point of liquids with the surrounding environmental pressure also depends upon the surrounding environmental pressure.

Different liquids (at a given pressure) boil at different temperatures. The standard boiling point defined by IUPAC as at (1982) the temperature at which boiling occurs under a pressure of 1 bar.

Apparatus: Hot plate, beaker, thermometer.

Procedure: 20 ml of ethanol was placed in a beaker and heated on the hot plate. As the ethanol began to boil, a thermometer was inserted in the ethanol and the boiling point reading was recorded.

3.3.2. ***Flash Point***

Introduction: flash point of a volatile liquid is the lowest temperature at which it can vaporize to from an ignitable mixture in air. To Measuring a liquid flash point an ignition source is required. At the flash point, the ignition source is removed.

Apparatus: Pensky Marten closed cup apparatus light source (lighter), thermometer, stirrer.

Procedure: the apparatus consist of a small cup into which the sample is put. The sample was allowed to cool to about 20 ^{0}C some of the sample was put in the cup and closed so that no vapour was lost.

The cup was gradually heated while being stirred continuously to distribute the heat uniformly in the cup. At the regular intervals of 3-5 ^{0}C, the cup was opened and vapour in the cup was able to produce a monetary flame. The temperature range that produced the flame was recorded.

Applications: flash point is used to described the characteristic of liquid fuel and helps to characterize the fire hazard of liquids.

3.3.3 Moisture content

Introduction: The moisture content of a liquid or substance is the amount of moisture or water present in the liquid, which is expressed in percentage (%).

Apparatus: oven, evaporating dish, measuring cylinder

Procedure: 20 ml 0f ethanol was measured and placed in the evaporating dish and was kept in the oven for a period of one hour, after drying the volume was measured and recorded.

V_0= Volume of dish ,

V_1= volume of dish + sample before drying

V_2= volume of dish + sample after drying

$\%V$ = moisture content

$\%V = (V_2 - V_0) / (V_1 - V_0) \times 100$

3.3.4 Octane Rating

Introduction: This is a measure of the resistance of petrol to engine knocking. Engine knocking can damage the engine quickly. It is an important test for measuring the anti-knock quality of the gasoline (petrol or motor spirit).

Performance number is used to estimate knocking characteristics of aviation gasoline of octane number higher than 100.The standard reference fuels knock rating above 100 octane number are iso-octane and its blend s with tetraethyl Lead (TEL). Ethanol has 109 RON (Reseach octane number) nd 90 MON (Motor octane number) which equates to 99.5 AKI (Anti-knock Index).

Procedure: $\text{Octane number} = 100 + \dfrac{\text{performance number} - 100}{3}$

Application: It is an important test for measuring the anti-knock quality of the gasoline.

Chapter Four

4.0 RESULTS AND DISCUSSIONS

4.1 Experimental Results

ble 4.1: Result of Ethanol Production using Saccharomyces Cerevisiae from Distillation ocess.

ample	Volume of Hydrolysate (cm^3)	Volume of Ethanol (cm^3)	Ethanol Concentration %
Aillet	150	16	10.66
	200	19	9.50
	250	23	9.20
	300	26	8.66

ble 4.2 Properties of Experimental Ethanol Sample Compared to Laboratory Ethanol.

Property Sample	Laboratory Grade Ethanol	Experimental Ethanol
Molecular formula	C_2H_5OH	C_2H_5OH
Molar mass	46.07 g/mol	46.07 g/mol
Appearance	clear, colourless liquid	clear colourless liquid
Boiling	78.4 ^{0}c	78.3 ^{0}c
Density	0.789 g/cm^3	0.787 g/cm^3
Flammability	Flammable	flammable
Solubility	Highly soluble	Highly soluble
flash point	13 ^{0}C	13.5 ^{0}C
moisture content	0.3 %	4.2 %
% purity	99.8 %	95.8 %
Octane Rating	99.5	66.99
Refractive index	1.36	1.31

4.2 Discussion of Result

From Table 4.1, It was observed that as the volume of hyrolysate increases from either 150cm3 to 200 cm^3,200 cm^3 to 250 cm^3,250 cm^3 to 300 cm^3,the yield of ethanol recovered was found to increase 16 cm^3 to 19 cm^3,19 cm^3 to 23 cm^3,23 cm^3 to 26 cm^3 respectively which shows that for one to obtain a higher yield ,a large quantity of hydrolysate has to used. Also it was observed that for every increase in the volume of hydrolysate ,a decrease in the concentration of ethanol is observed that is 10.66 % to 9.50 %, 9.50 % to 9.20 % , 9.20 % to 8.66 % respectively ,which shows that as the volume of hydrolysate increases, the ethanol concentration (relatively quantity of ethanol recovered) gradually decreases.

Table 4.2 shows the properties of ethanol produced experimentally and the standard laboratory ethanol, The produced ethanol has the boiling point of 78.3 ^{0}C while Badgers (2002).from his research got boiling point of 78.2 ^{0}C which when compared with standard requirement 78.4 ^{0}C This research product was found to be averagely best of the two.

In the current table, the produced ethanol has the density of 0.787 g/cm^3 which shows that it met the standard laboratory grade ethanol of 0.789 g/cm^3 This is an indication that the produced ethanol can be used as fuel.

However, it was observed that the produced ethanol has the moisture content of 4.2 % which shows that it still contain amount of water present when compared with the standard laboratory which has a moisture content of 0.3 % .This result when compared to standard it was found not in agreement, as such this will reduce the percentage purity of ethanol.

Indeed going by the flash point results the laboratory ethanol has the standard flash point 13 ^{0}C meanwhile the experimental produced ethanol has flash point 13.5 ^{0}C, The flash point of the ethanol produced agreed with flash point standard but differ a little from the standard, as such the produced ethanol still be suitable for fuel usage.

Chapter Five

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

It was deduced that a high yield of ethanol will be obtained using moderate quantity of hydrolysate and the produced ethanol was found to meet the standard laboratory grade ethanol. The produced ethanol will be suitable for use as fuel.

The production of bio-ethanol from millet was achieved in the laboratory through the controlled combination of various units operations, hence agricultural by-products can be utilized in this form.

5.2 Recommendations

The following recommendations are made;

- Agricultural wastes as a source of ethanol or biofuel should further be focus for research purposes.
- Various types of hydrolysis method should be carried out on millet in the production.
- Research should be carried out using other substrate and micro-organism could be employed to carry out various processes involved in ethanol production.

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APPENDIX A

DENSITY

Density of C_2H_5OH

Weight of beaker = 0.78 g

Weight of beaker and ethanol = 20.46 g

Weight of $25cm^3$ of ethanol = 20.46 – 0.78 = 19.68 g

$$\text{Density} = \frac{\text{mass of ethanol}}{\text{volume of ethanol}}$$

$$= \frac{19.68}{25}$$

Density = $0.787 g/cm^3$

APPENDIX B

DETERMINATION OF PERCENTAGE ETHANOL YIELD

- Volume of 1st hydrolysate = 150 ml

Volume of distillate obtained from 15 ml = 16 ml

Yield (%) = $\frac{16}{150}$ x 100 = 10.66 %

- Volume of 2nd hydrolysate = 200 ml

Volume of distillate obtained = 19 ml

Yield (%) = $\frac{19}{200}$ x 100 = 9.50 %

- Volume of 3rd hydrolysate = 250 ml

Volume of distillate obtained = 23 ml

Yield (%) = $\frac{23}{250}$ x 100 = 9.20 %

- Volume of 4th hydrolysate = 300 ml

Volume of distillate obtained = 26 ml

Yield (%) = $\frac{26}{300}$ x 100 = 8.66 %

APPENDIX C

% Purity = 100 – moisture Content

Moisture Content = 4.2 %

Hence % Purity = 100 – 4.2

% Purity = 95.8 %

APPENDIX D

Octane Rating

$$\text{Octane Number} = 100 + \frac{(\text{Performance number} - 100)}{3}$$

Performance Number = % Purity of Ethanol = 95.8

$$\text{Octane number} = 100 + \frac{(0.958 - 100)}{3}$$

$= 100 + (- 33.014)$

$= 66.99$