

**A COMPARATIVE STUDY OF ACID – ALKALINE
HYDROLYSIS OF SAWDUST**

BY

HARUNA MOHAMMED

98/7061EH

**DEPARTMENT OF CHEMICAL ENGINEERING
FEDERAL UNIVERSITY OF TECHNOLOGY
MINNA**

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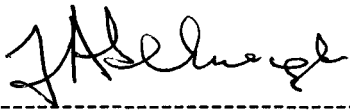
**DEPARTMENT OF CHEMICAL ENGINEERING
SCHOOL OF ENGINEERING AND ENGINEERING TECHNOLOGY,
FEDERAL UNIVERSITY OF TECHNOLOGY, MINNA,
NIGER STATE, NIGERIA**

**A PROJECT SUBMITTED IN PARTIAL FULFILLMENT OF
THE REQUIREMENT FOR THE AWARD OF DEGREE OF
BACHELOR OF ENGINEERING (B. ENG)**

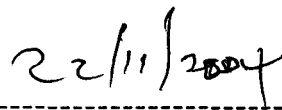
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CERTIFICATION

I hereby certify that I have supervised, read and approved this project carried out by MR. HARUNA MOHAMMED of the Department of Chemical Engineering in the school of Engineering and Engineering Technology, Federal University of Technology, Minna and have found it adequate in scope and quality for the partial fulfillment of the award of the Bachelor Degree in Chemical Engineering.



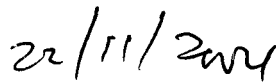
Dr. F. Aberuagba
(Project Supervisor)



Date



Dr. F. Aberuagba
(Head of Department)



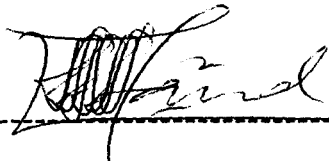
Date

(External Examiner)

Date

DECLARATION

I, MOHAMMED HARUNA, with registration number 98/7061EH of the Department of Chemical Engineering, School of engineering and Engineering Technology, Federal University of Technology Minna, declare that this write up is my work and is submitted in partial fulfillment of the award of Bachelor of Engineering (B. Eng) of the Federal University of Technology, Minna. Every detail as well as information obtained from published and unpublished work has been duly acknowledged.



Signature

22ND NOV, 2004

Date

DEDICATION

I dedicated this project work to Almighty Allah, the source of my inspiration from birth who has guide and protect me through every moment of my life and all those who have given me support in one form or the other especially my parents.

ACKNOWLEDGEMENT

My profound gratitude goes to almighty Allah for his divine inspiration, affection, guidance and protection over my entire life. I will like to extend my appreciation to my caring supervisor in person of Dr. F. Aberuagba for encouraging and supporting me through the rigorous of this project, may God bless you and your family abundantly.

I will like to express my heartfelt gratitude to my Dearing parents: Mallam Haruna and Mallama Salamatu Mamud for their various forms of support rendered to me; may Allah bestow his mercy on you all. Also, I will like to acknowledge the formidable support of my family members, Yunusa, Ibrahim, Fatimatu, Basiratu and Aishatu; I love you all, thanks for being there for me. I'm also grateful to uncle Ishaq Mamud and aunty Hadiza Ahamad for their advices and support, may Allah reward you all, I say thanks a million.

Thanks to the Head of Department Dr. F. Aberruagba, and all the lecturers in Chemical Engineering Department. Thanks to all the staffs in Biochemistry Department especially Mallam Dauda Ibrahim. I will also like to say thanks to all my friends: M. Abdullahi, Mustapha S. I., Awwal M., Abubakar M., Farouq U., Yusuf A. and Yusuf D. I am also grateful to Mallam Dauda Ogbeha and Tijani Abdulkadir for their support, and encouragement, and all those I cannot seem to think of at present I love you all.

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ABSTRACT

The comparative study of the kinetics of acid and alkaline hydrolysis of sawdust (Mahogany – khaya – 1 vorensis) has been carried out in a 250ml beaker, which serve as batch reactor. The effect of temperature and concentration on hydrolysis reaction were investigated for both media. The results obtained shows that optimum temperature for glucose production was 80^oc in the media, while 0.4M NaOH and 2.5M Sulphuric acid are the optimum concentration for alkaline and acid medium respectively with the later given a better yield. The hydrolysis reaction of sawdust in both medium is described by the first order kinetics, with activation energy of 39.65KJ/gmol and 43.42KJ/gmol for acid and alkaline catalyzed reaction respectively. This indicates higher energy barrier in alkaline hydrolysis of the sample.

CHAPTER ONE

INTRODUCTION

1.0

Underneath the bark of a tree is a hard fibrous substance that makes up the trunks and large branches called wood.

Wood has been of enormous importance to human existence and it enhanced the advancement of civilization. The primitive man not only used it in the construction of his shelter, but was able even with his crude stone implement to fashion out canoes, farm implements and utensils of different kind.

With the advance of civilization, wood became the basic raw material in building houses furniture, vehicle, Wagons etc. For the manufacturing and processing industries, wood has been used extensively in the production of paper, synthetic fibre etc. Some hard wood are treated chemically or heated under controlled conditions to yield a number of chemicals, wood alcohol acetic acid and charcoal among others.

Similarly, wood also forms the basis for lots of labour intensive industries like sawmills, Plymills etc. In some technological advanced countries, wood cellulose and lignin are used in cellophane celluloid, pentosan and alcohol production.

A vital significant of wood was in the creation of jobs or employment opportunities for the general populace, because in the already mentioned use of woods, both skilled and unskilled labour are required to process it to the desired product.

However, wood processing produces a large quantity of wood waste which also have high economic value if well harnessed. Particularly, the use of wood waste such as saw dust, as raw material for processing industries will help to reduce the problem of unemployment, produce food for both adult and infants, and reduces the crises of pollution by processing in to products like glucose, alcohols, petrochemicals, biochemical etc. The abundance of cellulose on the earth, especially in most dry wood waste

(sawdust) makes it imperative to process such waste in to valuable product rather than burning them in sheer ignorance.

Glucose, a monomer of cellulose and other carbohydrate polymers, is an essential product due to the fact that it is a great source of instant energy in human body system. It is commonly known as grape sugar or dextrose, and present in fruits such as grapes, honey, and in the sap of plants etc.

Furthermore, glucose is used commonly as food sweetener and preservatives in living organism. However, due to economic reason (poverty, poor living standard), a lot of individuals do not have access to glucose in its pure form because it is expensive, despite the fact that it is needed by the body for immediate energy supplement.

Therefore, it becomes highly necessary to increase the production of glucose from readily available and cheap raw material such as saw dust in order to reduce cost and increase accessibility.

1.1 SCOPE OF WORK

The scope of this project limits on the hydrolysis of sawdust by comparing the kinetics of acid and alkaline hydrolysis: these include; reaction order, effect of concentration, effect of temperature and glucose yield of each method from saw dust.

1.2 AIMS AND OBJECTIVES

1. To determine the effect of temperature, acid, and alkaline concentration on the hydrolysis of saw dust.
2. To determine the rate expression for the hydrolysis reactions
3. To determine the activation energies for the hydrolysis reaction in acid and alkaline medium.
4. To establish the best medium for the hydrolysis reaction.

CHAPTER TWO

LITERATURE REVIEW

2.0

2.1 PHYSIOLOGY OF A TREE

Plants germinate, grow, develop, mature and die. Plant physiology is the study of the processes of how and why each plant behaves in its own peculiar way. It is the study of the organization and operation of the processes in the plant that order its development and behaviour.

Botanically, trees differ from other plants only in degree, not in kind but as a result of their large size, slow development and long maturity, they have reached a high degree of specialization in certain directions.

Trees are divided into two classes:

1. Soft wood (monocotyledons).
2. Hard wood (Dicotyledons).

Hard wood are deciduous woods while soft woods are conifers. Hard wood have broad leaves which they ordinarily lose annually. Most soft woods have either scale like leaves as in ciders or needle like leaves as in pines which most species retain all year. Hard wood and soft wood are misleading terms in that they have no direct reference to the hardness or softness of the wood. Such hard woods such as cotton wood have softer wood than white pines or true firs, which are both classed as soft woods. In general however, hard wood are harder than soft wood.

2.2 WOOD FORMATION

The dicotyledonous plant growth are classified in two namely: Primary and Secondary growth.

The Primary growth essentially permits the extension in length, and any increase in the thickness of stems and root results mainly from an enlargement of cells in lateral direction.

In many trachophyte primary growth is the only means of increase body size. However, not only in length but also in thickness through lateral increase of cells number.

These plants under go processes of secondary growth. Apart from comparatively enormous increase in stem and root, the large scale result of secondary growth is the development of bark and of wood. Wood tends to be formed in relatively large quantities and new layers are added each year to those already accumulated. Plant of this type develop as trees and become recognizably woody in appearance.

2.2.1 WOOD COMPOSITION

Wood is a mixture of three natural polymer; hemi cellulose, and lignin in an approximate abundance of 50:25:25% respectively.

Wood also contains extractives (tannis, starch, colouring matter, oil resins, fats, waxes) and ash forming minerals. The extractives are not part of the wood structure but contribute to the wood, such properties of colour, odour, taste and resistance to decay (Irwin P. T, 1982).

2.3 BIODEGRADATION

Saccharification is the degradation of polysaccharide like cellulose to sugars. Therefore, biodegradation of wood is the degradation of polysaccharides of sugar using microorganism. The process is of such economic importance that it has been intensively studied so it is surprising to note that few of the major organism have been seriously studied with a view to their productive use under normal condition, the main agents of wood decay are fungi, bacteria, only being significant in water logged timber.

Wood rotting fungi are traditionally split to five different groups according to their mode of action and effect on the wood structure (Norkrans, 1967; Duraseketal, 1967 and Liesa 1979).

Blue stain fungi and associates and fungi imperfect. They cause little polymer break down or physical damage but do grow inside wood cells and cause discoloration.

The degrade polysaccharide generally cause only slight modification to lignin. The brown rot fungi are basi-diomycete which preferentially attack soft woods.

They have little effect on lignin but extensively degrade hemi-cellulose and cellulose. The third group is the white rot fungi, which are basi- diaomycete, which preferentially degrade lignin and hemicelluloses. This fungi shows preferences for hard wood over soft wood. The last group is the soft rot, which attack both hard and soft wood and often occur in situation of high moisture.

2.4 CELLULOSE

2.4.1 INTRODUCTION

Nature's building block, it is the principal structural material of all trees and plants and forms their cell walls. It is widely distributed in nature. It has been estimated that higher plant yearly as a result of photosynthesis, synthesizes nearly 10,000,000 tones of cellulose. The proportion of cellulose to total carbohydrate found in plant may vary in various types of woods from 30 to 40 percent and to more than 98 percentage in the seed hair of the cotton plant. Cotton fibre is cellulose purest and most familiar naturally occurring form. Cotton fibre is over 90% cellulose; wood, about 50 percent; straw, 30% cellulose occurs commonly in useful forms in such seed fibres as cotton and kapok; in such stem (blast) fibre as flax, jute and hemp; and in wood, bagasse, rice husks, and other various plant products.

Many useful products such as plastics or fabrics are derived from cellulose, but the manufacture of paper is the largest single commercial use of cellulose (Brian and Allan, 1995).

2.4.2 PREPARATION

During the preparation of cellulose, raw plant material is treated with hot alkali, this treatment removes most of the lignin, the hemicelluloses and mucilaginous components. For the production of glucose, plant material is treated with petroleum ether to remove the tough coating substances.

After been treated with hot alkali, the cellulose is then processed to produce paper and fibres. The high resistance of cellulose to chemical or enzymatic breakdown is important in the manufacture of paper and cloth. Pure cellulose can also be hydrolysed to form D-glucose, $C_6H_{12}O_6$, with a yield of about 95 percent.

2.4.3 CELLULOSE DERIVATIVES

Cellulose derivatives are the synthetic compounds formed by the reaction of naturally occurring cellulose with acids, alkalis, anhydrides, or alkyl halides.

The synthetic compounds derived from these reactions are in the form of esters and ethers.

The esters include: cellulose nitrate, cellulose acetate, cellulose propionate, cellulose acetate butyrate, and cellulose acetate phthalate. The ethers on the other hand are mainly composed of methyl cellulose, hydroxymethyl cellulose, ethylhydroxyethyl cellulose, carboxymethyl hydroxyl cellulose. The note worthy biological stability of cellulose, is dramatically illustrated by trees; the life span of which may be several thousand years.

Enzymes capable of breaking down cellulose are generally found among only several species of bacteria and molds. The apparent ability of termites to utilize cellulose as an energy source depends on the presence in their intestinal tracks of protozoans that can break it down.

Similarly, the single celled organisms living in the rumina of sheep and cattle are responsible for their ability to utilize the cellulose present in typical grasses and other feeds.

2.4.4 CHEMICAL STRUCTURE

Though cellulose and its derivation has found a wide commercialization in their use, it was not until 1934 that W.N. Haworth suggested its currently accepted chemical structure (figure1). It had been known since 1913 that its empirical formula, determined on a probably washed and dried specimen, was $C_6H_{10}O_5$ (carbon, 44.4 percent; hydrogen, 6.2 percent).

It was also known through work of Herman strandinger, and Kurt frendenberg that it was a longer chain polymer molecule with repeating glucosidic residues. Each unit has three free hydroxyl groups, one being primary ($-CH_2OH$) and two being secondary ($>CH. OH$). By 1920 Emilfisher had clarified the structure of the simple sugars, and in that same year, X-ray work on cellulose showed the first clear diffraction pattern of the fibres. Typical X-ray fibres are shown for cellulose, regenerated cellulose and cellulose acetate.

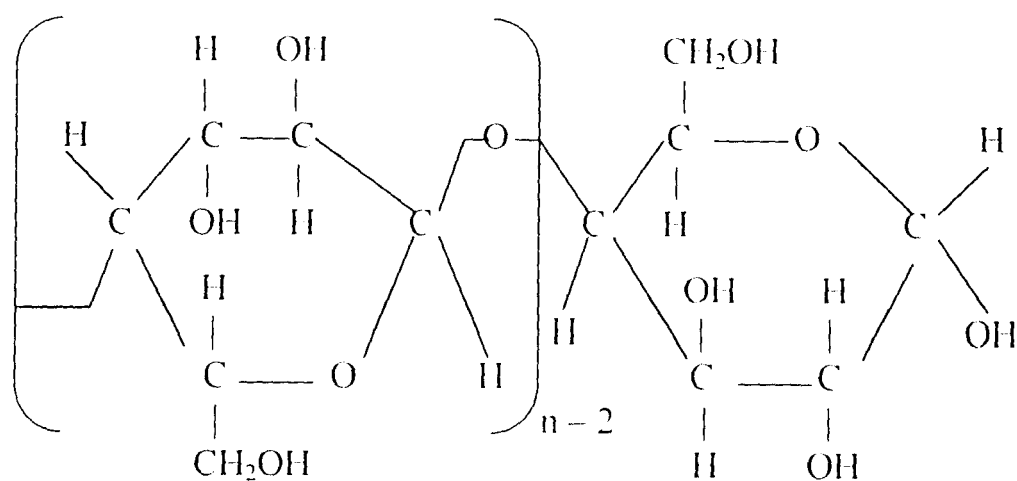


FIGURE 1

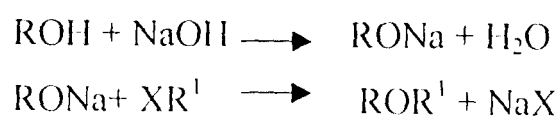
Molecular structure of cellulose: - It is a long chain polymer composed of glucosidic residues (n indicates a large number of such residues) joined by ether bridges in the (1), (4) positions.

2.4.5 PHYSICAL AND CHEMICAL PROPERTIES

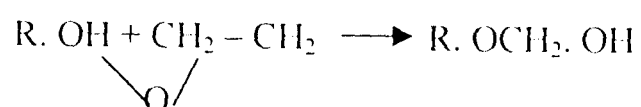
Cellulose is insoluble in water and organic solvents e.g. Benzene, alcohol, ether, acetone and chloroform. From the structure shown in figure 1, cellulose is seen to be a long chain polymer composed of $C_6H_{10}O_5$ (glucosidic) residues joined by ether bridges in 1,4 position.

The three hydroxyl groups on each glucose unit can be esterified with such organic agents as a mixture of acids and acid anhydrides with a suitable catalyst such as sulphuric acid. Inorganic acid for example mixture of sulphuric acid and nitric acids can be used to give the nitrate ester.

Ethers can be formed through the action of concentrated sodium hydroxide to form "Soda cellulose" followed by reaction with an alkyl halides thus:



The action of ethylene oxide or propylene oxide yields hydroxylated ethers thus:



Adjacent chains of cellulose have strong polar attraction for one another, because of the presence of hydroxyl groups and the geometry of the molecule. These forces are so strong that there are no common solvents, which will disrupt them and thus dissolve cellulose. These free hydroxyl groups are also responsible for the high moisture absorption of cellulose.

Etherification or simple etherification lowers moisture absorption and increases solubility in common solvents. Attack by aqueous acid splits the chain at the 1,4.oxygen link; complete splitting yields glucose, a simple sugar. The original chain length varies with the source of cellulose. It is longest in the native state but diminishes, as the cellulose is isolated purified, and converted to derivatives (McGraw Hill, 1983).

Even mechanical shear such as grinding will shorten the original long chain found in nature. As the polymer chain length degrades, especially below certain minimum values, gross physical properties are affected.

Oxidizing agents attacks cellulose in several preliminary ways with out chain scission. Further action in the presence of moisture, as in weathering generally ruptures the chains and increases the number of aldehydes groups.

Since aldehyde groups are easily oxidized to carboxyl groups, the carboxyl content, practically absent in native cellulose, rises sharply in weathering or oxidation. As with polymers, cellulose breaks down on weathering due to the combined action of oxygen, moisture, acidic components in the atmosphere and sunlight. The ultra violet components of sunlight are an important factor and many good ultra violet screening agents prolong the life of cellulose desirables.

Acidic components such as oxides of nitrogen or sulphur found in all industrial atmospheres-accelerate decomposition often at a faster rate than sunlight it self.

2.5 GLUCOSE

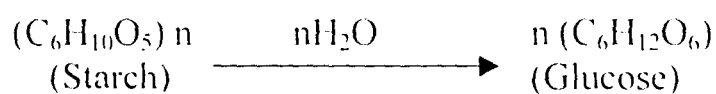
Glucose is a simple sugar and it is the most common of naturally occurring sugar. It is otherwise known as "dextrose" or "grape sugar" in honey and in sap of plant. There are three forms of glucose all of which have the formula: $C_6H_{12}O_6$ (Brian and Allan, 1995).

The three forms differ in certain physical properties. Their chemical properties however are similar. Glucose of all forms is soluble in water and has a sweet taste, although their sweetening or ability is less than that of sucrose or ordinary table sugar. The molecule of glucose contains an aldehyde group, CHO, and five-hydroxyl group, OH, which are characteristics of alcohols.

Chemically therefore, glucose may behave as an aldehyde or as an alcohol. The most common form of glucose, also called dextrose, grape sugar and corn sugar occurs widely in plants and in the blood of man and other animals. It forms colourless crystals that melt at 46°C (236°F). It is an important source of energy for the body since it can be utilized directly with out any intervening digestive process. For this reason, glucose is widely used in hospitals to feed patients intravenously when they are unable to ingest orally.

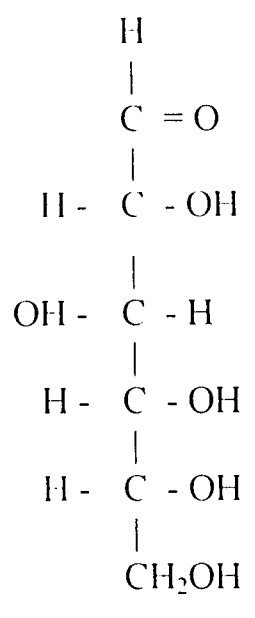
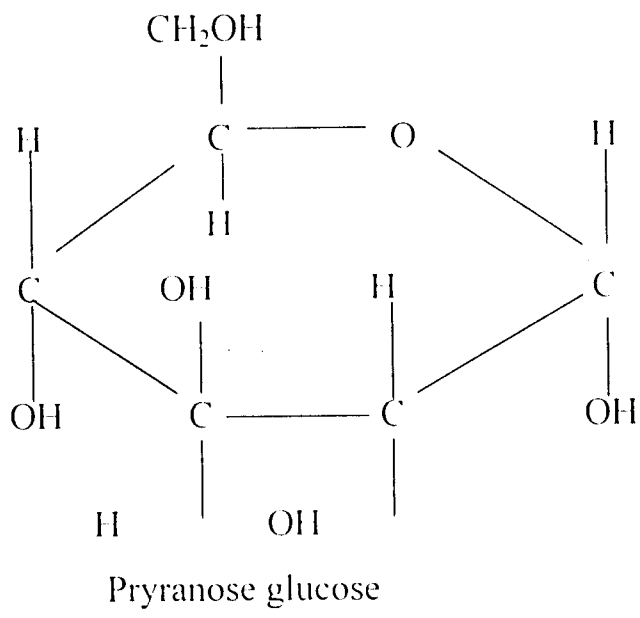
Glucose is also used in the manufacture of products such as candy (sweets) and jam.

Commercially, glucose is prepared by the hydrolysis of starch. In this process, a starchy substance is heated with water that contains a little hydrochloric acid. The product known as commercial glucose is a thick, syrupy, yellowish liquid that contains about 30 percent dextrose. It is used in the tobacco curing and in the tanning industry. The reaction for the production of glucose from starch is shown below.



2.5.1 STRUCTURE

Glucose occurs in solution in a ring form called pyranose and as well as aliphatic (open) straight chain. In the solid crystals, however research has shown that they exist in the ring form.



Adjacent chains of cellulose have strong polar attraction or one another because of the presence of hydroxyl groups and the geometry of the molecule. These forces are so strong that there are no common solvents, which will disrupt them and thus dissolve cellulose. These free hydroxyl groups are also responsible for the high moisture absorption of cellulose.

Etherification or simple etherification lowers moisture absorption and increases solubility in common solvents. Attack aqueous acid splits the chain at the 1,4.oxyen link; complete splitting yields glucose, a simple sugar. The original chain length varies with the source of cellulose. It is longest in the native state but diminishes as the cellulose is isolated purified and converted to derivatives.

Even mechanical shear such as grinding will short the original long chain found in nature. As the polymer chain length degrades especially below certain minimum values gross physical properties are affected.

Oxidizing agents attacks cellulose in several preliminary ways with out chain scission. Further action in the presence of moisture, as in weathering generally ruptures the chains and increases the number of aldehydes groups.

Since aldehyde groups are easily oxidized to carboxyl groups, the carboxyl content, practically absent in native cellulose, rises sharply in weathering or oxidation. As with polymers, cellulose breaks down on weathering due to the combined action of oxygen, moisture, acidic components in the atmosphere and sunlight. The ultra violet components of sunlight is an important factor and many good ultra violet screening agents prolong the life of cellulose desirables.

Acidic components such as oxides of nitrogen or sulphur found in all industrial atmospheres-accelerate decomposition often at a faster rate than sunlight it self (McGraw Hill, 1983).

2.5.2 USES OF GLUCOSE

The digestion of cellulose in to glucose has received a high attention in the research world. The power or ability to transform this organic chemical (cellulose) has extremely important implication, as a break through into it digestion will have enormous impact on the world food supply, economy and geopolitical balance of power. It will also influence greatly, the ways and type of products produced by the chemical industries and it utilization by the consumers.

The synthesised glucose can be used for the following:

- a – Fermentation of glucose to ethanol by *sacchonomyces cervicae* (yeast) or *pseudomonas mobilis* (bacterium). Ethanol can be used as gasoline or processed further to make common petrochemicals.
- b – Production of single cell protein as food for livestock or even for humans.
- c – Starting raw material in the production of a wide variety of chemicals and fuels. This is usually carried out with the help of micro – organisms, e.g. in conversion of glucose into solvents such as acetone and butanol by *clostridium acetobutylicum*.

2.6 HYDROLYSIS (BRIEF SUMMARY)

Hydrolysis literally means destruction, decomposition, or alteration of a chemical substance by water.

It's used to describe chemical reactions where water is one of the active reactants in the reaction. It is often a decomposition reaction in which the water breaks apart the other reactant to form two or more different compounds.

This is implied in the term, which comes from Greek word meaning "water" and "dissolution" (HYDRO - LYSIS).

In some hydrolysis reaction however, a water molecule reacts with the other compound by adding on to it, and catalysts are often used to bring about hydrolysis. Degree of hydrolysis is the fraction of the ions that react with the water (Brian and Allan, 1995).

In terms of aqueous solutions of the electrolytes, the term hydrolysis is applied especially to reactions of cation (positive ions) with water to produce a weak base or of both weak acid and weak base is then said to be hydrolysed.

Thus, inorganic chemistry hydrolysis is the partial decomposition of salt dissolved in water into an acid or alkali. In organic chemistry, the term hydrolysis is used more broadly to include reactions where organic compounds are decomposed by solutions of acid or alkalis in water. Organic hydrolysis reactions are common in industry e. g ethyl alcohol; an important industrial raw material may be obtained by the hydrolysis of ethylene in the presence of phosphoric acid catalyst (McGraw Hill, 1983).

In addition, esters are widely hydrolysed by alkaline to give an alcohol and an organic salt of the alkali concerned. Hydrolysis of ester is called saponification.

In the manufacture of soap, fats which contain a mixture of triglycerides (esters formed from the trihydric alcohol glycerol and fatty acids such as stearic, oleic and palmitic) are saponified using sodium hydroxide (NaOH) to give mixture of sodium salts of fatty acids and glycerol.

Glycerol is used in the manufacture of the industrial chemical phenol (C_6H_5OH) by Reschig process.

Hydrolysis of starch results in dextrose (a type of glucose), in the process, glycosidic linkages are broken to release glucose (McGraw Hill, 1983).

2.7 HYDROLYSIS METHODS

Hydrolysis can occur in different ways by different means; four main methods of hydrolysis are however established and they are as follows:

- Acid hydrolysis
- Alkaline hydrolysis
- Enzyme hydrolysis and
- Direct microbial conversion

Nevertheless, this project investigates the comparative effect of an acid and alkali on hydrolysis rate.

2.7.1 ACID HYDROLYSIS

Acid hydrolysis can be carried out with dilute or concentrated acids. Concentrated acid hydrolysis has the advantage of proceeding at low temperatures. The sugar formed is not degraded. However, concentrated acid proceedings required large amount of acid to wet the feed stock. Acid recovery is thus necessary and this is a very expensive operation. Hence, virtually all-recent process development work has been done using dilute acids. Cellulose hydrolysis in dilute requires relatively high temperature and care should be taken to avoid the decomposition of the sugar product (Harris et al, 1963 and Millet et al, 1976).

Advantages of acid hydrolysis are:

- 1 – Acid hydrolysis has short reaction time
- 2 – It has been extensively practiced at all levels and with better yield.
- 3 – The acid catalyst is cheap and readily available.
- 4 – Suitability of any cellulose feed stock to acid hydrolysis particular food.

2.7.2 ALKALINE HYDROLYSIS

Alkaline hydrolysis can be carried out with dilute or concentrated alkalis. Two things govern cellulose hydrolysis in an alkaline buffer; the peeling reaction and the breakage of inter molecular glucosidic linkage of the cellulose. This peeling reaction is the progressive erosion of monosaccharide links from the reducing end of the polysaccharide; this peeling of monosaccharide is frequently more definite than other methods such as enzyme hydrolysis (which degrade from the non-reducing end of the molecule where branched structures has to be analysed owing to the availability of only one reducing end to the molecule.

Alkaline hydrolysis is in all forms similar to acid hydrolysis since they are both chemical degradation techniques. It is carried out with aqueous alkali like sodium hydroxide (NaOH). However, it requires large quantity to wet the feedstock. Alkali recovery is thus necessary which is quite an expensive operation. For commercial purposes, dilute alkali should be used with temperatures ranging from 200°C and above so as to reduce cost of alkali recovery.

Advantages of alkaline hydrolysis are:

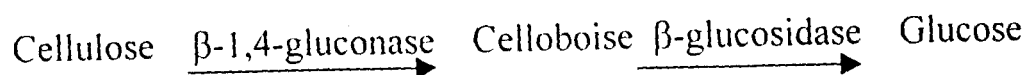
- 1- Short reaction time relative to other methods.
- 2- The alkali is readily available and cheap.
- 3- Suitability to any cellulose feedstock

2.7.3 ENZYME HYDROLYSIS

Enzyme hydrolysis involves specific enzyme reaction where no secondary reactions with products or other components are catalysed under the condition and does not require the use of expensive corrosion resistance alloys. However, the conversion of cellulose in to glucose is now known to consist of two steps in the enzyme system of *Trichoderma virida*.

In the first step beta -1,4 gluconase breaks the glucosidic linkage to cellulose, which is a glucose dimer with a beta-1,4 bond as opposed to matose, a counterpart with an alpha-1,4 bond.

Subsequently, this beta-1,4 glucosidic linkage is broken by beta-glucosidase as shown below:



Advantage of Enzyme hydrolysis

- (1) Specific enzyme reactions. No secondary reaction with product or other components is catalysed under the condition of temperature and pH.
- (2) Mild operating condition (atmospheric pressures, 4.5 to 5.0 pH and 45°C) do not require the use of expensive corrosion resistant alloys and pressure vessel constructions.
- (3) The cause for loss in yield is eliminated because production of clean sugar streams for further processing is enhanced.
- (4) Costly neutralisation and purification equipment is not needed.

On the hand, enzyme hydrolysis has some minor set backs like:

- 1- Costly enzyme production, which makes the process an expensive one.
- 2- Enzyme recovery is difficult.

2.7.4 DIRECT MICROBIAL CONVERSION

The idea of direct microbial conversion of cellulose involves the production of a sample product by a single organism growing on a cellulose substrate. A more elaborate approach is the involvement of two organisms, where one degrades cellulose and yields products for hexose and pentose sugars for consumption by a second organism, which also produces the derived product allowing more complete utilization of the ligno cellulose (Wang, 1991; Brian and Allan, 1995).

However, this concept is not easily achieved in practice.

CHAPTER THREE

EXPERIMENTS

3.0

3.1 MATERIALS:

3.1.1 ALKALI

Sodium hydroxide pellets were used for alkaline hydrolysis process

3.1.2 ACID:

5M concentrated Sulphuric acid with specific gravity of 1.8 and purity of 98% was also used for acid hydrolysis process.

3.1.3 SAW DUST (Mahogany – khaya-Ivorensis)

Saw dust was collected at timber shed along Maitumbi road in Minna Niger State.

3.1.4 CHEMICALS AND REAGENTS

The chemicals and reagents used include:

Diethyl ether.

8.0M Sodium hydroxide

6M Sulphuric Acid

Low alkalinity Copper reagent

Arsenomolybdate reagent

Standard Glucose

3.2 APPARATUS AND EQUIPMENTS

3.2.1 Hydrolysis Reactor:

250ml beaker was used as stirred reactor.

Water Bath:

Two (2) Thermostatic water baths were used to carry out the hydrolysis.

3.2.2 Calorimeter:

A colorimeter model 6051 was used to monitor glucose concentration of various samples.

3.2.3 Oven:

A gallon lamp electric oven was used to carry out the sample drying process.

3.2.4 OTHER APPARATUS:

Digital weighing balance, sieves, Beaker (100ml). Filter paper, funnel, conical flasks, test tube racks, measuring cylinder, pipettes, spatula and stop watch.

3.3 PROCEDURE

3.3.1 PRE-TREATMENT

MILLING – The sawdust obtained was of large size and therefore to achieve effective hydrolysis, it's reduced to 250 μ m by grinding and sieving with sieve of 250 μ m aperture.

3.3.2 REMOVAL OF EXTRACTIVE

The presence of hemicelluloses and lignin provides protective sheath around the cellulose, this makes it difficult to hydrolyse the cellulose by the hydrolysing agent. Therefore, it is paramount to remove or modify the protective sheath for effective hydrolysis to occur.

The samples of sawdust were leached to extract fibre, fats, lignin, proteins, lipids and hemicelluloses (pentosans). 10 grams of sample (sawdust) was treated with 40ml of diethyl ether in a 50ml beaker and covered for three (3) hours.

The resulting residue was then washed with distilled water, filtered and dried in oven.

3.3.3 LIGNIN ISOLATION

This is the process of precipitating cellulose with concentrated acid mainly sulphuric acid. 10grams of treated sawdust was mixed with 40mls of 6M sulphuric acid for four (4) hours. The filtrate containing dissolved cellulose was further treated with 70ml of 8.0m Sodium hydroxide which precipitate the cellulose. The

precipitate was washed thoroughly with distilled water, filtered and dried. The percentage cellulose was found to be 31.38%.

3.3.4 PREPARATION OF STANDARD GLUCOSE CALIBRATION CURVE

0.1 gram of standard D. glucose was diluted in 100ml-distilled water. This was designated as the standard D – glucose solution. 2ml, 6ml, 8ml and 10ml of the standard solution are put in to the conical flasks and then were made up to 20ml with distilled water.

2ml of copper reagent were added to 2ml of each diluted samples, and then boiled for 10 minutes. The resulting solutions were allowed to cool and to it was added 2ml of Arsenomolybdate reagent, shaken and optical density were read using colorimeter at 470nm. The results were plotted as a standard calibration curve.

3.4 HYDROLYSIS STAGE

3.4.1 ALKALINE HYDROLYSIS

3.4.1.1 Effect of Temperature on Sawdust Hydrolysis in Alkali

40grams of Sodium hydroxide pellet was dissolved in 1000ml of distilled water to obtain 1 molar solution. To obtain 0.4ml, 0.5ml, 0.6ml of sodium hydroxide solution, $M_1V_1 = M_2V_2$ relationship were used, where M stands for molar concentration and V is the volume in mls.

10grams of the sample was treated with 50ml of 0.4m of sodium hydroxide in a 250ml beaker. The beaker was covered and placed in a thermostatic water bath at a preset temperature of 65^oc for three hours.

2ml of the solution was taken from it after every 30 minutes in to a test tube, and to it was added 2ml of copper reagent, boiled for 10minutes and allowed to cool. To the resulting solution was added 2ml of Arsenolybdate reagent and then made up to 25ml with distilled water.

The absorbance was then read from a colorimeter at 470nm. This was done after every 30minutes until the three (3) hours period

elapses. The same procedure was repeated at temperatures of 70°C, 75°C and 80°C.

3.4.1.2 Effect of Alkali Concentration on Sawdust Hydrolysis at Constant Temperature of 80°C.

0.5M and 0.6M sodium hydroxide solutions were prepared from 1M solution by diluting 50ml and 60ml with 50ml and 40ml of distilled water respectively to obtain 100ml of each concentration. 1.0grams of the sample was hydrolysed with 50ml of 0.5M Sodium hydroxide at 80°C, and 2ml aliquot was taken at 30minutes interval for three (3) hours. At each withdrawal stage the aliquote was mixed with 2ml of copper reagent and boiled for 10 minutes. The resulting solution were allowed to cool and to it were added 2ml of Arsenomolydate reagent and made up to 25ml, after which the optical density are read and noted. The same procedure was followed for 0.6ml sodium hydroxide at 80°C.

3.4.2 ACID HYDROLYSIS

3.4.2.1 Effect of Temperature on Sawdust Hydrolysis at Constant Acid Concentration

2.5M of sulphuric acid was prepared from 5M sulphuric acid using the relation $M_1V_1 = M_2V_2$ as stated earlier in the alkaline hydrolysis stage.

1.0grams of sample was treated with 50ml of sulphuric acid in a 250ml beaker. The beaker was then covered and placed in a thermostatic water bath at a preset temperature of 65°C for 120 minutes. 2ml of the solution was taken from it after every 20 minutes into a test tube, and to it were added 2ml of copper reagent boiled for 10 minutes and allowed to cool. To the resulting solution was added 2ml of Arsenomolybdate reagent and made up to 25ml with distilled water. The optical density were used read with colorimeter at 470nm.

The same sequence was followed at 20 minutes interval until the 120 minutes period elapses. The procedure is repeated at 70°C, 75°C and 80°C temperatures.

3.4.2.1 Effect of Acid Concentration on Sawdust at Constant Temperature of 80°C.

3.5M and 4.5M of sulphuric acid were prepared from 5M acid using $M_1V_1 = M_2V_2$ relationship.

1.0 grams of sample were mixed with 50ml of 3.5M sulphuric acid in a 250ml beaker, and placed in a thermostatic water bath at preset temperature of 80°C. 2ml aliquote was taken from the solution after 20minutes, to it was added 2ml of low alkalinity copper reagent and boil for 10minutes after which it was allowed to cool. To the resulting solution were added 2ml of Arsenomolybdate reagent and made up to 25ml with distilled water. The absorbance was read with a colorimeter at 470nm. This was done after each 20minutes until the 120minutes period elapses. The procedure was repeated for acid concentration of 4.5M.

CHAPTER FOUR

4.0 GRAPHICAL RESULT

The standard glucose calibration curve is represented by Figure 4.1, which reveals a linear relationship between the absorbance and glucose concentration. This curve serves as the basis for the derivation of other concentration at different temperature and medium concentration.

4.1 EFFECT OF TEMPERATURE ON THE HYDROLYSIS OF SAWDUST AT CONSTANT MEDIUM CONCENTRATION

The effect of temperature on the hydrolysis of sample was analysed over the range of 65^oc to 80^oc using 0.4M NaOH and 2.5M H₂SO₄ for alkaline and acid medium respectively. These are illustrated in figure 4.2a and 4.3a, which shows an increase glucose concentration with increasing temperature and time, given a highest amount of glucose at 80^oc.

4.2 EFFECT OF MEDIUM CONCENTRATION ON THE HYDROLYSIS OF SAWDUST

Figure 4.2b and 4.3b illustrates the effect of varying medium concentration on the amount of glucose released at constant temperature of 80^oc using 0.4M to 0.6M NaOH for alkaline medium and 2.5M to 4.5M H₂SO₄ for acid medium.

These figures show a decrease in the concentration of D-glucose as medium concentration increases, though, increases as the hydrolysis period is elongated, with a better release of glucose in the acid medium.

4.3 DETERMINATION OF RATE CONSTANT AT DIFFERENT TEMPERATURE IN ACID AND ALKALINE MEDIA

The determination of the rate constants of reactions in the media are illustrated in figure 4.4 and 4.7 for alkaline and acid medium respectively. These figures are a plot of in $[a_0/(a_0 - x)]$

against time for various temperature gives a positive slope which represent the rate constant at the temperature. The rates are tabulated in table 4.4.1 and 4.8.1 using 0.4M NaOH and 2.5M H₂SO₄ for alkaline and acid medium respectively.

4.4 DETERMINATION OF ACTIVATION ENERGY

The activation energies of the media where obtained from the plot of $\ln k$ against $1/T$ which are illustrated in figure 4.5 and 4.8 for alkaline and acid medium respectively.

A negative slope was obtained from the plot, which represent $(-E_a/R)$ in the Arrhenius equation under logarithmic function as elaborated in the appendix.

4.5 GLUCOSE YIELD AT VARYING TEMPERATURE USING CONSTANT MEDIUM CONCENTRATION

Figure 4.6a and 4.9a using 0.4M NaOH and 2.5M H₂SO₄ for alkaline and acid medium represent the yields of glucose from both media respectively over a temperature range of 65°C to 80°C. The figures shows that yield increases with time and temperature at a constant media concentration, with a higher yield been recorded in the acid medium.

4.6 GLUCOSE YIELD AT VARYING MEDIUM CONCENTRATION AT CONSTANT TEMPERATURE OF 80°C

Figure 4.6b and 4.9b are plots of yield against time at varying media concentration of 0.4M to 0.6M NaOH for alkaline medium and 2.5M to 4.5M H₂SO₄ for acid medium.

This figures reveals a fall in the yield of glucose as the medium concentration increases. This means that increase in concentration reduces the amount of water molecules available for hydrolysis to take place at that temperature.

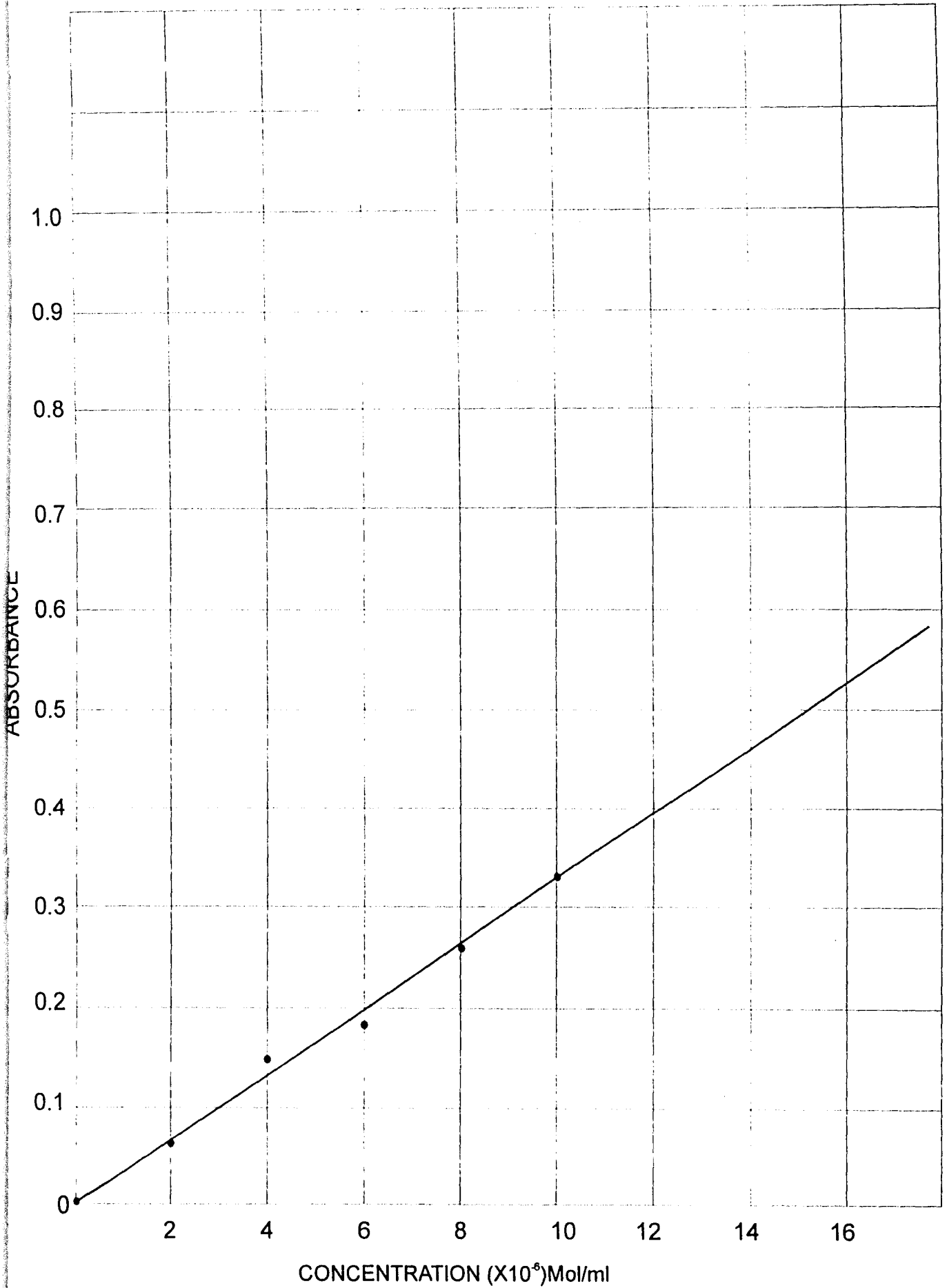


FIGURE 4.1: STANDARD GLUCOSE CALIBRATION CURVE

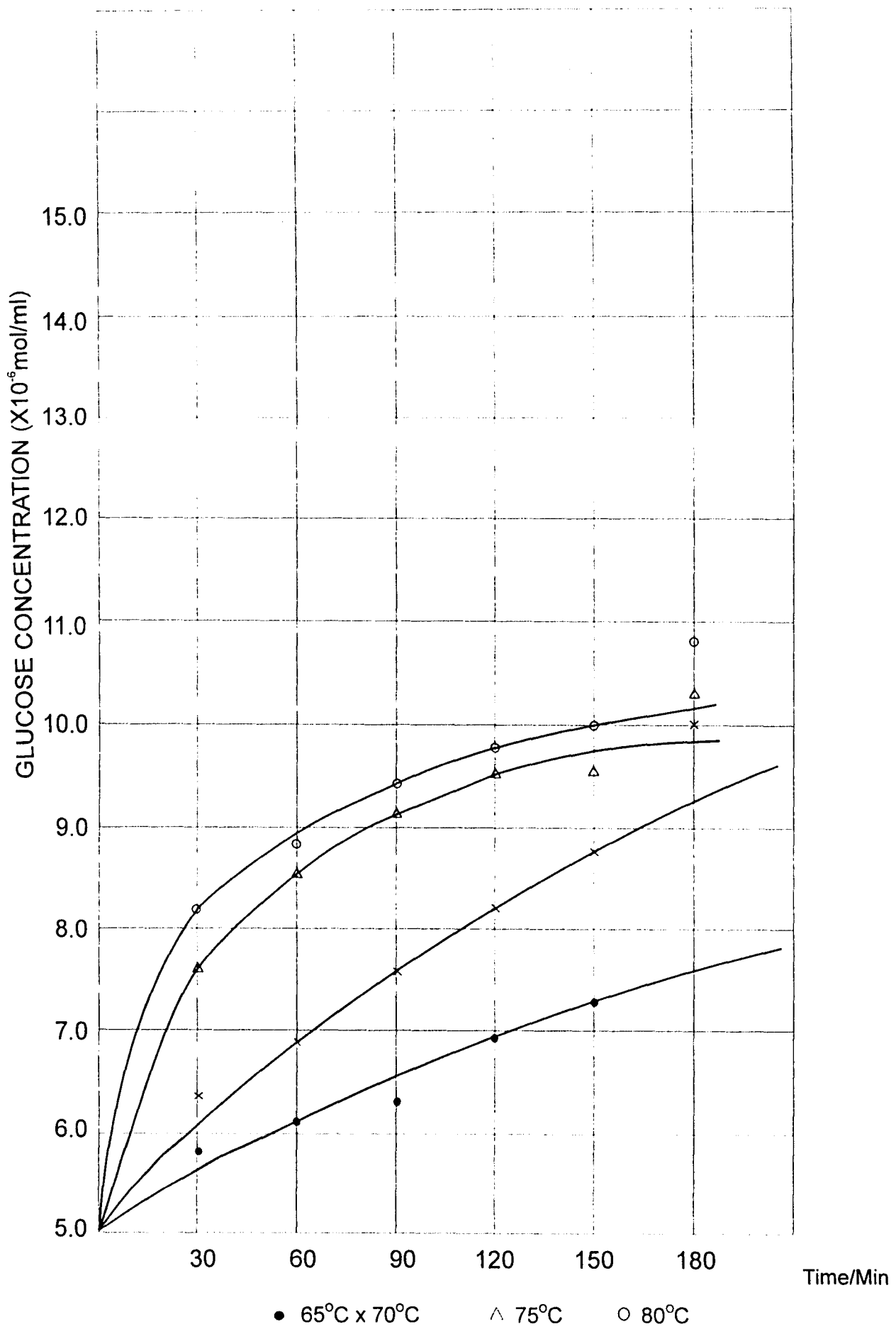


FIGURE 4.2a : PLOT GLUCOSE CONCENTRATION VS TIME AT ALKALI CONCENTRATION OF 0.4M NaOH

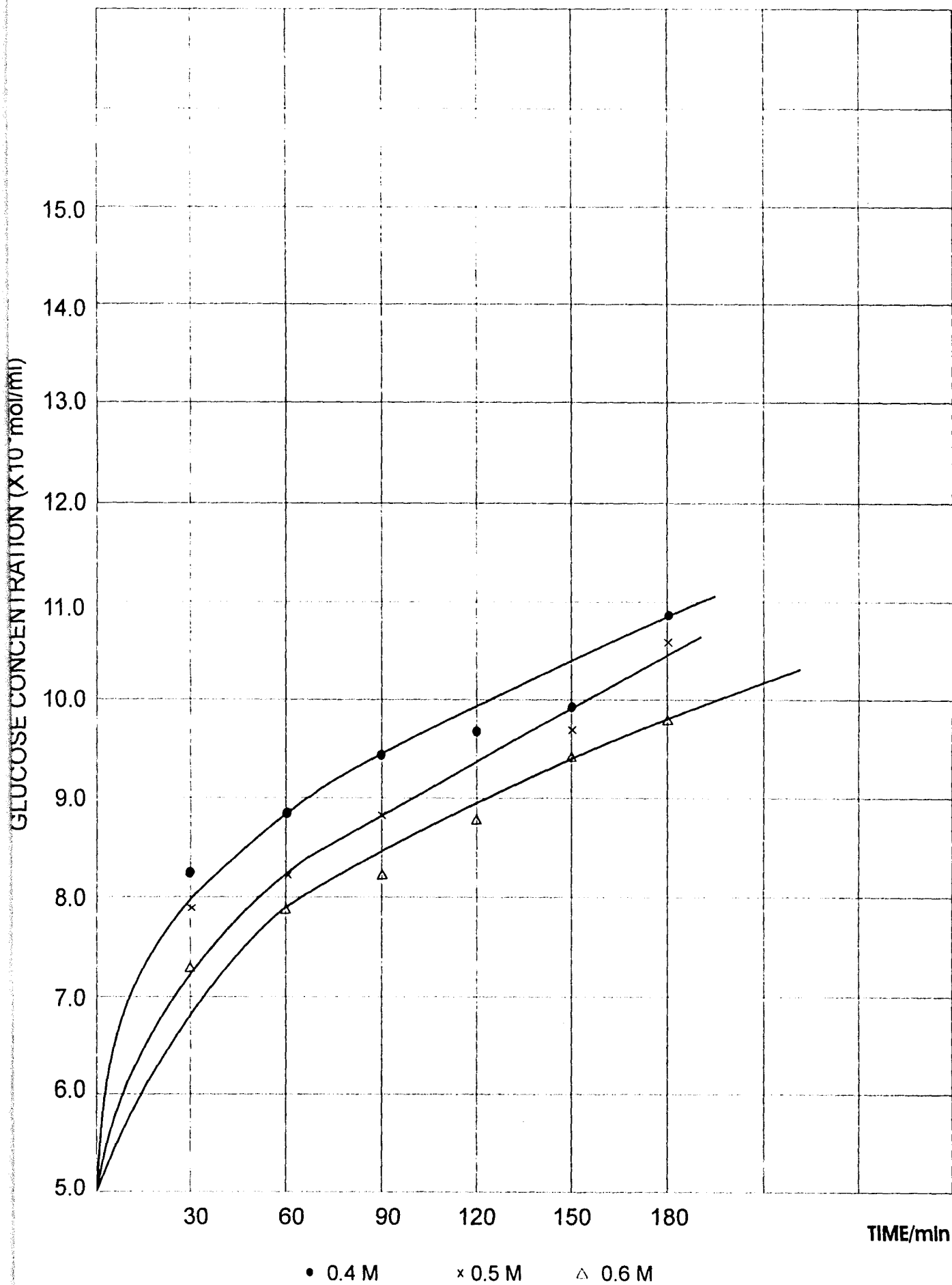


FIGURE 4.2b: PLOT GLUCOSE CONCENTRATION VS TIME AT 80°C FOR VARYING ALKALI CONCENTRATION

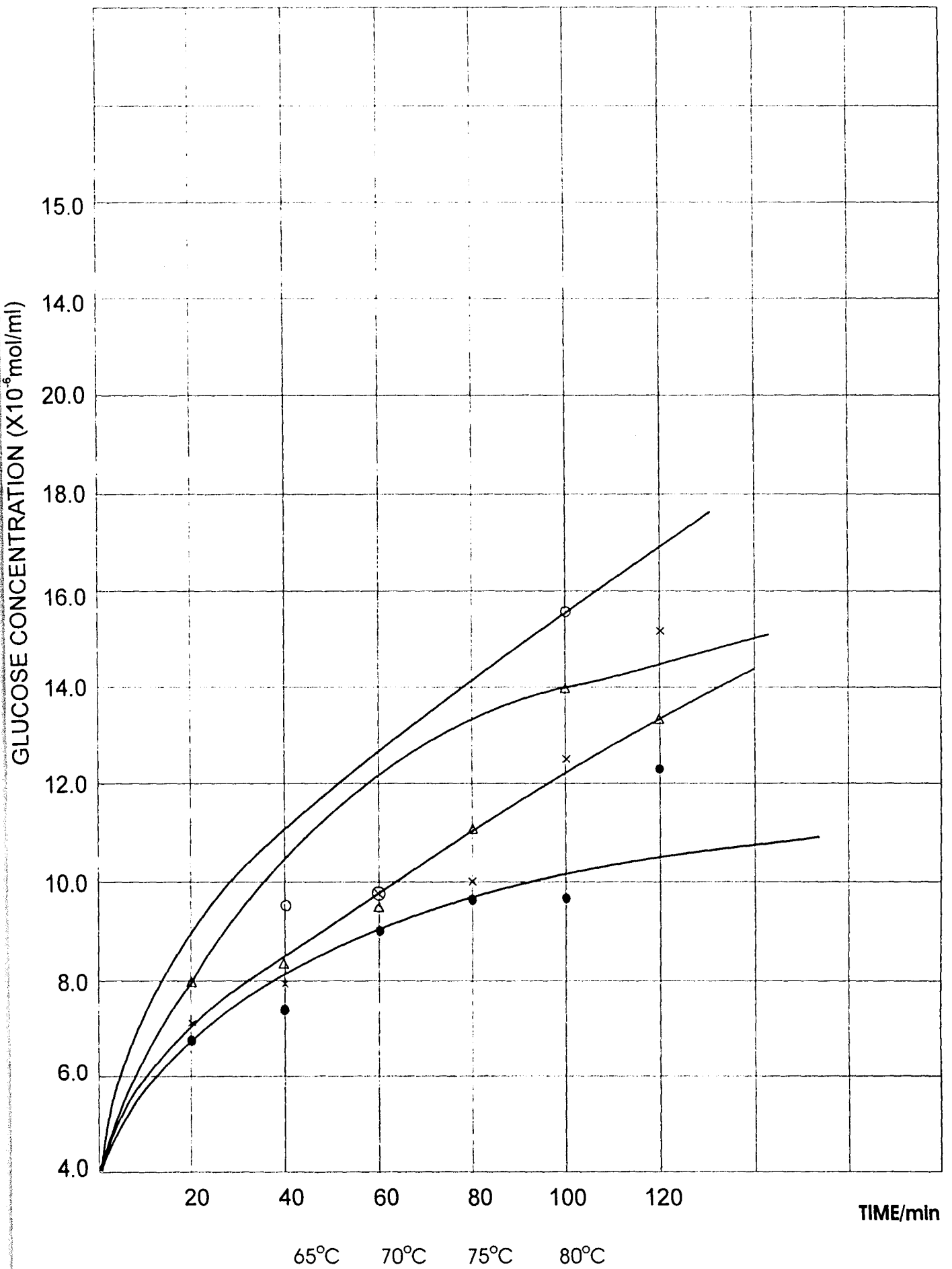


FIGURE 4.3a : PLOT GLUCOSE CONCENTRATION VS TIME AT ACID CONCENTRATION OF 2.5M H₂SO₄

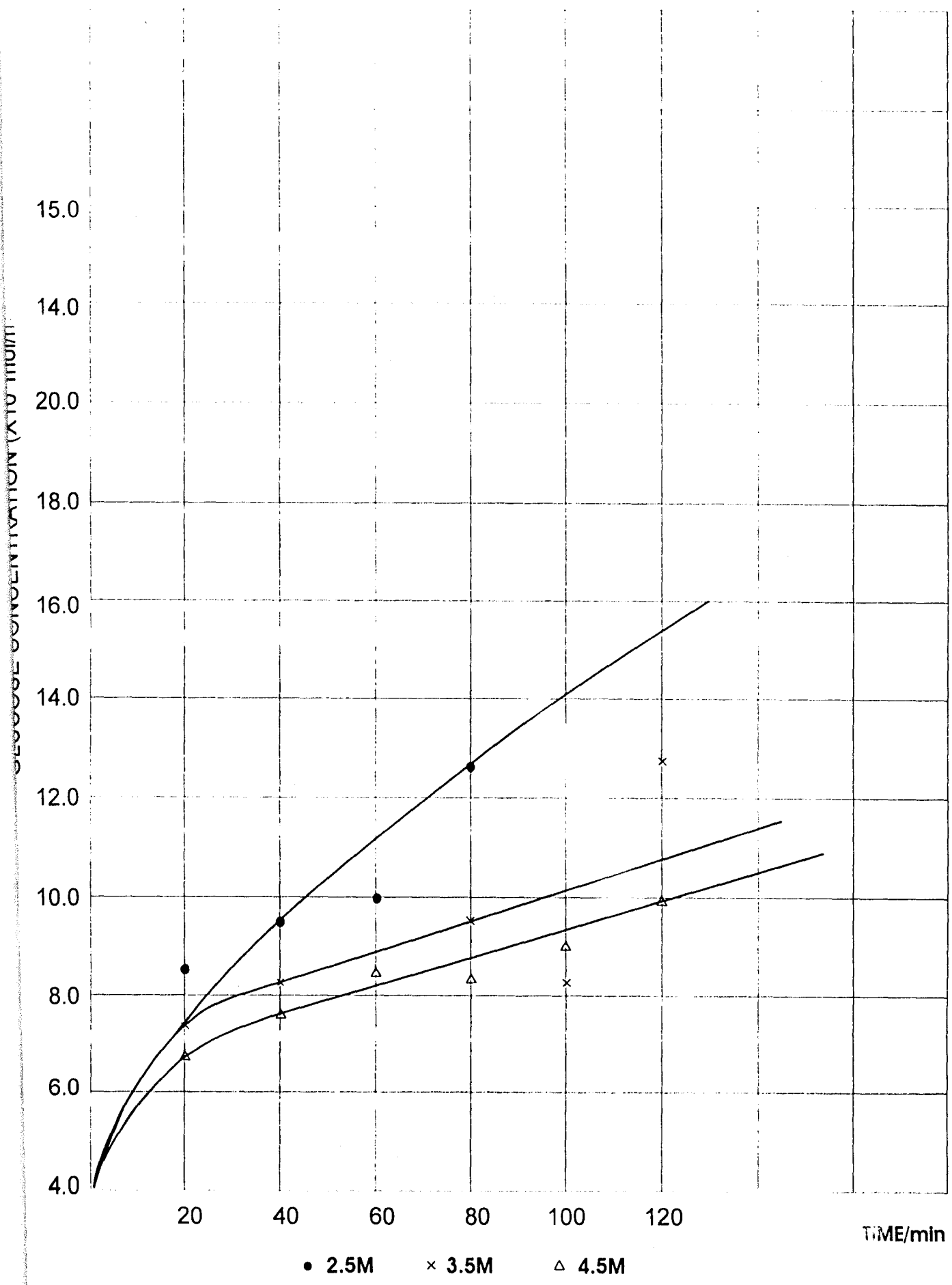


FIGURE 4.3b : PLOT GLUCOSE CONCENTRATION VS TIME AT 80° FOR VARYING ACID CONCENTRATION

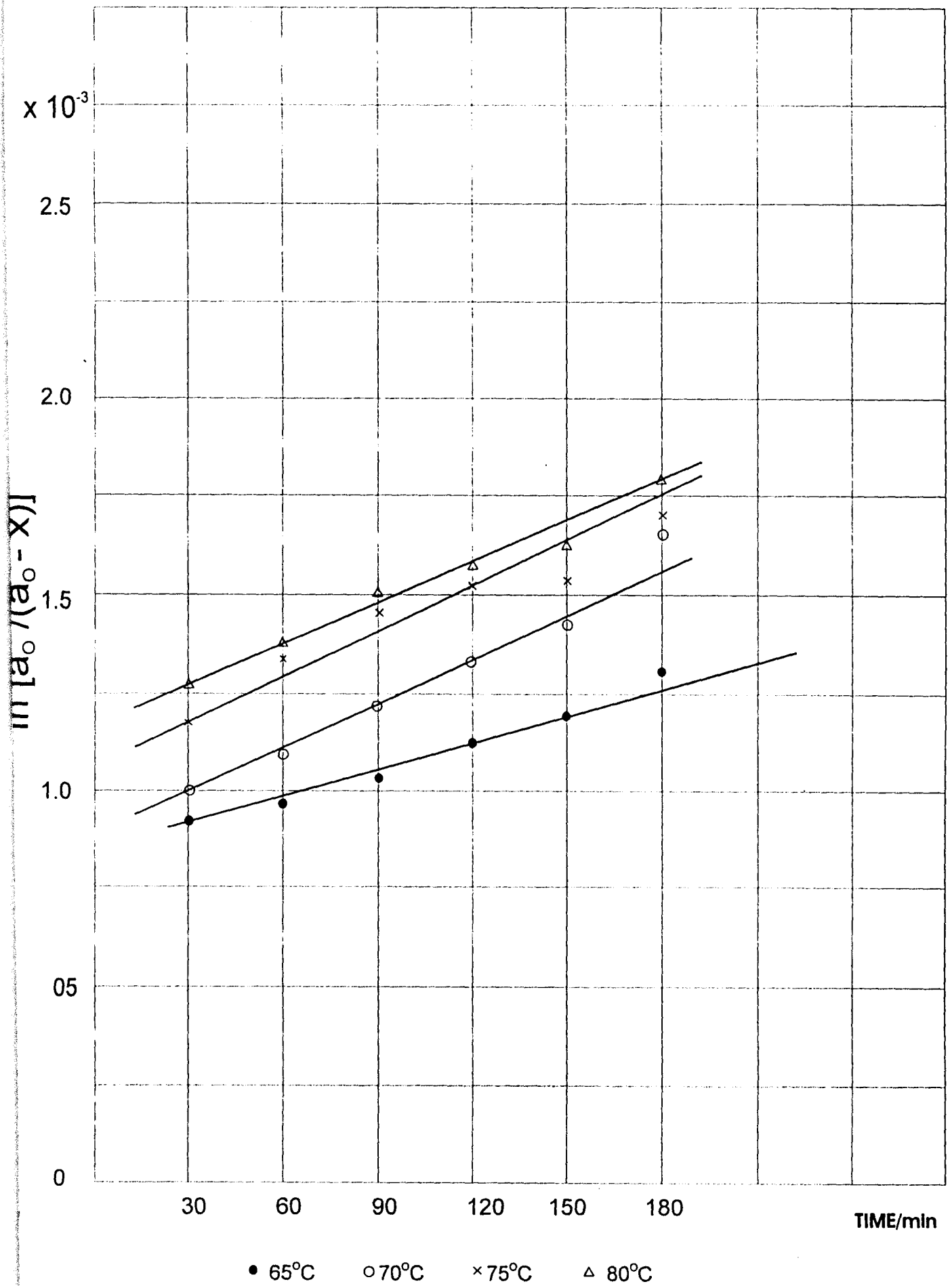


FIGURE 4.4: PLOT OF $\ln [(a_0/(a_0-x))]$ VS TIME FOR VARYING TEMPERATURE IN ALKALI MEDIUM

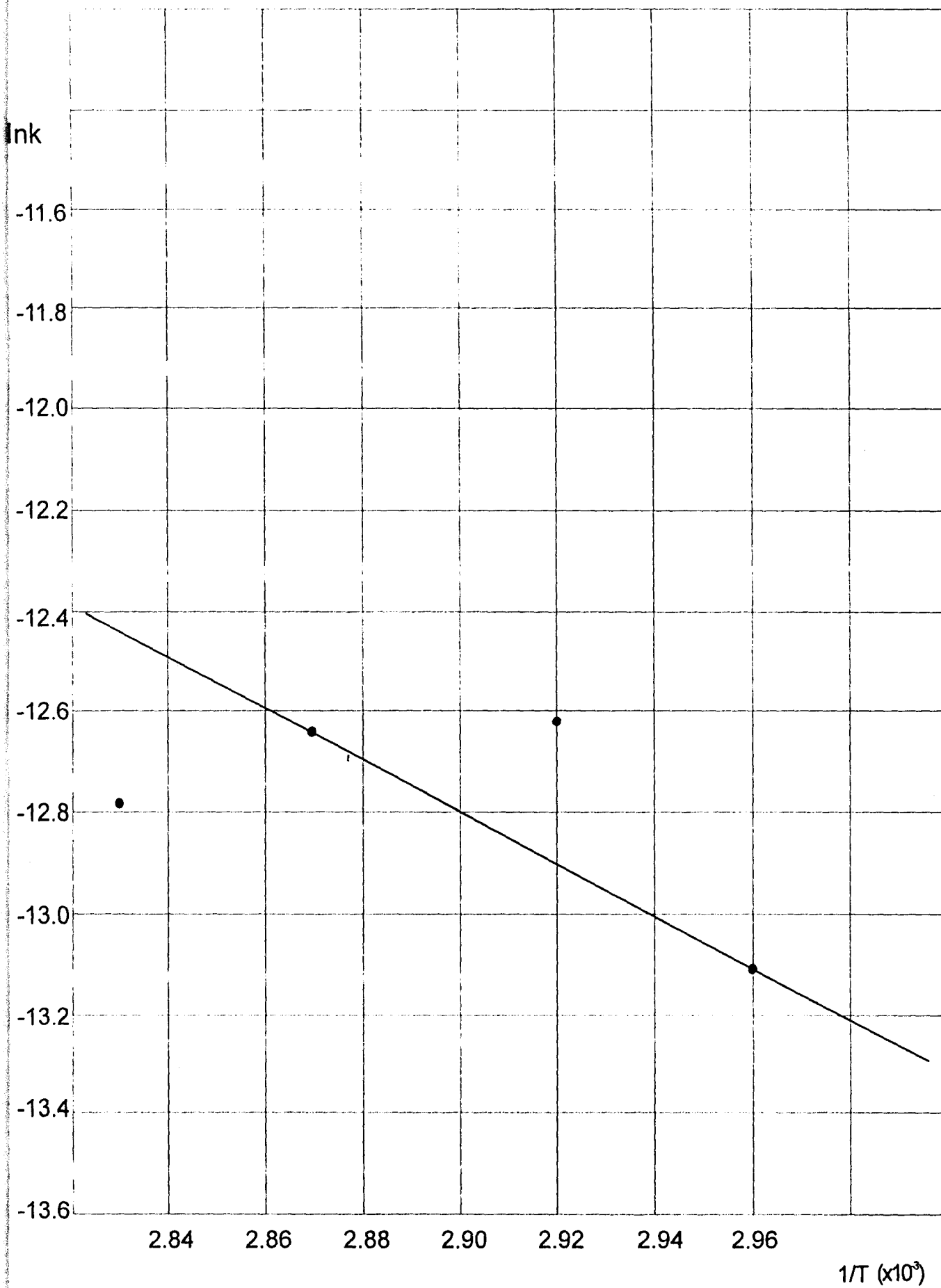


FIGURE 4.5: PLOT OF ln K VS 1/T ALKALINE MEDIUM

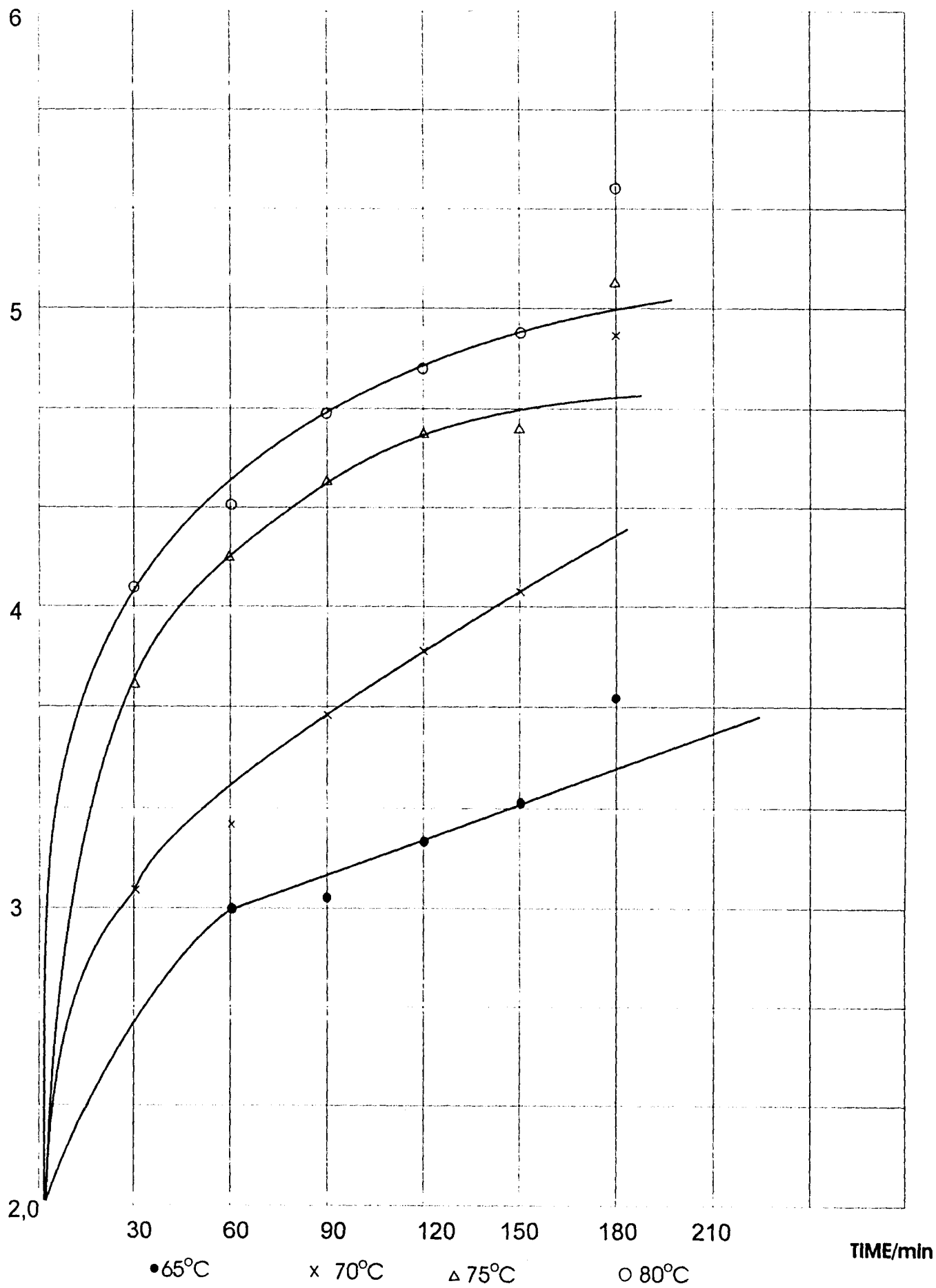


FIGURE 4.6a: PLOT OF GLUCOSE YIELD VS TIME FOR VARYING TEMPERATURE IN ALKALINE MEDIUM

GLUCOSE YIELD (X 10³ g)

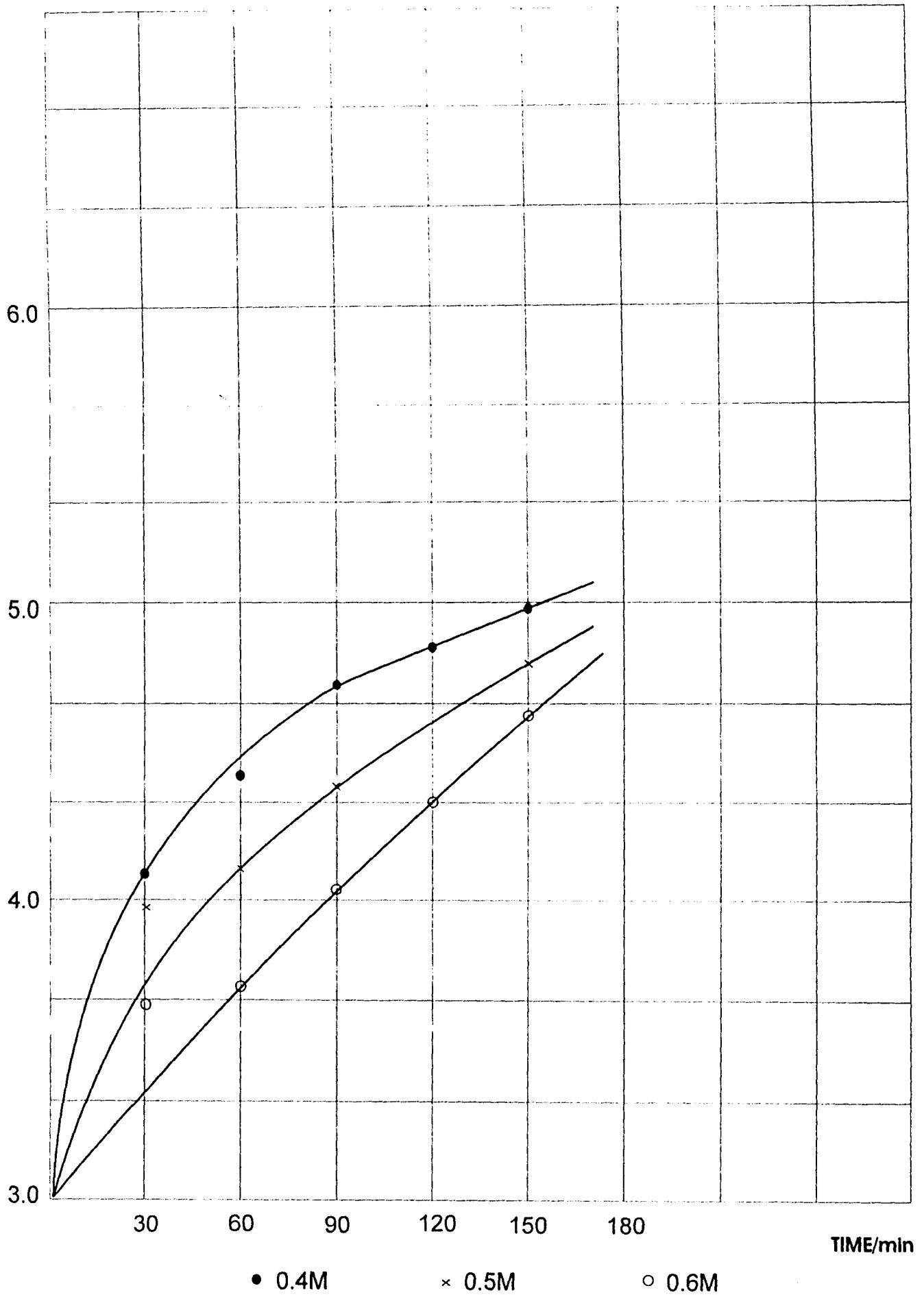


FIGURE 4.6b: PLOT OF GLUCOSE YIELD VS TIME AT 800C FOR VARYING ALKALI CONCENTRATION

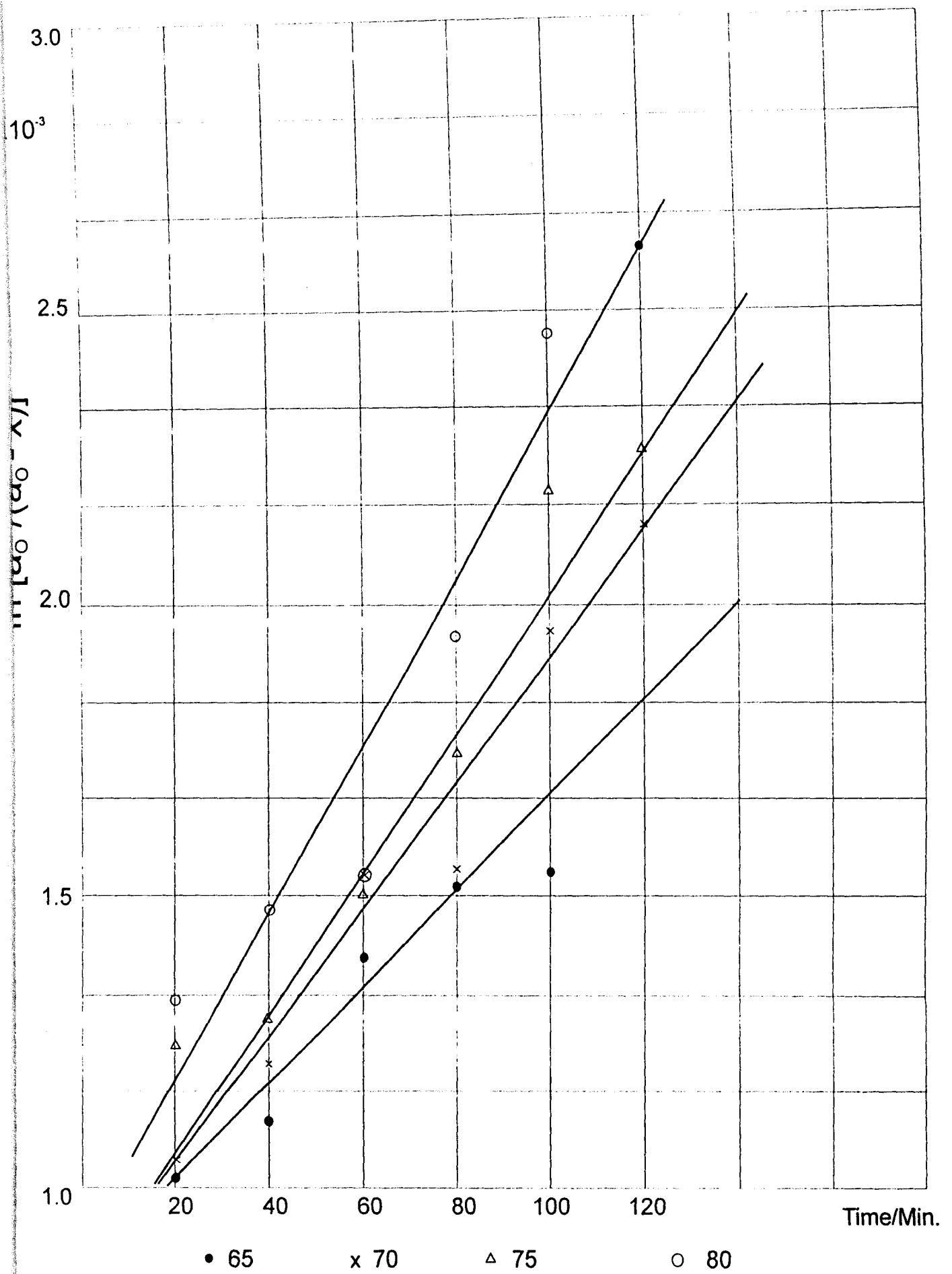


FIGURE 47: PLOT OF $\ln \left[\frac{a_0}{a_0 - x} \right]$ VS TIME FOR VARYING TEMPERATURE

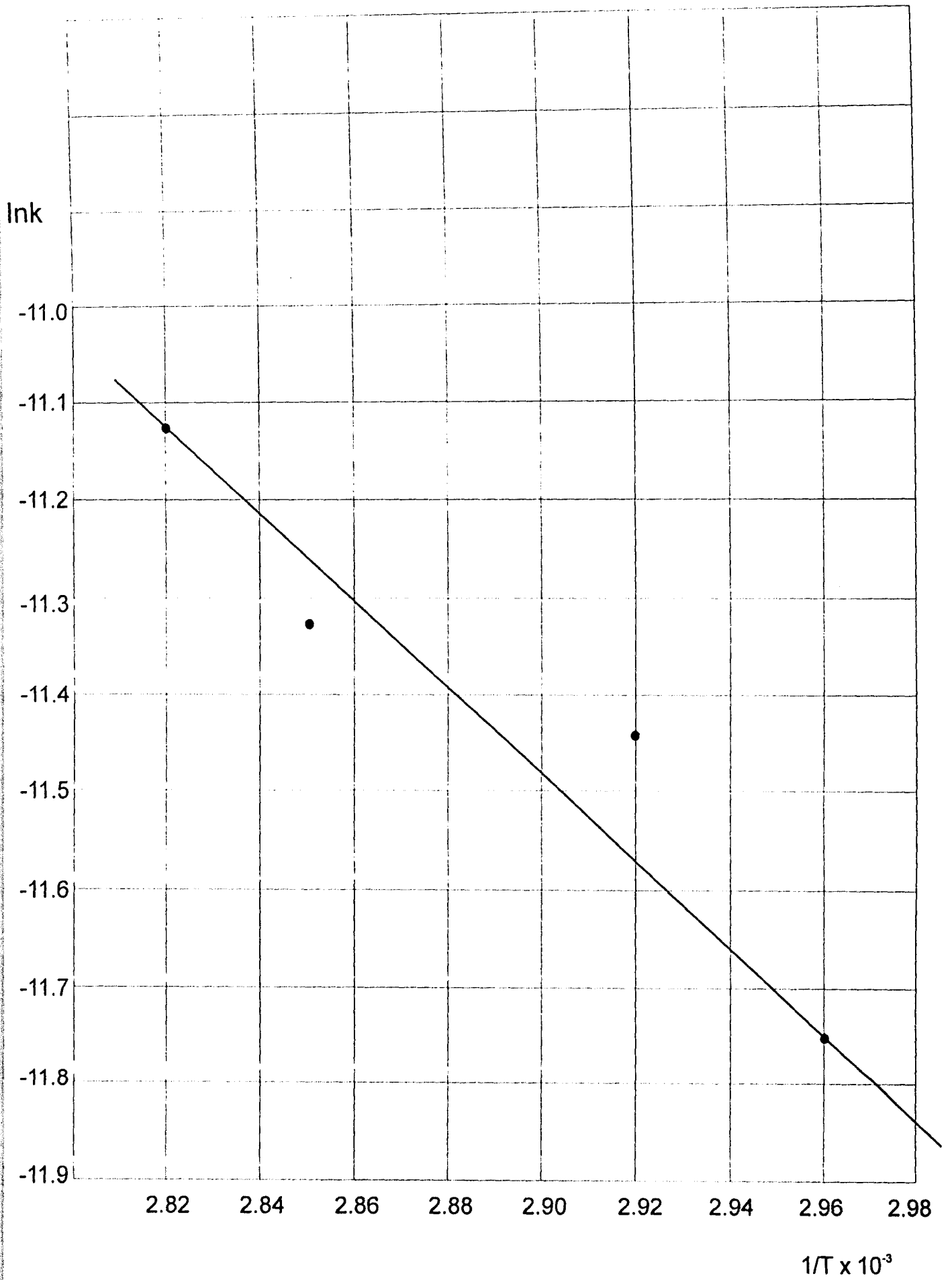


FIGURE 4.8: PLOT OF $\ln K$ VS $1/T$ FOR ACID MEDIUM

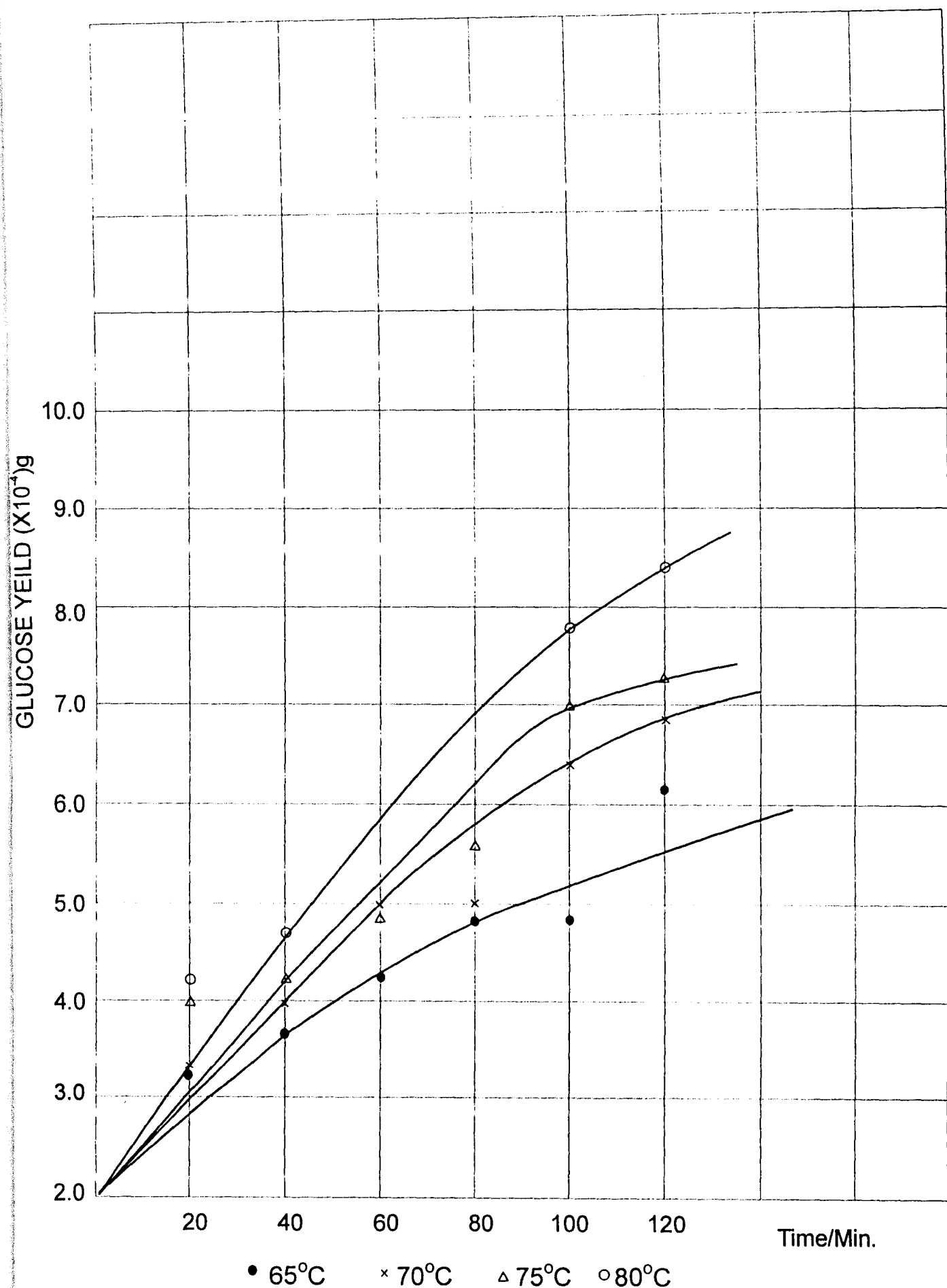


FIGURE 4.9a: PLOT OF GLUCOSE YIELD VS TIME FOR VARYING TEMPERATURE AT 2.5M H₂SO₄.

GLUCOSE YIELD (X 10³ g)

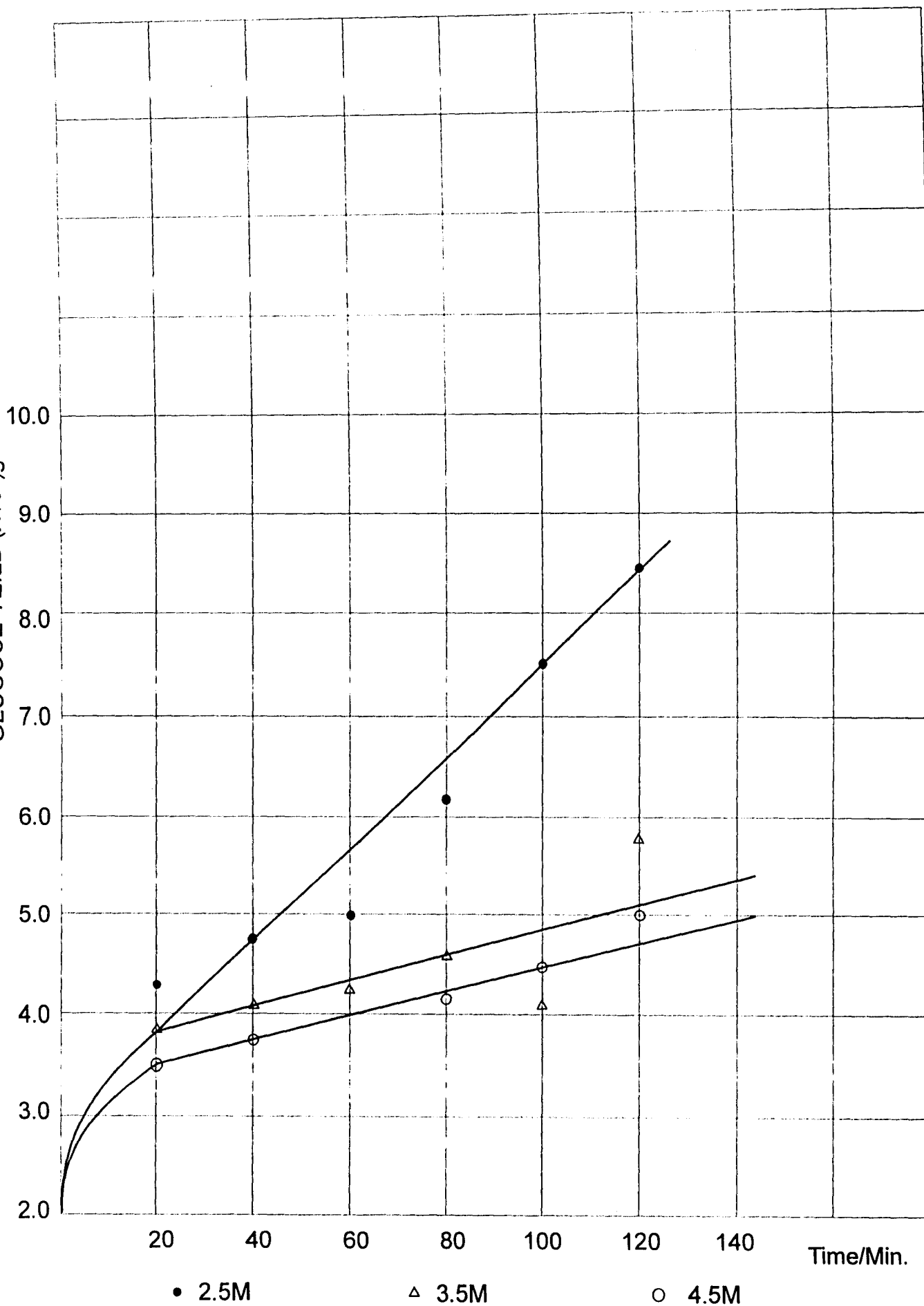


FIGURE 4.9b: PLOT OF GLUCOSE YIELD VS TIME AT 80°C FOR VARYING ACID CONCENTRATION.

CHAPTER FIVE

DISCUSSION OF RESULTS

The glucose standard calibration plot gives a linear graph from the origin, as can be seen in figure 4.1, which forms the basis for the subsequent glucose concentration at various temperature and medium concentrations.

The amount of glucose liberated as a function of time for the hydrolysis of saw just under alkaline and acid medium, and at various temperature of 65^oc to 80^oc are illustrated in figure 4.2_a and 4.3_a respectively. These figures show the effect of temperature at constant concentration of 0.4M NaOH and 2.5M H₂SO₄. An increase in glucose concentration was observed as temperature and time increases, hence, the concentration of glucose in an hydrolysis reaction is dependant on time and temperature.

Similarly, figures 4.2_b and 4.3_b depict the effect of medium concentration on hydrolysis at constant temperature of 80^oc. 0.4M – 0.6M NaOH and 2.5M - 4.5M H₂SO₄ were used and a decrease in glucose concentration was observed. This implies that, an increase in medium concentration reduces the amount of water molecules available for complete splitting of cellulose to take place, thereby resulting in to low production of glucose at a given temperature. Also, from the plot of glucose yield against time as shown in figure 4.6 (a and b) and 4.9(a and b) for alkaline and acid medium respectively, and at various temperatures, using 0.4M NaOH and 2.5M H₂SO₄, an increase in glucose yield was obtained as temperature, and time increases, though acid medium recorded a better glucose yield.

Hydrolysis of sawdust in both acid and alkaline medium proceeds with a first order kinetics as illustrated in figure 4.4 and figure 4.7 for alkaline and acid medium respectively. The rate constants estimated for acid and alkaline medium are tabulated below

Temperature °C	Rate constant (K) min ⁻¹ x 10 ⁻⁵	
	Acid	Alkaline
65	0.8	0.2
70	1.08	0.33
75	1.20	0.32
80	1.48	0.28

Analysis of the effect of temperature on reaction was further carried out using rate constant value obtained above.

From arrhenius law which basis in the plot of $\ln k$ vs. $1/T$ as illustrated figure 4.8 for alkaline and acid respectively. The activation energy was obtained to be 43.42 KJ/gmol for alkaline medium and 39.65 KJ/gmol for acid medium. This however, proves that alkaline catalysed hydrolysis encounter stronger energy barrier than acid catalysed reaction in the hydrolysis of sawdust. Therefore, a better medium for hydrolysis of sawdust with respect to this research work is 2.5M of sulphuric acid at a relatively high temperature of 80°C.

CHAPTER SIX

CONCLUSION

Based on the results obtained from this research work, D-glucose is present in substantial amount in sawdust.

The glucose concentration and yield depends on.

- Concentration of the medium
- Operating temperature for hydrolysis
- Duration of hydrolysis process.

Higher glucose yield is achieved with increase in temperature and time provided there is an appreciable degree of accuracy. The yield of glucose is higher under acid catalysed hydrolysis.

The rate of hydrolysis for both acid and alkaline medium follows a first order kinetic with activation energy of 39.65KJ/gmol and 43.42KJ/gmol for acid and alkaline reaction respectively, which are lower than the reported value of 142.53 KJ/gmol for alkaline hydrolysis of rice husk.

The hydrolysis reactions are stable at high temperature for low concentration medium in order to speed up the process, hence, 2.5M sulphuric acid medium at 80°C gives a better consideration.

RECOMMENDATION

The study on the hydrolysis of sawdust in acid and alkaline medium has been a tremendous success. It has proven that sawdust cellulose indeed contains a substantial amount of D-glucose which can be a great source of the energy supplement. Therefore, due to the high percentage of cellulose and its abundance it has become a necessity to carry out further research on increased yield and actual production of D-glucose through hydrolysis. This will definitely become a major source in future with increased resources utilization.

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APPENDIX ONE

1.0 THE SOMOGYI – NELSON PROCEDURE FOR QUANTITATIVE DETERMINATION OF REDUCING SUGAR

The basis of Somogyi-Nelson method for the determination of the reducing sugar quantitatively depends on the ability of the reducing saccharides to reduce Copper (II) ion to Copper (I) ion in both alkaline and acid medium.

Heating of sugar with an aldehyde group in either alkaline or acid solution will result to the breakdown of sugar into smaller units with reducing properties, which then reduces Copper (II) ion to red coloured Cuprous oxide which in turn reduces molybdate to blue coloured compound.

1.2 REAGENT PREPARATION

1.2.1 LOW ALKALINITY COPPER REAGENT

12 grams of Rochelle salt (Potassium Sodium tartrate) 24 grams of anhydrous Sodium Carbonate were dissolved in 250ml distilled water.

16grams of sodium hydrogen Carbonate was added to the solution. A solution of 144g anhydrous Sodium Sulphate in 500ml of water was boiled to expel the air, which is mixed to the former and made up to 800ml. This was labelled solution "A".

A Solution of 4 grams of Cupric Sulphate pentahydrate in 400ml of water was prepared. 150ml water containing dissolved 32 grams of anhydrous solution was boiled to expel the air. The former is mixed with the later and made up to 200ml. This was labelled "B".

Solution "A" and "B" were allowed to stand for one week and clear supernatant of solution of A and B were used in the ratio 4:1.

1.2.2 ARSENO MOLYBDATE REAGENT

21 ml of 98% Sulphuric acid was added to 25g of ammonium molybdate in 450ml of water followed by the addition of second solution containing 3 grams of Disodium hydrogen arsenate heptahydrate ($\text{Na}_2\text{HASO}_4 \cdot 7\text{H}_2\text{O}$) in 25ml of water, the resulting solutions were mixed for 24hrs at 37^oc and stored in brown bottle.

APPENDIX TWO

DETERMINATION OF CELLULOSE CONTENT IN ONE GRAM OF SAWDUST SAMPLE

In the experimental result, a cellulose of 3.138 percent was obtained using 10 grams of sawdust by mass as reference.

Therefore,

10 grams of sawdust contains 3.138g of cellulose

1 gram contains $\frac{1 \times 3.138}{10} = 0.3138$ grams

Similarly, the amount of D-glucose contained in 0.3138g of cellulose dissolved in 50ml of acid/alkaline solution will be

$$a_0 = \frac{0.3138}{50} = 6.28 \times 10^{-3} \text{g/ml.}$$

KINETICS OF FIRST ORDER EQUATION

The integral rate for first order reaction is given as:

$$\frac{-dC_A}{dt} = K C_A \text{ ----- (1)}$$

Rearranging and integrating equation (1)

$$\frac{dC_A}{C_A} = \int K dt \text{ ----- (2)}$$

Taking limits of integration from C_{A0} to C_A

$$\int_{C_{A0}}^{C_A} \ln C_A = -Kt$$

$$\ln \frac{C_A}{C_{A0}} = -Kt$$

$$\ln \frac{C_{A0}}{C_A} = Kt \text{ ----- (3)}$$

$$\text{But } C_A = C_{A0} - x$$

$$\therefore \ln \left(\frac{C_{A0}}{C_{A0} - x} \right) = Kt$$

hence

$$\ln \left(\frac{a_0}{a_0 - x} \right) = Kt \text{ ----- (4)}$$

From Equation (4)

X = Concentration of glucose produced from sawdust sample
due to hydrolysis

K = Reaction rate constant (min^{-1})

t = Time of Reaction (minute)

a_0 = Initial Cellulose concentration in sample

APPENDIX THREE

3.0 DETERMINATION OF ACTIVATION ENERGY [E_{ACT}] OF THE HYDROLYSIS RATE OF SAWDUST SAMPLE IN ACID AND ALKALINE MEDIA

3.1 ACTIVATION ENERGY OF ACID HYDROLYSIS:

Form figure 4.8 which is a plot of $\ln K$ vs $1/T$, a slope was obtained and the reaction rate was found to be well fitted by Arrhenius law:

$$K = K_0 \text{ or } \ln K_0 + (-E/RT)$$

Where K_0 = Frequency factor

E_{ACT} = Activation energy

R = Universal gas constant

Therefore, the slope of the plot of $\ln K$ vs $1/T$ gives $(-E/R)$.

$$- \text{slope} = -E/R$$

$$\text{Slope} = E/R$$

$$E_{ACT} = \text{Slope} \times R$$

$$\text{Slope} = 4769.23, R = 8.3143 \text{ KJ/kgmol}$$

$$E_{ACT} = \frac{4769.23 \times 8.3143}{1000}$$

$$E_{ACT} = 39.65 \text{ KJ/gmol.}$$

3.2 SIMILARLY, ACTIVATION ENERGY FOR ALKALINE HYDROLYSIS:

From the figure 4.5 which is a plot of $\ln K$ vs $1/T$ in alkaline medium. Following Arrhenius law

$$K = \ln K_0 + (-E/RT)$$

$$\text{The slope} = -E/R = -5222.22$$

$$-E/R = -5222.22$$

$$E_{ACT} = 5222.00 \times R$$

$$E_{ACT} = \frac{5222.22 \times 8.3143}{1000} = 43.42 \text{ KJ/gmol.}$$

APPENDIX FOUR

4.0 RESULTS

Table 4.1 shows the data for the standard calibration curve for Glucose concentration varying from 0.00 mol/ml to 10mol/ml of standard D-Glucose at 470nm.

Table 4.1 standard calibration Data

Glucose concentration ($\times 10^{-6}$ mol/ml)	Optical Density (Absorbance)
0.00	0.00
2.0	0.07
4.0	0.15
6.0	0.19
8.0	0.26
10.0	0.33

ALKALINE HYDROLYSIS RESULT

4.2 Effect of Temperature on Alkaline Hydrolysis of sawdust.

Table 4.2.1: Data for hydrolysis of sample at 65°c using 0.4mol NaOH solution for three (3) hours.

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
30	0.19	5.80
60	0.20	6.05
90	0.21	6.35
120	0.23	6.90
150	0.24	7.30
180	0.27	8.20

Table 4.2.2: Data for the hydrolysis of sample at 70^oc using 0.4mol NaOH solution for three (3) hours.

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
30	0.21	6.35
60	0.23	6.90
90	0.25	7.60
120	0.27	8.20
150	0.29	8.80
180	0.33	10.00

Table 4.2.3: Data for the hydrolysis of sample at 75^oc using 0.4mol NaOH solution for three (3) hours.

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
30	0.25	7.60
60	0.28	8.50
90	0.30	9.10
120	0.31	9.40
150	0.31	9:40
180	0.34	10.30

Table 4.2.4: Data for the hydrolysis of sample at 80°C using 0.4mol NaOH solution for three (3) hours.

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
30	0.27	8.20
60	0.29	8.80
90	0.31	9.40
120	0.32	9.70
150	0.33	10.00
180	0.36	10.90

Effect of Alkali Concentration on the Hydrolysis of Sawdust at 80°C

Table 4.2.5: Data for the hydrolysis of sample at 80°C using 0.5mol NaOH solution for three (3) hours

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
30	0.26	7.90
60	0.27	8.20
90	0.29	8.80
120	0.32	9.70
150	0.32	9.70
180	0.35	10.60

Table 4.2.6: Data for the hydrolysis of sample at 80°C using 0.6mol NaOH solution for three (3) hours.

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
30	0.24	7.30
60	0.26	7.90
90	0.27	8.20
120	0.29	8.80
150	0.31	9.40
180	0.32	9.70

4.3 ACID HYDROLYSIS RESULT

Data below shows the effect of temperature on acid hydrolysis of sawdust at constant concentration of 2.5M sulphuric Acid.

Table 4.3.1: Data for hydrolysis of sample at 65°C using 2.5M sulphuric Acid

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
20	0.22	6.70
40	0.24	7.30
60	0.30	9.10
80	0.32	9.70
100	0.32	9.70
120	0.41	12.40

Table 4.3.2: Data showing the hydrolysis of sample at 70°C using 2.5M of sulphuric Acid.

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
20	0.23	6.90
40	0.26	7.90
60	0.33	10.00
80	0.33	10.00
100	0.42	12.80
120	0.45	13.70

Table 4.3.3: Data showing the hydrolysis of sample at 70°C using 2.5M of sulphuric Acid.

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
20	0.26	7.90
40	0.28	8.50
60	0.32	9.70
80	0.37	11.20
100	0.46	14.00
120	0.48	14.60

Table 4.3.4: Data showing the hydrolysis of sample at 80°C using 2.5M of sulphuric Acid

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
20	0.28	8.50
40	0.31	9.40
60	0.33	10.00
80	0.41	12.40
100	0.51	15.50
120	0.55	16.80

Effect of varying Concentration of Acid at constant Temperature of 80°C

Table 4.3.5: Data showing the sample Hydrolysis at 80°C using 3.5M of Acid

Time (Min)	Optical Density (Absorbance)	Glucose concentration ($\times 10^{-6}$ mol/ml)
20	2.50	7.60
40	0.27	8.20
60	0.28	8.50
80	0.31	9.40
100	0.27	8.20
120	0.38	11.50

Table 4.3.6: Data showing the sample Hydrolysis at 80⁰c using 4.5M of sulphuric Acid

Time (Min)	Optical Density (Absorbance)	Glucose concentration (x10 ⁻⁶ mol/ml)
20	0.23	6.90
40	0.25	7.60
60	0.28	8.50
80	0.28	8.50
100	0.30	9.10
120	0.33	10.00

4.3 Kinetic Analysis of Alkaline Hydrolysis of Sawdust

Table 4.4.1 to 4.4.4 Show the relationship $\ln[a_0/(a_0 - x)]$ with change in time for temperatures ranging from 65⁰c to 80⁰c at a constant concentration of 0.4mol NaOH, this are illustrated in figure 4.4 similarly, tables 4.4.4 to 4.4.6 show the relationship of $\ln[a_0/(a_0-x)]$ with changes in time for concentration ranging from 0.4mol to 0.6mol NaOH at constant temperature of 80⁰c. This is also illustrated in figure 4.

Table 4.4.1: Data for plotting $\ln[a_0/(a_0-x)]$ versus time for determination of rate constant at 65⁰c using 0.4mol NaoH solution with an initial cellulose concentration (a₀) of 6.28 x 10⁻³ g/mol

Time (Min)	Glucose concentration (x10 ⁻⁶ mol/ml)	$\ln[a_0/(a_0-x)]$ (x10 ⁻³)
20	5.80	0.92
40	6.05	0.96
60	6.35	1.01
80	6.90	1.10
100	7.30	1.16
120	8.20	1.31

Table 4.4.2: Data for plotting $\ln[a_0/(a_0-x)]$ versus time for determination of rate constant at 70°C using 0.4mol NaOH solution with initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0-x)]$ ($\times 10^{-3}$)
20	6.35	1.01
40	6.90	1.10
60	7.60	1.21
80	8.20	1.31
100	8.80	1.40
120	10.00	1.59

Table 4.4.3: Data for plotting $\ln[a_0/(a_0-x)]$ versus for determination of rate constant at 75°C using 0.4mol NaOH solution with an initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0-x)]$ ($\times 10^{-3}$)
20	7.60	1.21
40	8.50	1.35
60	9.10	1.45
80	9.40	1.50
100	9.40	1.50
120	10.30	1.64

Table 4.4.4: Data for plotting $\ln[a_0/(a_0-x)]$ versus for determination of rate constant at 80°c using 0.4mol NaOH solution with an initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0-x)]$ ($\times 10^{-3}$)
20	8.20	1.31
40	8.80	1.40
60	9.40	1.50
80	9.70	1.55
100	10.00	1.59
120	10.90	1.74

Table 4.4.5: Data for plotting $\ln[a_0/(a_0-x)]$ versus for determination of rate constant at 80°c using 0.5mol NaOH solution with an initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0-x)]$ ($\times 10^{-3}$)
20	7.90	1.26
40	8.20	1.31
60	8.80	1.40
80	9.70	1.55
100	9.70	1.55
120	10.60	1.69

Table 4.4.6: Data for plotting $\ln[a_0/(a_0-x)]$ versus for determination of rate constant at 80⁰c using 0.6mol NaOH solution with an initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0-x)]$ ($\times 10^{-3}$)
20	7.30	1.16
40	7.90	1.26
60	8.20	1.31
80	8.80	1.40
100	9.40	1.50
120	9.70	1.55

4.4 Determination of Activation Energy using Temperature Dependency

Table 4.5.1 shows the relationship between $\ln k$ and $(1/T)$ for temperature ranging from 65⁰c to 80⁰c and is illustrated in figure 4.5 where the slope are used to determine the activation energy of the Alkali hydrolysis reaction.

Table 4.5.1: Data for plotting the curve of $\ln k$ against $(1/T)$ for the sample which were obtained from the graphs of 65⁰c, 70⁰c, 75⁰c and 80⁰c as illustrated in figure 4.5

T (°c)	K ($\times 10^{-5}$ /min)	$\ln k$	T (°c+273)K	1/T ($\times 10^{-3}$)
65	0.2	-13.22	338	2.96
70	0.33	-12.62	343	2.92
75	0.32	-12.65	348	2.87
80	0.28	-12.79	353	2.83

4.6 Conversion and Yield of Glucose from Hydrolysis Reaction in an Alkaline Medium

Table 4.6.1 to 4.6.4 shows the percentage conversion and glucose yield in the hydrolysis reaction obtained at various temperature range of 65°C to 80°C and tables 4.6.4 to 4.6.6 shows for various concentrations ranging from 0.4mol to 0.6mol NaOH. This are illustrated in figure 4.6(a) and 4.6(b)

Table 4.6.1: Conversion and yield at 65°C using 0.4mol NaOH after three hours with initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	Conversion (%)	Yield (g) $\times 10^{-4}$
30	5.80	0.092	2.89
60	6.05	0.096	3.01
90	6.35	0.1011	3.17
120	6.90	0.1099	3.45
150	7.30	0.1162	3.65
180	8.20	0.1306	4.10

Table 4.6.2: Conversion and yield at 70°C using 0.4mol NaOH after three hours with initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	Conversion (%)	Yield g $\times 10^{-4}$
30	6.35	0.1011	3.17
60	6.90	0.1099	3.45
90	7.60	0.1210	3.80
120	8.20	0.1306	4.10
150	8.80	0.1401	4.40
180	10.00	0.1592	5.00

Table 4.6.3: Conversion and yield at 75°c using 0.4mol NaOH after three hours with initial cellulose concentration of 6.29×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	Conversion (%)	Yield (g) $\times 10^{-4}$
30	7.60	0.1210	3.80
60	8.50	0.1354	4.25
90	9.10	0.1450	4.55
120	9.40	0.1497	4.70
150	9.40	0.1497	4.70
180	10.30	0.1640	5.15

Table 4.6.4: Conversion and yield at 80°c using 0.4mol NaOH after three hours with initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	Conversion (%)	Yield (g) $\times 10^{-4}$
30	8.20	0.1306	4.10
60	8.80	0.1401	4.40
90	9.40	0.1497	4.70
120	9.70	0.1545	4.85
150	10.00	0.1592	5.00
180	10.90	0.1736	5.45

Table 4.6.5: Conversion and yield at 80^oc using 0.5mol NaOH after three hours with initial cellulose concentration (a₀) of 6.28x10⁻³g/mol

Time (Min)	Glucose concentration (x10 ⁻⁶ mol/ml)	Conversion (%)	Yield (g) x 10 ⁻⁴
30	7.9	0.1258	3.95
60	8.20	0.1306	4.10
90	8.80	0.1401	4.40
120	9.70	0.1545	4.85
150	9.70	0.1545	4.85
180	10.60	0.1688	5.30

Table 4.6.6: Conversion and yield at 80^oc using 0.6mol NaoH after three hours with initial cellulose concentration (a₀) of 6.28x10⁻³g/mol

Time (Min)	Glucose concentration (x10 ⁻⁶ mol/ml)	Conversion (%)	Yield (g) x 10 ⁻⁴
30	7.30	0.1162	3.65
60	7.80	0.1258	3.95
90	8.20	0.1306	4.10
120	8.80	0.1401	4.40
150	9.40	0.1497	4.70
180	9.70	0.1547	4.85

4.7 Kinetic Analysis of Acid Hydrolysis of Sawdust

Table 4.7.1 to 4.7.4 shows the relationship of $\ln[a_0/(a_0-x)]$ with time for temperatures ranges of 65⁰c to 80⁰c at constant acid (sulphuric acid) concentration of 2.5m. this are illustrated in figure 4.7. similarly, table 4.7.4 to 4.7.6 shows the relationship of in $[a_0/(a_0-x)]$ with time for varying concentration ranging from 2.5M to 4.5M sulphuric acid at constant temperature of 80⁰c. This are also illustrated in figures.

Table 4.7.1: Data for plotting $\ln[a_0/(a_0-x)]$ versus time for determination of rate constant at 65⁰c using 2.5M sulphuric Acid solution with an initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0-x)]$ ($\times 10^{-3}$)
20	6.70	1.07
40	7.30	1.16
60	9.10	1.45
80	9.70	1.55
100	9.70	1.55
120	12.40	1.98

Table 4.7.2: Data for plotting $\ln[a_0/(a_0-x)]$ versus time for determination of rate constant at 70°C using 2.5M sulphuric Acid solution with an initial cellulose concentration of 6.28×10^{-3} g/mol.

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0-x)]$ ($\times 10^{-3}$)
20	6.90	1.10
40	7.90	1.26
60	10.00	1.59
80	10.00	1.59
100	12.80	2.04
120	13.70	2.18

Table 4.7.3: Data for plotting $\ln[a_0/(a_0-x)]$ versus time for determination of rate constant at 75°C using 2.5M sulphuric Acid solution with an initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0-x)]$ ($\times 10^{-3}$)
20	7.90	1.26
40	8.50	1.35
60	9.70	1.55
80	11.20	1.79
100	14.00	2.23
120	14.60	2.33

Table 4.7.4: Data for plotting $\ln[a_0/(a_0 - x)]$ versus for determination of rate constant at 80°C using 2.5M sulphuric Acid solution with an initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0 - x)]$ ($\times 10^{-3}$)
20	8.50	1.35
40	9.40	1.50
60	10.00	1.59
80	12.40	1.98
100	15.50	2.47
120	16.80	2.68

Table 4.7.5: Data for plotting $\ln[a_0/(a_0 - x)]$ versus time for determination of rate constant at 80°C using 3.5M sulphuric Acid solution with an initial cellulose concentration of 6.28×10^{-3} g/mol.

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	$\ln[a_0/(a_0 - x)]$ ($\times 10^{-3}$)
20	6.90	1.10
40	7.60	1.21
60	8.50	1.35
80	8.50	1.35
100	9.10	1.45
120	10.00	1.59

4.8 Determination of Activation Energy using Temperature Dependency

Table 4.8.1 shows the relationship ranging $\ln k$ and $1/T$ for temperature ranging from 65°C to 80°C and are illustrated in figure 4.8 where the slope are used to determine the activation energy of Acid hydrolysis reaction

Table 4.8.1: Data for plotting the curve of $\ln k$ against $1/T$ for the sample, which were obtained from the graphs of 65°C , 70°C , 75°C and 80°C as illustrated in figure 4.8

$T/^{\circ}\text{C}$	K ($\times 10^{-5}/\text{min}$)	$\ln k$	T ($^{\circ}\text{C}+273$) K	$1/T$ ($\times 10^{-3}$)
65	0.8	-11.74	338	2.96
70	1.08	-11.44	343	2.92
75	1.20	-11.33	348	2.87
80	1.48	-11.12	353	2.83

4.9 Conversion and Yield of Glucose from initial Cellulose Concentration of $6.28 \times 10^{-3} \text{g/mol}$ in an Acid medium.

Table 4.9.1 to 4.9.4 shows the percentage conversion of cellulose of cellulose and yield of glucose in the acid hydrolysis at various temperature ranges of 65°C to 80°C . Also, table 4.9.4 to 4.9.6 shows the effect of concentration on the conversion and yield. This are illustrated in figures 4.9(a) and 4.9(b).

Table 4.9.1: Conversion and yield of glucose at 65°C using 2.5M sulphuric Acid after 120minutes with an initial cellulose concentration of 6.28×10^{-3} g/mol

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	Conversion (%)	Yield (g) $\times 10^{-4}$
20	6.70	0.1067	3.35
40	7.30	0.1162	3.65
60	9.10	0.1354	4.25
80	9.70	0.1545	4.85
100	9.70	0.1545	4.85
120	12.40	0.1975	6.20

Table 4.9.2: Conversion and yield of glucose at 70°C using 2.5M sulphuric Acid after 120minutes with an initial cellulose concentration of 6.28×10^{-3} g/mol.

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	Conversion (%)	Yield (g) $\times 10^{-4}$
20	6.90	0.1099	3.45
40	7.90	0.1592	3.95
60	10.00	0.1592	5.00
80	10.00	0.1592	5.00
100	12.80	0.2038	6.40
120	13.70	0.2182	6.85

Table 4.9.3: Conversion and yield of glucose at 75°C using 2.5M sulphuric Acid after 120 minute with an initial cellulose concentration of 6.28×10^{-3} g/mol.

Time (Min)	Glucose concentration ($\times 10^{-6}$ g/mol)	Conversion (%)	Yield (g) $\times 10^{-4}$
20	6.70	0.1250	3.95
40	8.50	0.1354	4.25
60	9.70	0.1545	4.85
80	11.20	0.1783	5.60
100	14.00	0.2229	7.00
120	14.60	0.2325	7.30

Table 4.94: Conversion and yield of glucose at 80°C using 2.5M sulphuric Acid after 120minutes with an initial cellulose concentration of 6.28×10^{-3} g/mol.

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	Conversion (%)	Yield (g) $\times 10^{-4}$
20	8.50	0.1354	4.25
40	9.40	0.1497	4.70
60	10.00	0.1592	5.00
80	12.40	0.1975	6.20
100	15.50	0.2468	7.75
120	16.80	0.2752	8.40

Table 4.9.5: Conversion and yield of glucose at 80°C using 3.5M sulphuric Acid after 120minutes with an initial cellulose concentration of 6.28×10^{-3} g/mol.

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	Conversion (%)	Yield (g) $\times 10^{-4}$
20	7.60	0.1210	3.80
40	8.20	0.1306	4.10
60	8.50	0.1354	4.25
80	9.40	0.1497	4.70
100	8.20	0.1306	4.10
120	11.50	0.1831	5.75

Table 4.9.6: Conversion and yield of glucose at 80°C using 4.5M sulphuric Acid after 120minutes with an initial concentration of 6.28×10^{-3} g/mol.

Time (Min)	Glucose concentration ($\times 10^{-6}$ mol/ml)	Conversion (%)	Yield (g) $\times 10^{-4}$
20	6.90	0.1099	3.45
40	7.60	0.1210	3.80
60	8.50	0.1354	4.25
80	8.50	0.1354	4.25
100	9.10	0.1450	4.55
120	10.00	0.1592	5.00