RESEARCH ON THE PRODUCTION AND CHARACTERISATION OF FURFURAL FROM RICE HUSK

BY

OBIEFULA GIDEON OKEREKE

REG. NO; 2001/11606EH

DEPARTMENT OF CHEMICAL ENGINEERING, SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY, FEDERAL UNIVERSITY OF TECHNOLOGY MINNA, NIGER STATE.

NOVEMBER, 2007.

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A RESEARCH PROJECT SUBMITTED TO THE DEPARTMENT OF CHEMICAL ENGINEERING IN PARTIAL FULFILMENT OF THE REQUIREMENT FOR THE AWARD OF THE BACHELOR OF ENGINEERING (B. ENG.) DEGREE IN CHEMICAL ENGINEERING, SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY MINNA

NOVEMBER, 2007

CERTIFICATION

This is to certify that this project work "RESEARCH ON PRODUCTION AND CHARACTERISATION OF FURFURAL FROM RICE HUSK" was carried out by OKEREKE GIDEON OBIEFULA under the supervision of ENG. (Mrs.) AISHA A. FARUK and submitted to the chemical engineering department, federal university of technology Minna in partial fulfillment of the requirement for the award of bachelor of engineering (B. eng) degree in chemical engineering department.

for .... 30/11/2007 ....

Project supervisor — Date

ENG. (MRS.) AISHA A. FARUK

....

Head of department — Date

DR. M.O. EDOGA

....

External Supervisor — Date

DECLARATION

I, OKEREKE GIDEON OBIEFULA, solemnly declare that this project work "Research on production and characterization of furfural from rice husk" was carried out by me as a final year project in the department of Chemical Engineering, Federal University of Technology, Minna and has never been carried out elsewhere.

.... 5-12-07 ....

OKEREKE G. OBIEFULA DATE

DEDICATION

This project work is dedicated to my God first, and my parents, Eng. Kelechi Peter Okereke and Mrs. Ann Okereke, also to my siblings too.

ACKNOWLEDGEMENT

My most appreciable gratitude goes to the Almighty God who gave me the sound health, patience and wisdom to accomplish my research work.

Also, my thanks go to my parents for their indispensable support through out my study B.Eng program both financially and morally as well.

I must not forget my supervisor ENG. (Mrs.) Aisha A. Faruk who guided me throughout the entire period of this research work.

My sincere appreciation goes to my twin brother, and the entire members of my family for their support. Not leaving my course mates as a whole, also, my friends, Busayo, Bright, Onaivi, and Max.

Finally, it has been rough, but God with his infinite mercy saw me through, so I thank God for that.

ABSTRACT

The process involve in this project work which is the "Research on production and characterization of furfural from rice husk" is a single stage acid digestion and followed by steam distillation and evaporation. The process is carried out by weighing a certain amount of crushed rice husk and discharging it into a flat bottom flask, a mineral acid, (diluted) is added and it is placed on the heating source and steam distillation is undergone to obtain a mixture of furfural and water. Water is then evaporated from the mixture to obtain a pure furfural.

So at the end of this research work, it was confirmed that 300 micrometer particle size and at 45 minutes and 60 minutes of 5 grammes of the said particle gave the highest percentage yield

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CHAPTER ONE

1.0 INTRODUCTION

The name Furfural is not uncommon among the industrialist. It is an industrial chemical derived from variety at agricultural by – products like corncobs, oats, rice husks, wheat bran and saw dust. The name Furfural comes from the latin word Furfur, meaning bran, referring to its usual source.

Importantly, the usefulness of Furfural made it paramount important to produce commodity from abundant agricultural waste materials and which will contribute positively to the economy of the country. All this while; the source of Furfural to Nigeria is by importation which is either from Arabian countries or venzualla.

And Furfural is a liquid aldehyde colourless when freshly prepared but turns yellowish when in contact with air and it is the most important member of the heterocyclic compound called Furans has a pungent aromatic odour, reminiscent of almonds when freshly distilled.

The Furans families can be produced by single acid digestion and followed by distillation and evapouration. It is identified by their double unsaturated ring of four (4) carbon atoms and on oxygen with the aldehye structure of –CHO group in two positions. Furfural has a molecular weight of 96.08g/mol, boiling point of 161.7°C and freezing point of -36.5°C. Also has a refractive index of 20°C and 25°C to be 1.5261 and 1.5235 respectively. Density at 1.16g/moldm^3. It is misicible with most of the common organic solvent but slightly miscible with saturated aliphatic hydrocarbons and it is not subject to degradation by dilute mineral acids.

Also it is colourless when freshly distilled, but turns yellowish in contact with air and it is the most important heterocyclic compound called furans. Finally, Furfural has a pungent aromatic odour remiscent of almonds. When freshly distilled.

Importantly, furfural is used as a solvent in petrochemical refining to extract dienes(which are used to make synthetic rubber) from other hydrocarbons.

Furfural as well as its derivatives furfural alcohol can be used either by themselves or together with phenol, acetone or urea to make solid resins. Such resins ar eused in making fiber glass, some aircraft components and automotive brakes.

Finally, Furfural is used as a chemical intermediate for the manufacture of solvent furans and tetrahydrofurans. Hydroxymethylfurfural has been identified in a wide variety of heat process in foods for insecticides, fungicides and herbicides.

1.1 AIM/OBJECTIVES

Production and characterization of Furfural from rice husk

OBJECTIVES

1) To improve the rice husk value.
2) To reduce unemployment.
3) To reduce dependence on importation.
4) To encourage local industries in production of Furfural for commercial purposes.

1.2 JUSTIFICATION

(1) Furfural production in Nigeria will be environmental friendly which in essence will reduce the amount of waste disposal that could result to pollution.

(2) The Furfural production will be responsible for direct and indirect job creation therefore increasing the household income annually.

(3) It is also necessary to produce Furfural locally in Nigeria which will reduce dependence on importation that will depend on certain polices.

(4) In order to increase crop demand created by Furfural. Industry will as well increase the rice husk value.

(5) Finally, the cost of production of Furfural locally will reduce therefore contributing positively to Nigeria's economy.

1.3 SCOPE OF WORK

The purpose of this my research project works are:

(1) To produce Furfural from rice husks thereby reducing the cost of importation.

(2) To identify Furfural from other compounds by characterization.

(3) To determine the mass distribution of rice husk using sieve analysis method.

(3) To determine the amount of Furfural produced per grams of each rice husks using mineral acids.

(4) To determine the density of Furfural and it refractive index.

CHAPTER TWO

2.0 LITERATURE REVIEW

Furfural is a liquid aldehyde, colourless when freshly prepared but turns yellowish in contact with air and it is the most member of the heterocyclic compound called furans. These furans are identified by their double unsaturated rings of four carbon atoms and one oxygen atom with the aldehyde structure of –CHO group in two positions. It has a pungent, aromatic odour reminiscent of almonds when freshly distilled. Htpp://www.orgsynthesis.org/orgsyn/prep.asp?prep=cv 0280.

2.1 HISTORY OF FURFURAL

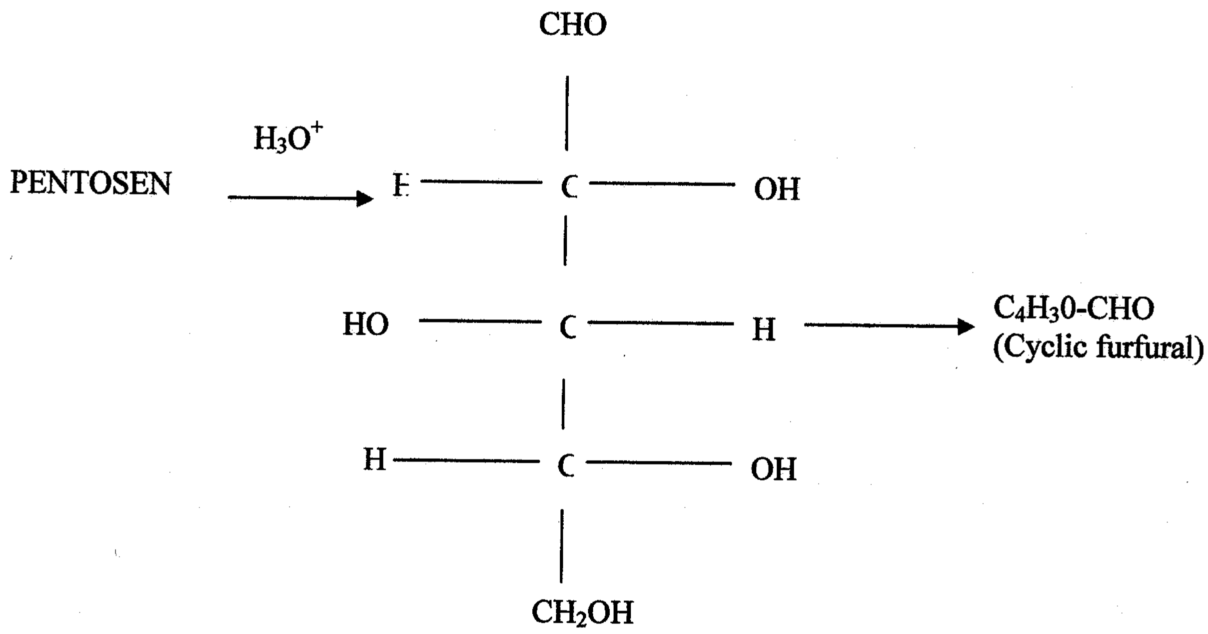
Theory reveals that furfural was first produced on a small scale in America in 1832 and after ninety (90) years of discovery, about two (2) thousand investigations were carried out and are reported in literature. Furfural was first produced in commercial quantity in 1922 from agricultural plant materials that contain pentosan. All the while the source of Furfural to Nigeria is by importation which is either from Venezuella or Arabian countries.

According to the published data of October 1978 estimated world capacity for furfural production was believed to be 180,000 metric tons with annual demand estimated at 136,000 tons. The world wide production and consumption of furfural for 1965, 1973 and 1978 are listed in table 2.1. Also as shown for those years of 1974 and 1975, domestic supply of furfural are more than ample and although world consumption has slipped between 8 and 10% from 1975 levels, new capacity continue to be announced or brought on stream to create a supply – demand inbalance. The January 1980 selling price was 1.14/kg (kirk othmer, 1980)

The world wide furfural supply / demand, 1000 metric tons.

2.2 COMMERCIAL PRODUCTION FURFURAL

Furfural is produced in batch or continues digester where the pentosan are hydrolysed to pentose and the pentoses subsequently cyclodehydrated to furfural.

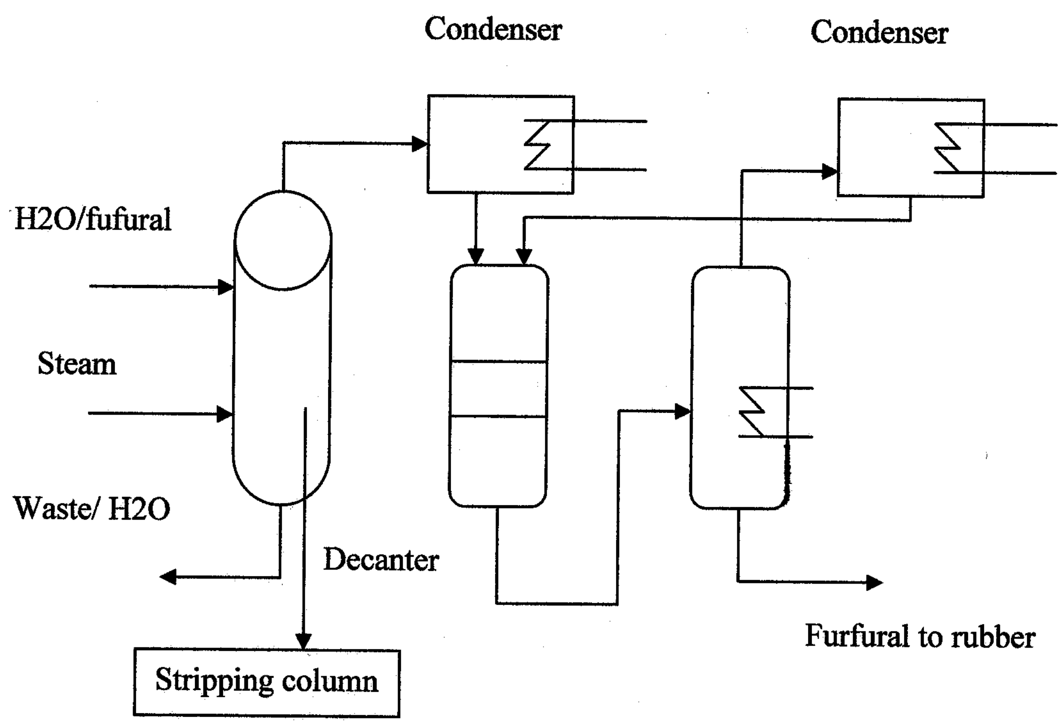


In this process, raw material is charged to the digester and treated with strong acid (inorganic), high pressure steam is introduced through the mass and rafter attaining operating temperature and pressure, furfural is steam distilled and furfual – water is fed to a stripping column to obtain pure furfural as the figure above. (MAXWELL OBOR, 1979)

2.2.1 SOURCES OF FURFURAL TO NIGERIA

All the while the source of furfural to Nigeria is by importation which is either from Venezuela or Arabian countries. Due to the economic importance of furfural, it is therefore necessary to produce furfural locally developing nation like Nigeria which will reduce dependence on importation that depend on certain policies. It will also reduce the cost of the commodity which will contribute positively to Nigeria economy.

Fig 2.2 DIAGRAM OF FURFURAL RECOVERY FROM AQUEOUS SOLUTIONS



2.2.2 PROCESS INVOLVED IN FURFURAL PRODUCTION

Many plant materials contain polysaccharides hemicellulose, a polymer of sugar containing five carbon atoms each. During commercial production, the furfural is produced by a single stage acid digestion system. The raw material which is dried grinded, rice husks is discharged into the digester (rotary digester) which when heated with dilute mineral acids

HCl This yield pentose (hydrolysis of hemicellulose to produce xylose). And followed by steam distillation process to remove the mixture of furfural and water.

So under the same condition of heat and acid, furfural is separated from water by evapouration water in this case is dehydrated (removed) by evaporation by losing its three molecules. In this case, furfural is produced.

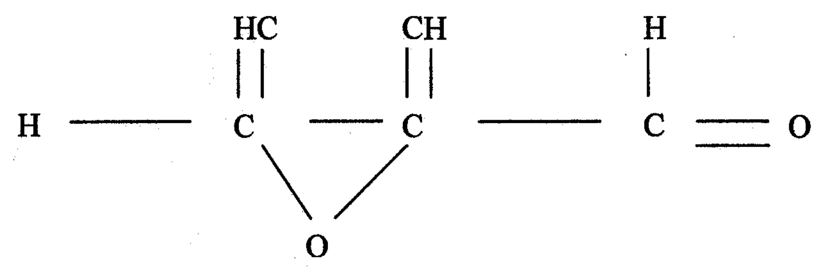
The reaction is shown below:

$$(C5H7O4)n \xrightarrow{nH2OH+} [HOCH2(CHOH)3CHO]n$$

The vapour leaving the digester is condensed and fed to the stripping column, the overhead which is rice furfural is condensed and cooled, it is further fed to a dehydrating column to remove the water that might still be in it. At the dehydrating column water is taken to overhead and the furfural is drained off from the base.

The acid digestion is the breaking down of cellulose material by action of heat and dilute acids (HCL,H2SO4) on the substance. The heat supply is just to fasten the rate of digestion. The acid converts the grinded rice husks to pentosan which is then converted to pentosen and the pentose formed contain furfural. The furfural is removed by a steam distillation through a continuous process.

The molecular structure of furfural is shown below:



(2 – furancarboxylaldehyde)

2.2.3 PROPERTIES OF FURFURAL

Furfural as a compound has variety of physical and chemical properties which makes it an important member of the heterocyclic compound called furans.

2.2.4 PHYSICAL PROPERTIES OF FURFURAL

From previous research , Furfural has a boiling point of 161.7oC and a melting point of -36.5oC. The refractive index at 20oC and 25oC are 1.526 and 1.5235 respectively. Also, its density decreases with increase in temperature. More so, at temperature of 20oC and 25oC, the density are 1.1598 and 1.1545g/mol respectively. Furfural is colourless when freshly distilled and turns yellowish aromatic odour reminiscent of almonds when freshly distilled. Ti dissolves readily in most polar organic solvents, but is only slightly soluble in either water or alkanes. Furfural (http://www.orgsyn ,/org syn/prep,asp=cv/po280.

2.2.5 CHEMICAL PROPERTIES

The chemical properties of furfural is that furfural participates in the same kind of reactions as other aldehydes and other aromatic compounds. Furfural is thermally stable in the absence of oxygen. When heated above 250oC by a researcher Jerry March ,1992s furfural decomposes explosively. At temperatures as high as 230oC exposure for many hours is required to produce detectable changes in the physical properties of furfural with the exception of colour. And when heated in the presence of acids, furfurals irreversibly solidifies into a hard thermosetting resin.

Furfural participates in catalytic hydrogenation in the presence of copper chromate catalyst to form furfuryl alcohol.

$$C4H3O.CHO \xrightarrow[Cu2cr]{} C4H3O\text{-}CH2OH$$

It demerizes to furoin in the presence of sodium cyanide catalyst.

$$C4H3OCHO + C4H3O \xrightarrow{NACN} C4H3O\text{-}CHOH\text{-}CO\text{-}C4H3O$$

Furfural undergoes oxidation reaction which forms furoic acid when reduced to furfurylalcohol and converted to furan by decarboxylation over a suitable catalyst.

Finally, undergoes a reaction with sodium hydroxide to form a carnnizaro reaction yielding furfurylalcohol and sodium furoate.

$$2C4H3.CHO + NAOH \longrightarrow C4H3O\text{-}CHO + C4H3OCOONa$$

2.3 THE RAW MATERIAL (RICE HUSKS)

Rice is the most important of the worlds cereals and forms the basis of diet of millions of people on south-eastern Asia, America, Europe and Africa. Infact it forms the stable food more than 50% of the worlds population and is one of the oldest grown crops. Rice is grown below sea level and it is about three (3) meters in height depending on the variety and environmental conditions under which it is grown. (Douglas, 1982). It requires rich loamy to clay soil retentive of water. Rice is an annual crop which when planted is harvested after four months. After harvesting when dried using sickles or combine harvester, the grains after removing them are dried in silos or bins. Rice in a well conditioned room loses moisture at the rate of 5% every 24 hours. Then after rice treatment , the rice is obtained for consumption and the rice husks are discarded for furfural production.

2.3.1 SAFETY FACTORS CONSIDERED IN FURFURAL ENVIRONMENT

Under normal circumstances, safety guide should be given to employee on the hazardous effect of furfural to their health. This was confirmed from the result of physical examination of workers on daily contact with furfural for as long as ten years, it will be observed that the skin of the employees get stained and darkens due to the furfural coming in contact with air.

When inhaled, furfural can cause symptoms similar to those of intoxication, including; Euphoria, headache, dizziness, nasua and eventual unconsciousness and death due to respirational failure.

So, contact with the skin irritates the skin and respiratory tract and can cause the lungs to fill with fluid.

Chronic skin exposure can lead to skin allergy to the substance, as well as unusual susceptibility to sunburn. In toxicity studies furfural has lead to tumars, mutations, and liver and kidney damage in animals. With all these, safety rules are obeyed by using the necessary rubber hand gloves.

2.3.2 STORAGE

Storage can simply be defined as the process where a commodity is kept over a period of time before used and still retains its quality. Storage is then found necessary in areas of storing furfural and rice husks.

2.3.3 STORAGE OF FURFURAL AND RICE HUSKS

Furfural is a white (colourless) liquid aldehyde when freshly distilled but darkens in contact with air (oxygen). An industrial furfural which is light yellowish turns brown in colour when it comes in contact with oxygen (air). Therefore, furfural is stored in an iron or steal equipment which is oxygen free. When stored in contact with air, there is gradual increase in acidity and polymer formation. The best way of storing furfural is to store it in an oxygen free atmosphere which will prevent the formation polymer acidity and darkening of its colour. While rice husks can be stored in an oxygen and moisture free environment that is made of steel or aluminum.

2.3.4 USES OF FURFURAL

Furfural as a compound has variety of applications which includes the following:

1.) Furfural as a selective solvent in the refining of oil.

Furfural is used as a selective solvent for separating saturated from unsaturated compounds in petroleum refining, gas, oil and diesel fuel. It is used in the refining of lubricating oil to increase the stability under operating conditions and to improve the viscosity temperature relationship for mineral oils ranging from diesel to lubricating oils. Furfural is used as a solvent in petrochemical refining to extract dienes (which is used to make a synthetic rubber) from other hydrocarbons.

Furfural, as well as its derivative furfurylalcohol can be used either themselves or in together with phenol, acetone, or urea to make solid resins. Such resins are used in making fiber glass, some aircraft components and automobile brakes. This production is due to corrosion resistance, high carbon yield, stability at elevated temperature, low fire hazard and excellent physical strength of thermosetting resins produced.

2.) Furfural in rotating disk contactor

In extraction process using furfural it has been developed for solvent extraction process. It has a contactor which consists of vertical column divided into a number of compartments by a series of stator rings. The rotating disk is supported by a staff centered in each compartment. The shaft is driven by an electric motor installed on the top of the column. Feeds are at the top and bottom and setting space is provided at both ends. Setting compartments in the top and bottom of the RDC are separated from the turbulent sections by calming grids. The rotating disc contactors are used I propane desulpalting unit and furfural extraction units. PDU (propane deashalting unit) liquid bottom and flows upward while higher density feed (short residue) is fed to the top of the contactor. The rotating disc aids the mixing and dispersion of the solvent (furfural) which is of higher density is fed to the upper section while the feed is introduced from the bottom.

3.) Furfural in cold Blending

Furfural is use as a reactive solvent in cold blending of pitch, forms a resinous impregnants that interact a carboxylization temperatures. These resin pitch combination have curing and carboxylization characteristics better than those of either component alone.

The cold blending process significantly improves the environment acceptance of pitch and offers industries an alternative to hot processing.

4.) Resinification of Furfural

The resinification of furfural initiated by acids or heat has been observed for years and many interpretations of the polymerization mechanism have been proposed. Because of the complexity of this polymerization key intermediate have not been isolated condition for polymerization either aqueous or anhydrous, inert or oxygen all affects the composition of the polymer. Recent work on resinification of furfural has introduced a new insight on the polymerization mechanism, particularly with respect to thermal reaction at 100oC – 250oC in the absence of air. Based on the isolation and characterization in the figures below, was proposed for the final resins.

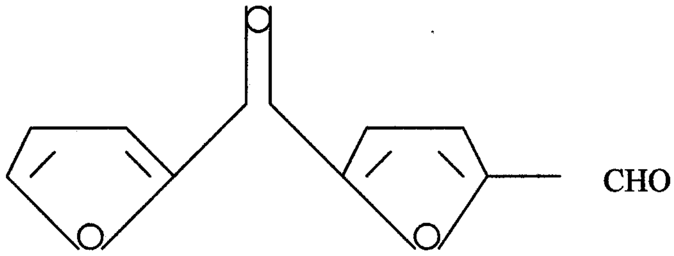


FIG. A

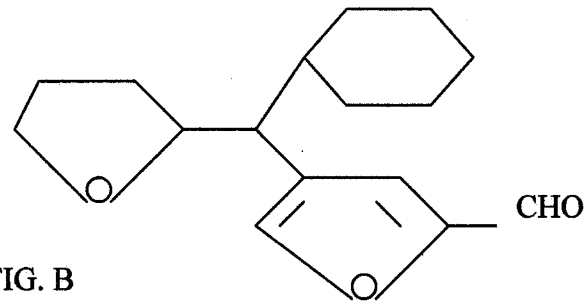


FIG. B

2.3.5 DISTILLATION

The separation of liquid mixtures into several components is one of the major operations in the chemical and petroleum industries, and distillation, the most widely used method of achieving this end is the key operation of the oil refinery throughout the chemist industry the demand for purer products, coupled with a relentless pursuit of greater efficiency has necessitated continued research into the techniques of distillation. For example; the separation of benzene from a mixture with toluene requires only a single unit as indicated in the figure below:

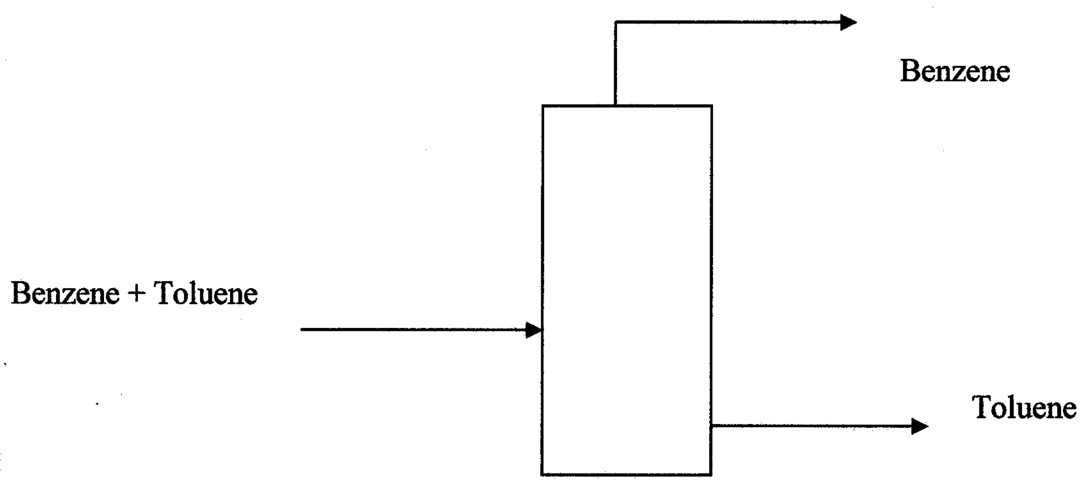


Fig 2.3 Seperation of Binary Mixtures (Distillation Column)

(Richardson and Coulson, 1991)

2.4 BATCH DISTILLATION

In batch distillation, the more volatile component is evaporated from the still which therefore becomes progressively richer in the less volatile constituent. Distillation is contained either until the residue of the still contains a material with acceptability low content of the volatile material, or until the distillation is no longer sufficient pure respect of the volatile content.

2.4.1 EXTRACTIVE DISTILLATION

Extractive distillation is a method of rectification similar in purpose to Azeotropic distillation. To binary mixture which is difficult or impossible to separate by ordinary means, a third component, termed solvent is added which alters the volatility of the original constituents, thus permitting the separation. The added solvent is lower, a low volatility and itself not appreciably vaporized in the fractionator. Example, the extractive separation (b.p=384K) from paraffin hydrocarbon of approximately the same molecular weight. This is either very difficult or impossible owing to low relative volatility or azeotropic formation, such a separation is necessary in the recovery mixture. Using Iso-octane (b.p 372.5K as an example of paraffin hydrocarbon). In this case Iso-octane is more volatile since phenol is being added to it, it relative volatility increases so that with as much as 83mol percent phenol in the liquid, the separation of toluene is easy.

(RICHARDSON AND COULSON, 1991)

2.4.2 DIFFERENTIAL DISTILLATION

The simplest example of batch distillation is a single stage differential distillation, starting with a still pot, initial full, heated at a constant rate. In this process the vapour formed on boiling the liquid is removed at once from the system. Since the vapour is richer in the more volatile component than the liquid, it follows that the liquid remaining becomes steadily weaker in this component, with the result that the composition of the product progressively alter. Thus, while the vapour formed over a short period is in equilibrium with the total vapour formed is not in equilibrium with the residual liquid. At the end of the process the liquid which has not been vapourized is removed as the bottom product.

2.4.3 FLASH OR EQUILIBRUM DISTILLATION

This method, frequently carried out as a continous process consist of vapourize fraction of the liquid feed in such a way that vapour evolved is usually jumped through a fired heater and enters the still through a valve where the pressure is reduced. The still is essentially a separator in which the liquid and vapour produced by the reduction in pressure have sufficient time to reach equilibrium. The vapour is removed from the top of the separator and is then usually condensed while the liquid leaves from the bottom.

In a typical pipestill used in the petroleum industry where for instance, a crude oil might enter at 440K and 400KN/M2 and leave at 520K about 900KN/M2, some 15 percent maybe vapourized in the process. The vapour and liquid streams may contain many components in such an application.

2.4.4 RECTIFICATION

In the two, process outlined previously the vapour leaving the still at any time is in equilibrium with the liquid remaining and there will normally be only a small increase in concentration of the more volatile component. The essential merit of rectification is that it enables a vapour to be obtained that is substantially richer in the more volatile component than the liquid left in the still. This is achieved by an arrangement known as a fractionating column which enables successive vapourization and condensation to be accomplished in one unit.

2.4.5 STEAM DISTILLATION

Where the material to be distilled has a high boiling point and particularly where decomposition might occur if direct distillation were employed, the process of steam distillation can be used. Steam is passed directly into the liquid in the still, the solubility of the steam in the liquid must be very low. Steam distillation is perhaps the most common example of differential distillation. Two cases are possible, the steam may be superheated and provided sufficient heat to vapourize the material concerned without itself condensing. Alternatively, some of the steam will condense producing liquid water phase. (COULSON AND RICHARDSON, 1991)

CHAPTER THREE

3.0 METHODOLOGY

The following instrument, material and reagents were used for furfural production and characterization using rice husk

Material

Rice husk is obtained from rice mill in Minna

Table 3.0

Equipment	Reagent
Steam generating kit	Hydrochloric acid
Clifton hot plate	Aniline
Staurt scientific hot plate	Distilled water
Flat bottom flask	Glacial acetate (acetic acid)
Condenser	Phloroglucinal crystal
Thermometer	
Beaker	
Sieves (300 micrometer 250, 500 micrometer	
Measuring cylinder (50ml)	
Stopper	
Retort stand	

3.1 Experimental procedures

3.1.0 PRETREATMENT OF RICE HUSK

After discarding rice husk as a waste, it was collected and sun dried for a period of ten days. Then, the rice husks were further dried in an oven for a long period of three days . After that, the dried rice husks were taken for grinding

3.1.1 SIEVE ANALYSIS

The grinded rice husk was introduced in a set of standard sieves during the process with different particle sizes of 250, 300 and 500micrometer from the top and the sieves were agitated properly for like about 20 minutes after which the rice husk on each sieves were collected and weighed using weighing balance.And then discharged into bottom flask for hydrolysis.

3.1.3 ACID DILUTION PROCESS

The volume of the acid used in each experiment for different concentrations is 30ml and the dilution was done by adding:

- 6ML of concentrated hydrochloric acid to 24ml distilled water at 20% concentration.
- 9ML of concentrated hydrochloric acid to 21ml of distilled water at 30% concentration.
- Then, 12ml of concentrated hydrochloric acid to 18ml of distilled water at 40% concentration.

3.2 STEAM DISTILLATION PROCESS

A steam generating kit was filled with water to a certain level and heat source such as STUART SCIENTIFIC HOT PLATE was placed under the kit, which was well connected to an electric current source. A particular weight of the particle size rice husk, 5grams was discharged into the flat bottom flask and 30ml of diluted hydrochloric acid at different percentage concentration was added to the rice husk in the flat bottom of the flask. Heat was gently applied to the bottom of the flask by the help of another electrical hot plate called CLIFFON HOT PLATE. Meanwhile, the condenser had already been clamped to a retort stand. So, as steam started coming out from the kit, and the mixture in the flat bottom flask started boiling, the steam from the kit was then collected in to the resulting mixture in the flask by the help of a delivery tube. Then, the clock was set for timing.

And, after the coming out of steam and the mixture boiling, the vapour from the flask passes through the condenser which then condenses the vapour which is the mixture of furfural and water. The distillate was then collected at the outlet of the condenser. Finally, the cooling medium in the condenser was water which was in counter current flow with the vapour.

3.3 EVAPOURATION

After distillation process, the distillate which contains the solution of furfural(with water) was further heated to evaporate three molecules of water. And this was done because furfural has a higher boiling point compare to that of water. Therefore, concentrating the furfural solution is an important process of obtaining the desired product called furfural. This was done from the literature that the boiling point of furfural is 161.7oC and that of water is 100oC, so water tends to evaporate before the desired product.

Further more, the process was carried out for a very long time at a regulated temperature of 100oC – 110oC. Also, after concentrating the mixture, the density of the residue furfural was determined by weighing accurately an empty 50ml measuring cylinder and weighed accurately also the cylinder filled with furfural and since density of a substance is the mass divided by the volume it occupies. The difference in weight of the filled measuring cylinder and empty measuring cylinder which is the mass of the furfural and the volume of 50ml cylinder was used to divide it.

3.4 CHARACTERIZATION

2ml of aniline was added to 2ml of glacial acetate (acetic acid) in 50ml measuring cylinder. Then the resulting solution was then added to a freshly prepared furfural. Also, the heating of 0.5g of phloroglucinal crystal with 2ml concentrated hydrochloric acid to 2ml of freshly prepared furfural as well.

CHAPTER FOUR

4.0 RESULTS AND DISCUSSION OF RESULTS

TABLE 4.1, % YIELD OF FURFURAL

S/NO	Time(min)	% Conc.	Particle size(mm)	Mass of conc. Furfural(g)	% yield
1	15.00	20.00	0.30	0.65	13.00
2	15.00	30.00	0.30	0.83	16.66
3	15.00	40.00	0.30	0.72	14.40
4	30.00	20.00	0.30	0.86	17.20
5	30.00	30.00	0.30	1.08	21.60
6	30.00	40.00	0.30	0.97	19.40
7	45.00	20.00	0.30	1.13	22.60
8	45.00	30.00	0.30	1.47	29.40
9	45.00	40.00	0.30	1.00	20.00
10	60.00	20.00	0.30	1.33	26.60
11	60.00	30.00	0.30	1.51	30.20
12	60.00	40.00	0.30	1.42	28.40

Mass of Rice Husk (grams) = 5.00

Volume of Acid (ml) = 30.00

TABLE 4.2 % CONCENTRATION OF ACID

For 15 minutes and 30 minutes

Time (minutes)	% yield	% concentration	Mass of Rice Husk
15.00	13.00	20.00	5.00
15.00	16.66	30.00	5.00
15.00	14.40	40.00	5.00
30.00	17.20	20.00	5.00
30.00	21.60	30.00	5.00
30.00	19.40	40.00	5.00

For 45 minutes and 60 mintues

Time(min)	% yield	% concentration	Mass of Rice Husk
45.00	22.60	20.00	5.00
45.00	29.40	30.00	5.00
45.00	20.00	40.00	5.00
60.00	26.60	20.00	5.00
60.00	30.20	30.00	5.00
60.00	28.40	40.00	5.00

4.1 DISCUSSION OF RESULTS

From my results on table 4.1 it is obvious that as the time increases, so also the percentage (%) yield increase too. It implies that the % yield of furfural is directly proportional to the time provided that the particle size and mass of the furfural are kept constant.

For fifteen minutes experiments, 13.00, 16.66 and 14.40% yield were gotten from 5g of rice husk using 20, 30 and 40% concentration of diluted acid. And looking at the table of results, it is seen that 30% concentration gave the highest percentage yield of furfural.

Also, for 30 minutes experiments, 17.20, 21.60 and 19.40% yield were gotten from the same 5g of rice husk using the same percentage concentration such as 20, 30 and 40%. Then, it signifies that for 30 minutes, it gave the highest percentage yields of furfural.

Moreso, for 45 minutes experiments, 22.60, 29.40 and 20.00% yield were gotten. Using the same particle size 300micrometer, the 30% concentration of acid gave me the highest % yield of furfural.

For 60 minutes, 26.60, 30.20 and 28.40% were finally obtained as the % yield of the furfural which in the same way showed that as time increases so also the percentage yield increases as well. In addition, the graphs of % yield of furfural against time were plotted. It showed that the graphs were straight line graphs which implies that as the time increases from 15 minutes to 60 minutes so also the percentage yield increased from 13.00 to 30% yield.

And it is done for different plots with different time. The first graph, graph A, shows that as the time increases from 15 minutes to 30 minutes the percentage yield increases too. The straight lines do not meet each other.

Also, as the time increases from 45 to 60 minutes in graph B, the percentage yield of furfural increases too. But in this case, the straight lines tend to cross each other at different points.

Finally, it can also be seen that the test carried out confirmed the presence of furfural.

CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

From this work, it can be seen that the importance of furfural cannot be over emphasized. In essence, furfural can be produced locally in Nigeria and thereby reducing huge foreign exchange in terms of importation. Also, it is quite interesting that rice husks are not waste but they can serve as a raw material for other products.

And finally, the test carried out confirmed that the distillate is furfural and the time, 60 (sixty) minutes gave the highest percentage yield of 30.20%.

5.2 RECOMMENDATION

1. Temperature of the experiment should be properly and well regulated.
2. Air tight evaporation should be employed using stopper.
3. Pressurized equipment should be used.
4. Dried steam should be used for the process distillation.
5. Forty five (45) and sixty minutes experiment should be recommended for the experiment in batchs.
6. Researches should be based on other raw materials like cotton seeds, corn cobs, wheat bran and saw dust.

REFERENCE

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APPENDIX

Yield = output / input × 100%

= 0.65 / 5 × 100 = 13%

= 0.83 / 5 × 100 = 16.66%

= 0.72 / 5 × 100 = 14.40%

= 0.86 / 5 × 100 = 17.20%

= 1.08 / 5 × 100 = 21.60%

= 0.97 / 5 × 100 = 19.40%

= 1.13 / 5 × 100 = 22.60%

= 1.47 / 5 × 100 = 29.40%

= 1.00 / 5 × 100 = 20.00%

= 1.33 / 5 × 100 = 26.60%

= 30.20 / 5 × 100 = 30.20%

= 1.42 / 5 × 100 = 28.40%

DETERMINATION OF DENSITY

Density = Mass / Volume

= Mass of Produced Furfural (g) / Volume of Furfural (ml)

At room temperature of 25°C, Mass of rice husks used = 5g

Volume of acid used = 30ml

Mass of Furfural produced = 4.086g

Volume of Furfural = 4ml

Therefore the Density = Mass of Furfural produced / Volume of Furfural

= 4.086 (g) / 4ml = 1.0215 g/ml

Density = 1.022 (g/ml)

Table 2.1 Worldwide Furfural Supply/Demand, 1000 metric tons

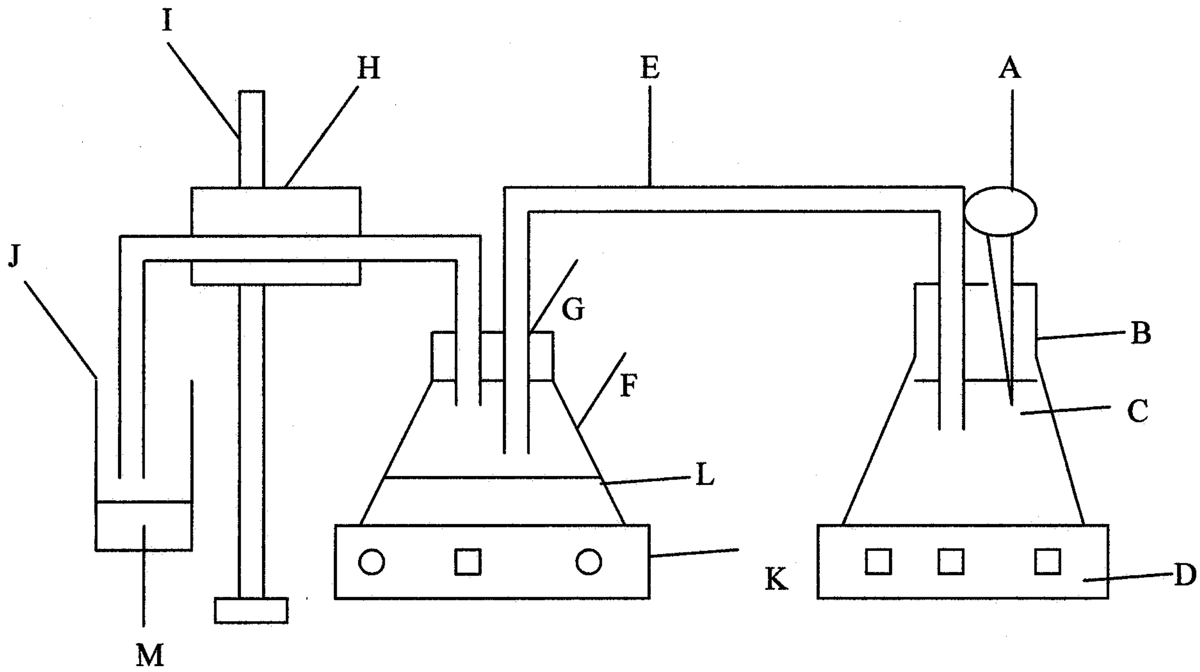
	1965	1973	1978
Capacity	100	141	204
Production	91	136	159
Consumption	91	136	136
U.S bulk Price $/kg	27.6	41.3	114

Includes 27,700 t estimates for eastern Europe.

Table 3.2 Data for Sieve size and Weight Retained

S/N	**Sieve size micrometer**	**Weight (g) retained**
1	250	34.19
2	300	33.16
3	500	32,13

DIAGRAM



Key

A. Thermometer

B. Steam generating kit

C. Water

D. Staurt scientific hot plate

E. Delivery tube

F. Flat bottom flask

G. Stopper

H. Condenser

I. Retort stand

J. Conical flask

K. Clifton hot plate

L. Mixture of rice husk and diluted acid

M. Mixture of furfural and water

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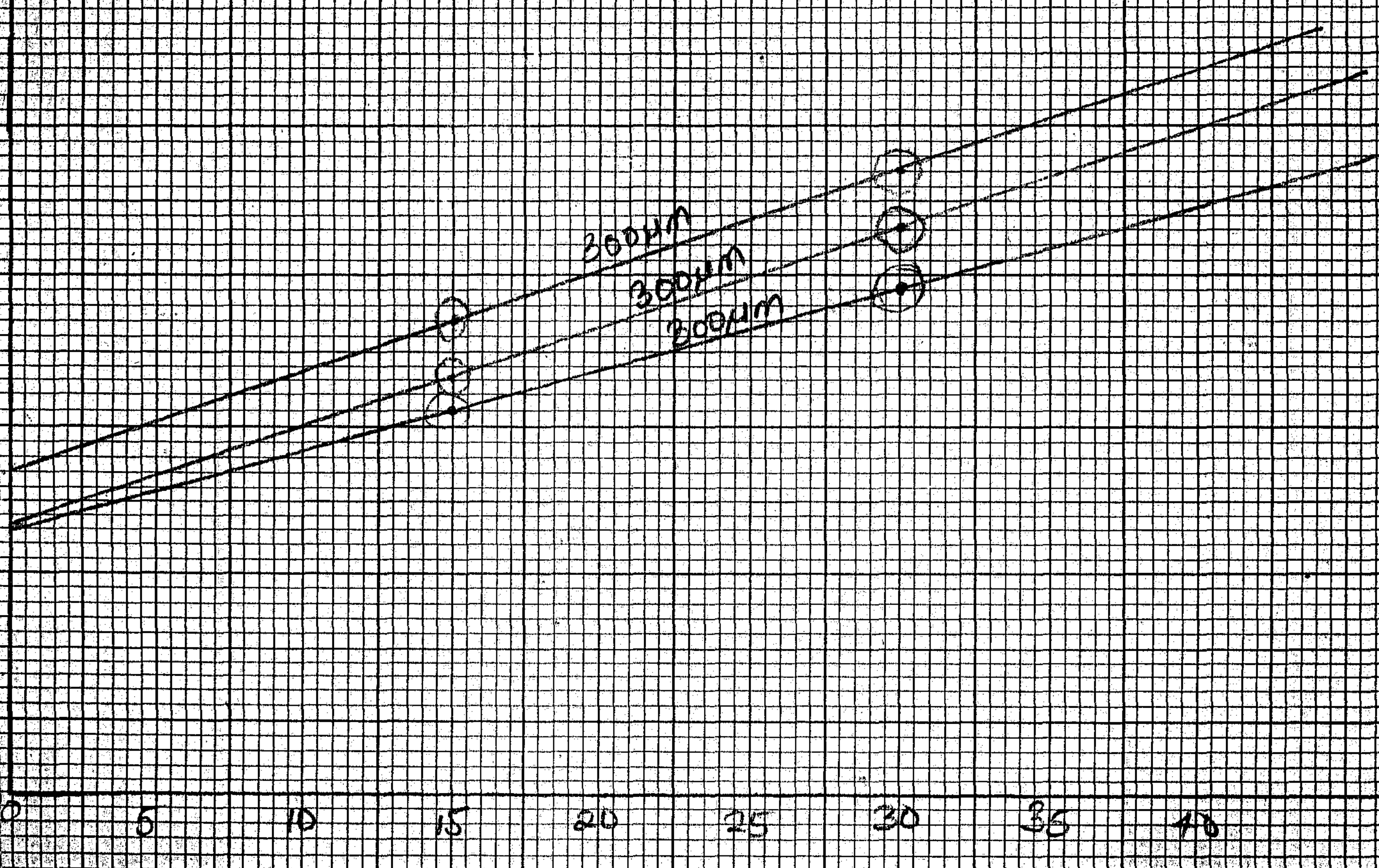
Name: GRAPH A Dept.: Reg. No:

TITLE: A GRAPH OF % YIELD AGAINST TIME

SCALE: 2cm REP 5% ON Y-AXIS, 2cm REP 5MIN ON X-AXIS



TIME (MINUTE

FOR 15MINUTES AND 30MINUTES

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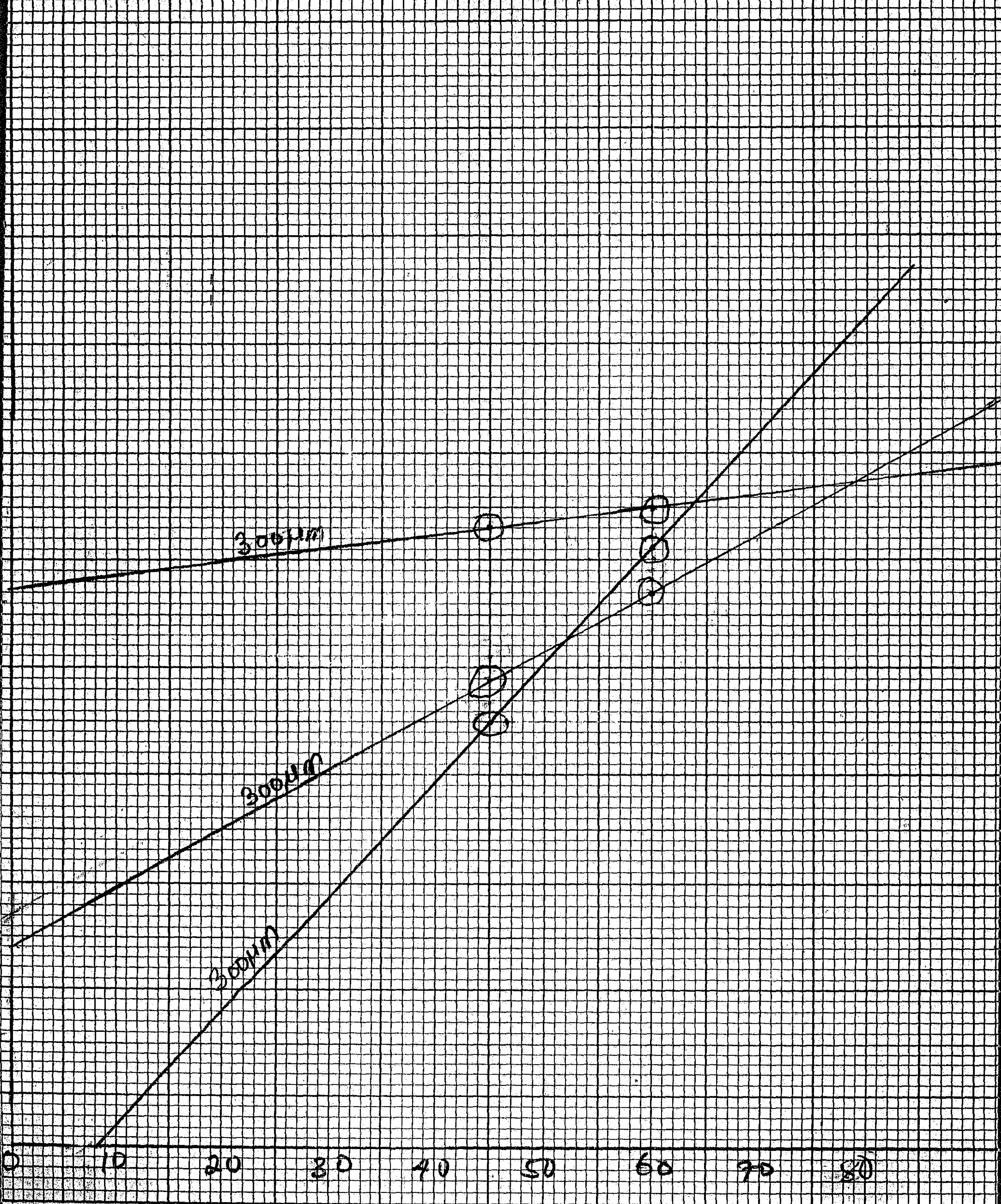
Name: GRAPH B Dept.: Reg. No:

TITLE: A GRAPH OF % YIELD AGAINST TIME (MINU

SCALE: 2cm rep 10 minutes ON X-AXIS, 2cm REP 5% ON Y-AXIS



FOR 45 AND 60 MINUTES