## THE EFFECT OF TEMPERATURE ON HYDROLYSIS OF CELLULOSE (SAW-DUST)

BY

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#### CERTIFICATION

This project report on the topic of the effect of temperature on hydrolysis of cellulose (saw-dust) using hydrochloric acid was written, compiled and submitted by Nwanze Cynthia to the department of Chemical Engineering of Caritas University Amorji Nike Enugu with the registration number CHE/2007/121 in partial fulfillment of the requirement for the award of a Bachelor of engineering (B.Eng) degree in Chemical Engineering has been approved by the undersigned

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#### **DEDICATION**

I dedicate this report to God Almighty for his love, mercy and kindness upon my life, which if not for his mercy I would not have made it to this point and to my lovely parents Nze and Lolo Nerius Nwanze, for their help and support that led to the success of this project report.

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#### ABSTRACT

The effect of concentration of hydrochloric acid on hydrolysis of cellulose (sawdust) to glucose was studied on this research project and the steps obtained to achieve this project involved treatment of saw-dust (cellulose) with different concentrations of the acid at constant temperature of  $80^{\circ}C$  (350k) for 30mins. This was followed by glucose analysis, some analysis or experiments were done on acid hydrolysis in order to study the effect of (HCL) acid on the hydrolysis of cellulose to glucose. The process used in this hydrolysis was acid hydrolysis in which HCL acid was used at constant temperature of  $80^{\circ}C$  and the saw-dust used [was obtained by grinding wood with saw] was weighed and mixed with water . Secondly, during this analysis/experiment, it was observed that hydrochloric acid hydrolyzed well from the readings gotten from each result that was carried out during the analysis. Then lastly, glucose analysis was carried out to determine the absorbance and glucose concentration. It was noticed that the best concentration of HCL acid during hydrolysis yields glucose concentration of 0.127g or 1.270%.

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#### **CHAPTER ONE**

#### **1.1 Introduction**

Cellulose is the name given to a long chain of atoms consisting of carbon, hydrogen and oxygen arranged in a particular manner it is a naturally occurring polymeric material containing thousands of glucose-like rings each of which contain three alcoholic OH groups. Its general each of which contain three alcoholic OH groups. Its general formula is represented as  $(C_6H_{10}O_5)n$ . the oh-groups present in cellulose can be esterifies or etherified, the most important cellulose derivatives are the esters.

Cellulose is found in nature in almost all forms of plant life's, and especially in cotton and wood. A cellulose molecule is made up of large number of glucose units linked together by oxygen atom. Each glucose unit contains three(3) hydroxyl groups, the hydroxyl groups present at carbon-6 is primary, while two other hydroxyl are secondary. Cellulose is the most abundant organic chemical on earth more than 50% of the carbon is plants occurs in the cellulose of stems and leave wood is largely cellulose, and cotton is more than 90% cellulose. It is a major constituent of plant cell walls that provides strength and rigidity and presents the swelling of the cell and rupture of the palms membrane that might result when osmotic conditions favor water entry into the cell. Cellulose is a fibrous, ought, water-insoluble substances, it can be seen in cell walls of plants, particularly in stalks, stems, trunks and all woody portions of the plant.

Cellulose is polymorphic, i.e there are number of different crystalline forms that reflect the history of the molecule. It is almost impossible to describe cellulose chemistry and biochemistry without referring to those different forms. Cellulose are gotten from cellulose, cellulose is also found in protozoa in the gut of insects such as termites. Very strong acids can also degrade cellulose, the human digestive system has little effect on cellulose. The world cellulose means  $\beta$ -1, 4- D glucan, regardless of source because of the importance of cellulose and difficulty in unraveling its secrets regarding structure, biosynthesis, chemistry, and other aspects, several societies are dedicated to cellulose, lignin, and related molecues.

#### **1.2 Definition of Terms**

Hydrolysis: means hydro (water) lysis (splitting) or breaking down of a chemical bond by the addition of water (H<sub>2</sub>O), it is by the introduction of the elements that make up water hydrogen and oxygen. The reactions are more complicated than just adding water to a compound, but by the end of a hydrolysis reaction, there will be two more hydrogen's and one more oxygen shared between the products, than there were before the reaction occurred.

Hydrolysis of cellulose therefore is the process of breaking down the glucosidic bonds that holds the glucose basic units together to term a large cellulose molecule, it is a term used to describe the overall process where cellulsose is converted into various sweeteners.

**Sugar:** is the generalized name for a class of chemically related sweet – flavored substances, most of which are used as food. They are carbohydrates, composed of carbon, hydrogen and oxygen. There are various sugar derived from different sources. Simple sugars are called monosaccharide's and include glucose cellos known as dextrose, fructose and galactose. The table or granulated

sugar most customarily used as food is sucrose, a disaccharide other disacclarides include maltose and lacoose. Chemicallydifferent substances may also have a sweet taste, but are not classified as sugar but as artificial sweeteners.

#### **1.3 STATEMENT OF THE PROBLEM**

The new government policies and economy through low quality products has imposed motivated researchers to explore the numerous domestic, industrial and economic importance of the Nigeria's major waste product which is "cellulose" which forms the bedrock of this project.

Sugar is a high demand for both domestic and industrial applications on daily basis in homes, small and medium scale industries etc this is why Nigeria government spends huge sums of money on importation of sugar and sugar products to meet the demand of citizens. Among the many processes of sugar production, is acid hydrolysis of (cellulose) has proved to be a process which encourages the production of high quality with minimum skill and

materials. This work is therefore an effort to encourage industrialist, researchers, and students to carry out more intensive studies on production of sugar from cellulose for production of sugar and enhanced economic resources for the nation.

#### **1.4 SCOPE AND OF STUDY LIMITATIONS**

This study is aimed at estimating the impact of some areas hindering the subject/project matter (disadvantages) the cellulose.

It is obvious that cellulose materials have been used, including newspaper, carboard, cotton, straw, sawdust, hemp and corncob. Monticell was insulated with a form of cellulose. Modern cellulose insulation, made with recycled newspaper using grinding and dust removing machines and adding a fire retardant, began in the 1950s and came into general use in the U.S during the 1970s.

The R value Rule" placed clear limitations on the claims that manufacturing and marketing firms can make about their product, then also the effect of regulations by the CPSC put most of the small producers of cellulose insulation out of business. The costs

incurred by increasing fire testing made cellulose more expensive and the bad publicity helped decrease demand.

Cellulose also has a few disadvantages. As compared to other insulation options, the R-value of 3.6 to 3.8 per inch is good but not the best. Many spray foams utilizes an environmentally harmful blowing agent, such as enovate HFC, cellulose does not.

**Dust:** Cellulose contains some small particles which can be blown into the house through inadequate seals around fixtures or minute holes.

**Wet-spray drying time:** We-spray provides the moisture requires a longer drying time before the drywall/sheet-rock is applied to a newly insulation.

#### **1.5 OBJECTIVES**

The principal aim of undertaking this project is to determine the effect of concentration of acid on the yield of glucose production by acid hydrolysis of cellulose.

Hydrolysis of cellulose into glucose using different concentration of hydrochloric acid.

Calculating and quantifying the yield of glucose from hydrolysis of cellulose using HCL acid.

In the experiment, cellulose from variety of sources will be subjected to depolymerization conditions.

#### **CHAPTER TWO**

#### **2.1 LITERATURE REVIEW**

Cellulose is an organic compound with the formula  $(C_6H_{10}O_5)n$ , a polysaccharide consisting of a linear chain of several hundred to over ten thousand  $\beta$  (1-4) linked D-glucose units. It is the structural component of the primary cell wall of green plants, many forms of algae and the oomycetes. Some species of bacteria secrete it to form biofilms. Cellulose is the most common organic compound on earth. For industrial use, cellulose today is mainly used to produce paperboard and paper; to a smaller extent it is converted into a wide variety of derivative products such as cellophane and range. Converting cellulose from energy corps into biofuels such as cellulose ethanol is under investigation as an alternative fuel source, some animals, particularly ruminants and termites, can digest cellulose with the help of symbiotic micro-organisms that lives in there guts. Humans can also digest cellulose to some extents, however it mainly acts as a hydrophilic bulking agent for feces and is often referred to as "dietary fiber",

I laboratory isolation of cellulose is used as the stationary phase for thin layer chromatography cellulose is further used to make highly absorbent sponges. Cellulose fibers are also used in liquid filtration. It's insulation male from recycled paper is becoming popular as an environmentally preferable material for building insulation. It can be treated with boric acid as a fire retardant.

#### 2.2 HISTORY

Cellulose was discovered in 1838 by the French chemist anselme payen, who isolated it from plant matter and determined its chemical formula cellulose was used to produce the first successful thermoplastic polymer, celluloid, by Hyatt manufacturing company in 1870. Hermann Staudinger determined the polymer structure of cellulose in 1920. The compound was first chemically synthesized (without the use of any biologically derived enzymes) in 1992, by Kobayashi and shoda.

Discovery: the word "cellulose" was coined just 37 years before the American chemical society was formed. It appeared first in 1839 in

the French academy's assessment on anselme payen's research on "ligneous matter", the then-current term for the combination of lignin and cellulose that forms the woody cell walls of trees and other plants. In 1838 payen reported that ligneous materials. One of these, the French chemist declared, had the same chemical composition as starch, but differed in structure and properties.

#### 2.3 PRODUCTS

The kraft process is used to separate cellulose from lignin; another major component of plant matter cellulose is the major constituent of paper, paper board, and card stock and of textiles made from cotton, linen, and other plant fibers.

Cellulose can be converted into cellophane, a thin transparent film, and into rayon, an important fiber that has been used for textiles since the beginning of the 20thcentury. Both cellophane and rayon are known as "regenerated cellulose fibers", they are identical to cellulose in chemical structure and are usually made from

dissolving pulp via viscose. A more recent method which is also friendly to the environment to produce a form of rayon is the lyoceu process. Cellulose is the raw material in the manufacture of nitrocellulose (cellulose nitrate) which is used in smokeless gunpowder and as the base material for celluloid used for photographic and movie films until the mid-1930s.

Cellulose is used to make water-soluble adhesives and binders such as methylcellulose and carbozymethyl cellulose, which are used in wallpaper paste. Microcystouline cellulose and powdered cellulose are used as inactive fillers in tablets and as thickeners and stabilizers in processed foods. Cellulose powder is for example used in kraft's parmesan cheese to prevent caking inside the tube.

Cellulose consists of crystalline and amorphous regions. By treating it with strong acid, the amorphous regions can be broken up, thereby producing nanocrystalline cellulose, a novel material with many desirable properties. Recently, nanocrystalline cellulose was used as the filler phase in bio-based polymer matrices to produce nanocomposites with superior thermal and mechanical properties.



Fig 4.2: Cellulose

#### 2.4 CELLULOSE SOURCE AND ENERGY STORE OF CROPS.

The major combustible component of non-food energy crops is cellulose, with lignin second, non-food energy crops are more efficient than dibble energy crops (which have a large starch component), but still compete with food crops for agricultural land and water resources. Typical non-food energy crops include industrial hemp (though outlawed in some countries), switch grass, miscanthus, salix (willow), and populous (poplar) species. Some bacteria can convert cellulose into ethanol which can then be used as a fuel.

In photosynthesis, crops use light energy to produce glucose from carbondioxide. The glucose is stored mainly in the form of cellulose granules, in plastids such as chloroplasts and especially anyloplasts. Glucose is soluble in water hydrophilic, binds much

water and the takes much space. Glucose in the form of starch on the other hand, is not soluble and can be stored much more compactly.

Cellulose for industrial conversion comes from wood and scores of minor sources such as kenaf paper and rayon are now made mostly from wood pulp, cotton linters (short fibers are used to spin yams) are now used in high quality writing and currency papers.

Cellulose forms very tightly packed crystallites, these crystals are sometimes so tight that neither water nor enzymes can penetrate them; cellulose consists of two cellulose molecules, crystalline and amorphous cellulose. The crystalline cellulose is insoluble because of the inability of water to penetrate cellulose, on the other hand amorphous cellulose allows the penetration of endogluconase, another subgroup of cellulose that catalyzed the hydrolysis of internal bonds. The natural consequences of this difference in the crystalline structure is that the hydrolysis rate is much faster for amorphous cellulose than crystalline cellulose.

Some other cellulose comes from the hairs (trichomes) on seeds examples; cotton, kapo and milk weed. A commercial bacterial

cellulose produce (cellulon) was introduced by weyer haeuser for use in foods, the product is called primacel and is available from Nutrasweet kelco. Now as of reconnect, cellulose from sugar bet pulp and as a fat substitute.

#### 2.5 STRUCTURE AND PROPERTIES

Cellulose has no taste, is odorless, is hydrophilic with the contact angle of 20-30°, is insoluble in water and most organic solvents, is chiral and is biodegradable it can be broken down chemically into its glucose units by treating it with concentated acids at high temperature.

Cellulose is derived from D-glucose units, which condense through  $\beta$  (1-4) – glycosidic bonds. This linkage motif contrasts with that for  $\alpha$  (1-4) – glycosidic bonds present in starch, glycogen, and other carbohydrates cellulose is a straight chain polymer: unlike starch, no coiling or branching occurs, and the molecule adopts an extended and rather stiff rod-like conformation, aided by the equatorial conformation of the glucose from one chain form hydrogen bonds with oxygen atoms on the same or on a neighbor

chain, holding the chains firmly together side by side and forming microfibrils with high tensile strength. This strength is important in cell walls, where the microfibrils are meshed into a carbohydrate matrix, conferring rigidity to plant cells.



Fig:4.3 Strand of Cellulose.

A strand of cellulose (conformation 1ἀ), showing the hydrogen bonds (dashed) within and between cellulose molecules. Compared to starch, cellulose is also much more crystalline whereas starch undergoes a crystalline to amorphous transition when heated beyond 60-70°C in water (as in cooking),cellulose requires a temperature of 320°C and pressure of 25Mpa to become amorphous in water. Several different crystalline structures of cellulose are known, corresponding to the location of hydrogen bonds between and within strands. Natural cellulose is cellulose i, with structures iἁ and iβ, cellulose produced by bacteria and algae enriched in iἁ while cellulose of higher plants consists mainly of  $i\beta$ . Cellulose in regenerated cellulose fibers is cellulose ii. The conversion of cellulose i to cellulose ii is irreversible, suggesting that cellulose 1 is metastable and cellulose ii is stable with various chemical treatments it is possible to produce the structures cellulose iii and cellulose iv.

Many properties of cellulose depend on its chain length or degree of polymerization, the number of glucose units that make up one polymer molecule. Cellulose from wood pulp has typical chain lengths between 300 and 1700 units; cotton and other plant fibers as well as bacterial cellulose have chain lengths ranging from 800 to 10,000 units. Molecules with very sall chain length resulting from the breakdown of cellulose are known as cellodextrins; in contrast to long-chain cellulose, cellodextrins are typically soluble in water and organic solvents.

Plant derived cellulose is usually found in a mixture with hemicelluloses, lignin, pectin and other substances, while microbial cellulose is quite pure, has a much higher water content, and consists of long chains.

Cellulose is soluble in cupriethylenediamine (CED), cadmiumethylenediamine (cadoxen), N-methylmorpholien N-oxide and lithium chloride/dimethylformamide middle. This is used n the production of regenerated celluloses (such as viscose and cellophane) from dissolving pulp.



Fig. 4.4: Triple strand of Cellulose

A triple strand of cellulose showing the hydrogen bonds (cyan lines) between glucose strands.

Assaying a cellulose- containing material: given a cellulosecontaining material, the carbohydrate portion that does not dissolve in a 17.5% solution of sodium hydroxide at 20°C is a cellulose, which is true cellulose. Acidification of the extract precipitates  $\beta$ cellulose. The portion tat dissolves in base but does not precipitate with acid is y cellulose.

Cellulose can be assayed using a method described by updegraff in 1969, where the fiber is dissolved in acetic and nitric acid to remove lignin, hemicelluloses, and xylosans. The resulting cellulose is allowed to react with anthrone in sulfuric acid, the resulting coloured compound is assayed spectrophotometrically at a wavelength of approximately 635mm. in addition, cellulose is represented by the difference between acid detergent fiber (ADF) and acid detergent lignin (ADL).

#### 2.6 **BIOSYNTHESIS**

In vascular plants cellulose is synthesized at the plasma membrane by rosette terminal complexes (RTCs). The RTCs are hexameric protein structures approximately 25nm in diameter, that contain the cellulose synthase enzymes that synthesise the individual cellulose chains. Each RTC flats in the cell's plasma membrane and "spins" a microfibril into the cell wall.

RTCs contain at least three different cellulose syntheses, encoded by lesA genes, in an unknown stoichiometry. Separate sets of genes are involved in primary and secondary cell wall biosynthesis.

Cellulose synthesis requires chain initiation and elongation, and the two processes are separate, CESA glucosyl transferase initiates cellulose polymerization using a steroid primer, sitosterol-beta-

glucosides, and UDP-glucose. Cellulose syntheses utilizes UDP-Dglucose precursors to elongate the growing cellulose chain. A cellulose may function to cleave the primer from the mature chain.

The concept of cellulose being synthesized by living organisms about "an acetic ferment which forms cellulose referring to the generation of cellulose by the gram negative rod, acetobacter xylinum, (Wikipedia,2013). Over the years, this microbe has led to the understanding of the structure and biosynthesis of cellulose. One of the earliest electrol microscopy studies of microbial cellulose was made in 1949 by Kurt muhlethaler. This led the way to numerous investigations of acetobacter in the 1960's by J. Ross Colvin and colleagues from Canada. (Wikipedia,2013)

In the late 1950's reclofsen proposed the concept that cellulose microfibrils must "grow" by tips growth from enzyme complexes. (Wikipedia,2013). In 1964, Reginald preston proposed the "ordered granule hypothesis" largely theoretical and based on the emerging freeze fracture technology at that time. It was not until 1976 that R. Malcolm Brown, Jr. and colleagues, then from the university of North Carolina at chapel Hill, discovered ordered membrane

associated aggregates associated with ends of developing cellulose microfibrils. This discovery was made in the green alga, oocystis apiculate. They named these putative enzyme complexes, TCs or "terminal complexes".

Isolation and purification of cellulose syntheses has been very difficult. The first breakthrough in this area came from purification of a acetobacer cellulose synthase from the brown lab in 1989. Identification of catalytic sub-unit of bacterial synthase by azido-UDP-glucose binding led to the first sequencing of a cellulose synthase by the Brown laboratory in 1990. Cellulose synthase associated proteins involved in accelerating the in vitro reactions were discovered by the Benziman laboratory in Israel. The first in vitro synthesis of cellulose came from work in the Brown laboratory (1993), and since this time, cellulose and callose biosynthesis have been physically separated; however, the purification of vascular plant cellulose synthase and its identification remain to this day as one of the mot difficult problems in plant biochemistry. (Young, 1986).

The genes for bacterial celluloses synthase were subjected to hydrophobic cluster analysis by a joint collaboration of the brown lab in Autin and the henrissat lab in Grenoble in 199s. using this information, delmer and her colleagues identified a cellulose synthase gene from cotton in 1996. Williamson and his lab in Australia also identified the gene for cellulose synthase in Arabidopsis in 1999. In the brown lab, the gene for cotton cellulose synthase was independently cloned and sequened. Then the DNA from this sequence was inserted into Escherichia coli which was coaxed into producing a polypeptide corresponding to the introduced gene. This polypeptide was extracted and purified and polyclonal antibodies produced to the recombinant cellulose sythnase catalytic subunit. In collaboration with the laboratory of Dr. Takao into in Kyoto, and using freeze fracture labeling, the two labs were able to prove unequivocally that the rosette subunit to cellulose synthase.

On a more novel approach, perfectly normal cellulose I has been synthesized synthetically in the brown lab (1994) using technology originally developed by Dr. kobayashi of Kyoto university in 1991.

Here, enzymatic polymerization utilizing  $\beta$ - cellobiosyl fluoride as the substrate monomer and celluloses of cellulose I in a reverse micelle, this very different approach may have some future industrial application.

On the other hand, focusing on native cellulose, the present time is very active in identifying cellulose syntheses genes from many plants and from various developmental stages. The next step will be the use of "antisense" to either halt, or the introduction of modified genes to literally modify the structure of cellulose synthesized by living organisms. Already transgenic plants with modified cellulose biosynthetic pathways are beginning to emerge in the literature as of 2000. Thus, the future for genetic modification of cellulose biosynthesis in plants and microbes promises a vast new future for the forest products and textile industries.

#### 2.7 BREAKDOWN (CELLUCOLYSIS).

Cellulose is the process of breaking down cellulose into smaller polysaccharides called cellodextrins or complete into glucose units; this is a hydrolysis reaction. Because cellulose molecules bind

strongly to each other, cellulolyses is relatively difficult compared to the breakdown of other polysaccharides.

Most mammals have only very limited ability to digest dietary fibres such as cellulose. Some ruminants like cows and sheep contain certain symbiotic anaerobic bacteria (like cellulomonas) n the flora of the rumen, and these bacterial produce enzymes called cellulose; the breakdown products are the used by the bacteria for proliferation. The bacterial mass is later digested by the ruminant in its digestive system (stomach and small intestine). Similarly, lower termites contains in their hindguts certain flagellate protozoa which produce such enzymes; higher termites contain bacteria for the job. Some termites may also produce cellulose of their own. Fungi, which in nature are responsible for recycling of nutrients, are also able to breakdown cellulose.

The enzymes utilized to cleave the glycosidic linkage in cellulose are glycoside hydrolyses including endo-acting cellulose and exo-acting glycosidase. Such enzymes are usually secreted as part of multienzyme complexes that many include doctrines and carbohydrate-binding modules.

#### 2.8 HEMICELLULOSE

Hemicelluloses is a polysaccharide related is cellulose that comprises about 20% of he biomass of most plants. In contrast to cellulose, hemicelluloses is derived from several sugars in addition to glucose, especially Xylose but also including mannose, galactose, rhamnose, and arabinose. Hemi- cellulose consists of shorter chains-around 200 sugar units. Furthermore, hemicellulose is branched, whereas cellulose is unbranched. (Wikipedia,2013).

#### 2.9 DERIVATIVES

The hydroxyl groups (-OH) of cellulose can be practically or fully reacted with various reagents to afford derivatives with useful properties like mainly cellulose esters and cellulose ethers (-OR). In principle, though not always in current industrial practice, cellulosic polymers are renewable resources. (Wikipedia,2013). Ester derivatives include:

Cellulose	Reagent	Example	Reagent	Group R
esther				
reagent				
Organic	Organic	Cellulose acetate	Acetic acid	H or-(c=o) CH <sub>3</sub>
esters	acids		and acetic	
			anhydride.	
		Cellulose	Acetic acid	-(c=o)CH <sub>3</sub>
		triacetate	and acetic	
			anhydride	
		Cellulose	Propanoic	H or –(c=o)
		propionate	acid	$CH_2CH_3$
		cellulose acetate	acetic acid	H or- (c=0) CH <sub>3</sub>
		Propionate	Propanoic	(C=O) CH <sub>2</sub> CH <sub>3</sub>
			acid –	
		Cellulose acetate	Acetic acid	H or-(=o)CH3
		Butyrate	butyric acid	-(C=O)
				$CH_2CH_2CH_3$

Inorganic	Inorganic	Nitrocellulose	Nitric acid or	H or $-NO_2$
			another	
Esters	Acids	(cellulose nitrate)	power full	
			nitrating	
			agent	
		Cellulose sulfate	Sulfuric acid	H or –so3 <sup>H</sup>
			or 1 another	
			powerful	
			10	
			sulfuring	
			agent	

The cellulose acetate and cellulose triacetate are film-and fiberforming material that fine a variety of uses. The nitrocellulose was initially used as an explosive and was an early film forming material with camphor, nitrocellulose gives celluloid.

## Ether derivatives include:

Water	Application	Е
Solubility		Num
		ber
Cold		E461
Water		
soluble		
Water	А	E462
insoluble	comm	
	ercial	
	thermopl	
	astic used	
	in	
	coating,	
	links,	
	binders,	
	and	
	controlle	
	d release	
	drug	
	Water Solubility Cold Water Soluble Water nsoluble	WaterApplicationSolubility

						tablets	
		Ethyl methyl	Chloromethane	-CH <sub>3</sub> or			E465
		cellulose	and	_			
			chloroethane	CH <sub>2</sub> CH <sub>3</sub>			
Hydroxyalk-	Epoxides	Hydroxyethyl	Ethylene oxide	CH2CH	Cold/hot	Gelling	
yl		cellulose		2OH	water	and	
					soluble	thickenin	
						g agent	
		Hydroxpropl	Propylene	-CH <sub>2</sub> CH	Cold		E463
		cellulose (HPC)	oxide	(OH)	water		
				CH <sub>3</sub>	soluble		
		Hydroxyethyl	Chloromethane	-CH <sub>3</sub> or	Cold	Viscosity	E454
		methyl cellulose	and ethylene	-CH <sub>2</sub>	water	modifier,	
			oxide	-CH	soluble	gelling	
				(OH)		foaming	
				-CH3		and	
						binding	
						agent	
		Ethyl	Chloromethane	CH2CH			E467
		hydroxyethyl	and ethylene	3			
		cellulose	oxide	OrCH2			
				CH2 OH			

Carboxyalkyl	Halogenated	Carboxymethyl	Chloroacetic	-	Cold/hot	Often	E466
				CH2CO	water	used as	
				ОН		its	
	Carbocylic acids	Cellulose			Soluble	Sodium	
		(CMC)				salt,	
						sodium	
						carboxy	
						methyl	
						cellulose	
						(NaCMC)	
					1		1

#### 2.10 FUNCTIONALITY

Cellulose has many uses as an anticake agents, thickner, and getting agent but these are generally subsidiary to its most important use or holding on to water cannot penetrate crystalline cellulose but dry amorphous cellulose absorbs water becoming soft and flexible some of this water is non-freezing but most is simply trapped. Less water is bound by direct hydrogen bonding if the cellulose has high crystallinity, but some fibrous cellulose products can hold onto considerable water in pores and its typically strawlike cavities, water holding ability correlating well with amorphous (surface area effect) and void fraction. As such water is super callable, this effect may protect against ice damage. Cellulose can give improved volume and texture particularly as a fat replacer in sauces and dressings but its insolubility means that all products will be cloudy.

Cellulose can also be said to function as follows:

- It is permeable of water gases
- It protects the protoplasm of the cell
- It is rigid and so gives structural support, shape and form to the cell.

#### 2.11 OCCURRENCES

Cellulose is mostly found in plants as micro fibril (1598). It usually forms the structurally strong framework in the walls. Cellulose (E460) is mostly prepared from wood pulp. Cellulose is made from sugar; it can also be processed to fuels and chemicals by microorganisms fermenting the sugar. some example; the conversion of cellulose to second generation bio-ethanol by yeast or the production of bio-plastic based on lactic acid from bacteria.

#### 2.12 CLASSIFICATION OF CELLULOSE

The plant's cell wall is composed of several chemicals, of which cellulose (made up of molecules of the sugar glucose) is the most important. Cellulose molecules are into fibrils which form the structural framework of the wall. The synthetic material called celluloid, as its name implies, was derived is treated with nitric acid, a derivative called cellulose nitrate is formed. An American inventor John Wesley Hyatt (1837-1920) found a way to soften cellulose nitrate by treating it with ethyl alcohol and camphor. The softened material could be molded into smooth, hard bills, thus, hyatt brought the game of billiards within the economic reach of more people and saved a few elephants.

Classification of cellulose is based on thus:

- Cellulose acetate
- Cellulose acetate butyrate
- Cellulose nitrate
- Methyl cellulose
- Ethyl cellulose
- Carboxyl methyl cellulose

#### 2.12.1 CELLULOSE ACETATE.

Cellulose acetate is a white, odorless tasteless, non-toxi solid. It is manufactured having substitution ranging from 2.3-3.0 or from 53 to 62.5% combined acetic acid. Cellulose acetate is obtained by treating cellulose with concentrated acetic acid or acetic anhydride in presence of a catalyst such as H2504.

 $N(C_6H_7O_2(OH_3) + 3n (H_3(OOH H_2504 => n (C_6H_7O_2(OCOCH_3)+C_3NH_2O cellulose triacetate.$ 

The resulting cellulose bioacetate is partially hydrolyzed to render it soluble in organic solvents such as acetone. It has good clarity, stability toughness, impact strength and resistance to UV radiation. It has a high dielectric strength, high tensile strength and resistance to mineral acids. It has good film and plastic strength. It is used for preparing fibers for textiles on one hand and for preparing transparent sheets on the other.

#### 2.12.2 CELLULOSE ACETATE BUTYRATE.

Cellulose acetate butyrate is a copolymer having lower water absorption that the cellulose acetate alone. It is prepared by

treating cellulose with a mixture of acetate acid on butyric acid in presence of  $H_2SO_4$  (catalyst). Its thermal stability and other properties are similar to that of cellulose acetate plastics but has better chemical and moisture stability, better dimensional stability and impact strength. It is used for tooth brush handles, combs, buttons, pens, automobile hardware.

#### 2.12.3 CELLULOSE NITRATE.

Cellulose nitrate is also know as nitro- cellulose it is prepared by reacting cellulose with nitric acid in presence of sulfuric acid which acts as a dehydrating agent. The partially nitrated cellulose mixed with camphor gives the so-called celluloid, which can be easily softened and moulded and the function of camphor is to act as a plasticizer to enhance the moulding properties.  $N(C_6H_{IO}O_5)$ .

Cellulose nitrate is inflammable and resistant to water but is attacked by strong acids becomes brittle at low temperature. It is also used as smokeless gunpowder, the cellulose nitrate plastics are transparent, strong and tough and the specific gravity is 51.35-

1.40, it's dimensional stability is poor as it slowly shrinks due to slow loss of plasticizer and hence is not used for precision parts.

#### 2 12 .4 METHYL CELLULOSE.-

Methyl cellulose can similarly be prepared from alkalis cellulose by treating with methyl chloride.

Cellulose-ONG+CH<sub>3</sub>CL=> cellulose OCH<sub>3</sub>+NaCl.

#### 2.12.5 ETHYL CELLULOSE.

Ethyl cellulose is prepared by treating cellulose first with caustic soda to produce the caustic cellulose and then allowing it to react with ethyl chloride to given an average of 2.0 ethyl groups per ½ ethyl cellulose monomer. Ethyl cellulose is the toughest of all cellulose plastic. Chisel handles, mallet heads and light, it has good chemical resistance and stable to heat and light, it has good electrical properties and hence films, foils, and sheets and made from it are used for electrical insulation purpose. Special compositions have been used to produce rain coat surgical tapes, laminated glass sheets and straw like extrusions for weaving

furniture seats its toughness and flexibility are retained through wide range of temperature, it has excellent solubility in many cheap solvents and hence foils and films with good strength and toughness can be prepared by casting its solutions.

Ethyl cellulose is obtained by the replacement of hydrogen atoms by ethyl groups in the reactive hydroxyl groupings in cellulose it is not necessary to complete the reaction as in the case of cellulose acetate.

Ethyl cellulose plastics are made by mixing ethyl cellulose with plasticisers and other ingredients either an open roils in an internal mixer such as Banbomy mixer.ethyl acetate moulding powders have widely been used for pressure and extrusion purposes.

#### 2.12.6 CARBOXY METHYL CELLULOSE.

Carboxy methyl cellulose is prepared as its sodium salt, by the reaction of actualize with NaOH (sodium hydroxide) and chloroacetic acid, it has been used as a thickening agent in textile additives, cosmetics cakes, ice cream etc it has also been used

university as a protective colloid in suspension and emulsion polymerization system.

Cellulose=> OH+2NaOH + ClCH<sub>2</sub> (OOH+ cellulose- OCH<sub>2</sub>CONa+ NaCl + 2H<sub>2</sub>0 carboxy methyl cellulose.

Generally, cellulose occurs in plant life, cotton or wood cannot be shaped, moulded, cast or extruded, for performing these processes it is necessary to modify it cellulose can be modified in the following manner.

#### 2.13 REGENERATED CELLULOSE.

• Cellulose is brought into the solution form, it is the temporary modified form of cellulose from this modified form fibres. Or films are shaped by spinning or casting and then regenerated to the original cellulose chain forming rayon or cellophane when cellulose is brought into a solution by means of a chemical reaction and it is reprecipited out of the solution to give essentially the same chemical structure in the form of film or fibre. It is said to be regenerated cellulose. In this transformation of shapes there is not much change in chemical and physical

properties of cellulose. Ieces and allowed to age under controlled conditions of temperature and humidity. During ageing, the high polymer molecule breaks down a little. The breakdown is indicated by better solubility in solvents.

- Rayon-rayon is the name given to fibres (filaments cut into small lengths) and continuous yarns made of regenerated cellulose. Rayon is also made by a method similar to that used in the manufacture of cellophane. The only difference is that the viscose solution is extruded through spinnerets into the acid bath instead of narrow slot into the acid bath. Thus, a major part of rayon and cellophane are derived from the easily hydrolysed xanthate of cellulose when the xanthate is extruded is solution into a bath of dilute acid as a sheet or thin stream. It is rapidly hydrolyzed, regenerating a slightly degraded form of cellulose as a sheet (cellophane) or as fibre (rayon).
- Under cellulose acetate the following are manufacture of cellulose acetate.:
- Pretreatment of cellulose- pure cellulose is direct at about 50%c to a moisture content of less than 4%. It is then pretreated with

small amount mineral acids in other to increase the reactivity of cellulose to break it down to make it more soluble.

Reaction with acetic anhydride- 100 parts of dry pretreated cellulose are mixed with a mixture of 200 parts of glacial acetic, 100 parts of concentrated H<sub>2</sub>SO<sub>4</sub>. The temperature is maintained between 10°C after 2hrs, the mixture first become fluid and then start thickening up when cotton fit in remains, the process gets completed of the process generally takes about 5hrs.

#### 2.14 USES OF SUGAR.

The common (most) of all sugar is granulated sugar which is used by many people to sweeten hot drinks and breakfast cereals sugar is used in variety of forms waters, gases etc if can also be brought in cubes, which are made by pressing wet sugar into mould before drying icing sugar, used to coat cakes and pastries, is made by grinding the sugar crystals into a fine powder. Other forms of sugars include caster sugar finer than granulated and sued for jams and jellies and brown sugar (for colour and flavor). Syrups and treacle (for and spreading) are liquid forms of sugar.

Sugar is not first a sweeten, it can be used in so many different ways:

- As a preservative- at the right concentration sugar helps to stop micro-organisms growing and so prevents find spoilage in jams and other preserves, it helps to produce subtle change sin flavor.
- Sugar gives the characteristic texture to a variety of foods.
- Sugar softens the acidity and sourness in foods such as mayonnaise, tomato products and fart tracts such as goose berries and grapefruit.
- It makes cakes light and open-textured when it is beaten with butter or eggs.
- Sugar helps to speed up the process so fermentation (by yeast) in baking which makes the dough rise, e,g in bread and tread cakes.
- To raise the boiling point or lower the freezing point which can be essential in some recipes eg making ice create etc.

#### 2.15 SUGAR IN FOODS

Sugar is added to many foods for flavor, texture sugar colour and safety. By reading the ingredients panel on food packaging tables you can see how many ordinary foods that contains sugars.

Some other uses of sugar include:

Sugar is use in healing of wounds

It can be added to concrete to acid the setting process

It is use by chemical manufactures to grow penicillin

It absorbs moisture and therefore keep your biscuits country.

It makes the "glass" used in film taunts.

It is also used in production of ethanol

It helps flowers stay fresher for longer when added to the water.

#### 2.16 FUNCTIONS OF SUGAR.

Glucose is sued in respiration to produce energy for the activities of plants and animals and is also stored in the muscles and live in the form of glycogen.  $C_6H_{12}O_5 + 6CO_2 + 6H_2O$ - energy.

Glucose is used to build up proteins or fats. These proteins are used for growth and for the building up of materials for new cell walls such as cellulose.

Sugar, either the pentose, ribose, or its 2-deaxy derivative; both in the furnanose from the structural components of nucleic acid. Deoxyribonudeic acid (DNA) present in the nucleic of cells is the generic material, and indirectly controls the synthesis of proteins.

#### **2.17 NATURAL POLYMERS OF SUGAR.**

Biopolymers of sugar are common in nature. Through photosynthesis pants produce glucose, which has the formula  $C_6H_{12}O_6$ , and convert it for storage as an energy reserve in the form of other carbohydrate such as starch, or (as in cane and beet) as sucrose, which the chemical formula  $C_{12}H_{22}O_{11}$ . Starch, consisting of two different polymers of glucose, is a readily degradable form of chemical energy stored by cells, and can be converted to other types of energy.

Another polymer of glucose is cellulose which is a linear chain composed of several hundred or though sand glucose units. It is used by plants as a structural component in their cell walls. Humans can only digest cellulose to a very limited extents, though ruminants can do so with the help of symbiotic bacteria in their gut. DNA and RNA are built up of the monosaccharides deoxyribose and ribose respectively. Deoxyribose has the formula  $C_5H_{10}O_4$  and ribose the formula C5H1005.

#### 2.18 TYPES OF SUGAR.

**Monosaccharides:**- glucose, fructose and galactose are all simple sugars, monosaccharides, with the general formula C6H1206. They have five hydroxyl groups (-OH) and a carbonyl group (c=o) and are cyclic when dissolved in water. They each exist as several isomers with dextor-and laevo-rotatory forms which cause polarized light to diverge to the right or the left.

**Glucose:** dextrose or grape sugar occurs naturally in fruits and justice and is the primary product of photosynthesis.most ingested

carbohydrates are converted into glucose during digestion and it is the form of sugar that is transported around the boodles of animals in the blood stream. It can manufactured from starch by the addition of enzymes or in the presence of acids. Glucose syrup is a liquid form of glucose that is widely used in the manufacture of food stuff. It can be manufactured from starch by enzymatic hydrolysis.

**Fructose** or fruit sugar occurs naturally in fruits, some root vegetables, cane sugar and honey and is the sweetest of the sugars. It is one of the components of sucrose or table sugar, it is used as a high fructose syrup which is manufactured from hydrolyzed corn starch which has been processed to yield part of the glucose into fructose.

Glucose does not generally occur in the free state but is a constituent with glucose of the disaccharide lactose or milk sugar, it is less sweet than glucose. It is a component of the antigens found on the surface of red blood cells that determine blood groups.

**Disaccharide:** sucrose, maltose and lactose are all compound sugars, disaccharides, with the general formula  $C_{12}H_{12}O_{11}$ . They are

formed by the combination of two monosaccharide molecules with the exclusion of a molecule of water.

Sucrose is found in the stems of sugar cane and roots of sugar beet. It also occurs naturally alongside fructose and glucose in other plants, particularly fruits and some roots such as carrots. The different proportions of sugars found in these foods determines the range of sweetness experienced when eating them molecule of sucrose with a molecule of fructose. After being eaten, sucrose by a number of enzymes known as surceases.

**Maltose** is formed during the germination of certain grains, most notably barley which sugar's name. a molecule o maltose is formed by the combination of two molecules of glucose. It is less sweet than glucose, fructose or sucrose. It is formed in the body during the digestion of starch by the enzyme amylase and is itself broken down during digestion by the enzymes maltase.

**Lactose** is the naturally occurring sugar found in milk. A molecule of lactose is formed by the combination of a molecule of galactose with a molecule of glucose, it is broken down when consumed into

its constituent part by the enzyme but some adults no longer form it and they are unable to digest lactose.

Methods of producing glucose from cellulose

Glucose is obtained form paper making sludge by first removing calcium carbonate from the sludge to produce a mixturesubstantially depleted of calcium and containing cellulose.

#### **CHAPTER THREE**

To obtain the aim and objective of this project "effect of temperature on hydrolysis of cellulose (saw-dust) using hydrochloric acid (NCL)" certain laboratory experience and work was carried out and hence this chapter will strictly base on the equipment/apparatus used during the analysis or practical with the procedures involved.

#### 3.1 Materials and Equipment

3.1.1 **Materials**: Sawdust cellulose used in the production of glucose production was collected from timber shade, new G.R.A, TRANS-Ekulu abakpa nike enugu.

#### **3.1.2** Apparatus:

- \* 250 beaker (x5)
- \* Round bottom flask
- \* Measuring Cylinder (100ml and 10ml)
- \* Test tubes (x5)
- \* Filter paper

- \* Funnel
- \* Heating mantle
- \* Stop watch
- \* Spatula
- \* Weighing balance
- \* Volumetric flask (500ml)
- \* Litmus paper
- \* Cuvette
- \* Visible spectrophotometer
- \* Universal PH.

## 3.1.3 Reagent

- \* Hydrochloric acid (5%) solution
- \* distilled water
- \* Benefit's solution
- \* Glucose solution

#### 3.2 Acid hydrolysis (HCL)

Aim and objective: the most important aim of this acid hydrolysis is to convert sawdust (cellulose) to glucose by using hydrochloric acid solution during hydrolysis analysis.

**Theory:** although acid hydrolysis was used in this experiment, but at the same time enzyme hydrolysis was the earliest method of hydrolysis of cellulose to glucose and to achieve a perfect result in acid hydrolysis an acid or different acids will be used at a constant temperature (80°C). The starting material was saw-dust which was obtained from the woods.

#### **3.2.1 Procedure:**

10g of (cellulose) sawdust was weighed and poured into a beaker, then 200ml of distilled water was added and stirred thoroughly.

The mixed solution was placed on a heating mantle to boil for 30mins at a constant temperature of 80°C, this saw procedure was also carried out into four different beakers containing 10g of sawdust each but instead of 200ml of distilled water, HCL acid was replaced for 5ml, 10ml, 15ml and 20ml. as of this time, the reaction

of acid (HCL) on the sawdust solution must have converted a good quantity of the sawdust to glucose after boiling living the four acid solution filtrate with yellow colour and the other non-acidic solution alternate with a very dark ox-blood colour after filtering with a filter paper and funnel into a round bottom flask.

The acidity of the sample was checked using universal PH, when the colour appear orange it is acidic but when neutral it is nonacidic. The solutions were poured into a cuveet, then placed on the visible spectrophotometer along side with a distilled water. The absorbance reading was determined at 610m wavelength

#### 3.2.2 Glucose analysis colorimetric (using Benedict's method).

**Aim:** to use a colorimetric analysis method and a calibration graph to identify the percentage of glucose in the unknown solution.

**Theory**: the commonest method of analyzing glucose/sugar in hydrolyzed (cellulose) sawdust is by using an instrument known as glucoment, DNS, fehling's solution test, or benedict's solution by colorimetric analysis method, although in this experiment beneficts solution was used and it was achieved by measuring the benedict's solution in each sample in equal proportion.

#### **3.2.3 Procedure:**

Five milli-litre of (HCL) glucose concentrations and standard glucose concentration was measured each in a different (5x) test tubes, then 10ml of benedict's solution was added to each of the concentration. The five test tubes were inserted into 250ml beaker containing water and was palced on a heting mantle at a constant temperature of 85%. The test tubes containing the solutions were removed from the hating mantle and it was allowed to cool, then solution absorbance reading was determined using.

# The glucose concentration (HCL) was determined using BEER LAMBAT LAW.

The beer- Lambert law (or beer's law) is the linear relationship between absorbance and concentration of an absorbing species. The formular is stated below: A=ELC

Were A= adsorption

E= epsilon) = adsorptivity of solution

L= wave length

## C= concentration

#### **CHAPTER FOUR**

#### **RESULTS AND DISCUSSION**

#### 4.1 RESULTS

This chapter is all about the obtained results and discussion of glucose content in each concentration of (HCL) acid at constant temperature. The table shown below is the results obtained from glucose analysis and glucose yield with HCL at different concentration.

Figure 4.1 shows the plotted graph of glucose concentration with HCL acid. The detailed calculation and tables are also shown.

Concentration	Absorbance (mg/dl)
(gram)	
0.01	0.065
0.02	0.163
0.03	0.247
0.04	0.346
0.05	0.459

concentration

Hydrochloric	Absorbance in	Concentration	Percentage C%
Acid (HCL) in	(mg/dl)	(grain)	
(ML)			
10	0.220	0.026	0.260
20	0.483	0.052	0.520
30	0.724	0.074	0.740
40	0.983	0.116	1.160
50	1.244	0.127	1.270

Table 4.1.2: Variation of absorbance with hydrochloric acid.

#### 4.2 **DISCUSSION**

The results gotten from the various experiments conducted under this research project of (HCL) acid on cellulose (saw-dust) conversion to glucose. The rate of hydrolysis is greatly influenced by acid (HCL) and this affect the glucose yield produced in figure 4.1.

Increase in concentration of acids like HCL enhances the hydrolysis process positively, as the cellulose (sawdust) hydrolyzed more with increase in concentration of acids. However, for standard glucose concentration, it was observed that the optimal yield of concentration, it was observed that the optimal yield of concentration of HCL acid was 1.270%.

While conducting experiment on acid hydrolysis it was noticed that HCL acid although was but also took time in breaking the glucosidic bond of cellulose, converting it to sugar or glucose.

Finally while conducting experiment on glucose analysis the results obtained showed that HCL acid solution has relatively high absorbance of glucose 1.244g moles at glucose concentration 1.270% unlike some other experienced analyzed acid and this shows that further research on HCL acid, will result in getting to equilibrium curve, because the higher the glucose concentrations, the higher the absorbance.

From Table 4.1, it is evident that the optimum concentration for standard glucose is 0.05g, and the reason is because after 5 minute of heating, the glucose concentration is required to settle under the testude when put into a cold water for another 5 minutes.

#### **CHAPTER FIVE**

#### **5.0 CONCLUSION AND RECOMMENDATION**

#### 5.1 CONCLUSION

Throughout the study, the researcher identified or noticed some possibility of using cellulose (saw-dust) for industrial sugar production through acid hydrolysis.

In my investigation the yield of glucose from hydrolyzed cellulose is affected by various acids which includes, HCL. It was observed that the highest yield of glucose was obtained at the optimum glucose and is at 1.270% for HCL. Thus, it is an evidence that the concentration of absorbance increases with an increase in glucose concentration at a fixed wavelength (610) and it is therefore assume that the best acid hydrolysis is hydrochloric (HCL) acid. Lastly the research finding also revealed that the production of sugar or glucose from local raw material such as cellulose (saw-dust) by acid hydrolysis could be considered viable economically.

#### **5.3 RECOMMENDATION**

The following recommendations were made by the researcher based on the results of the study.

- I recommend that since HCL acid is assumed to be the best, it should be widely used in acid hydrolysis in other to get the highest yield that can result to the obtaining of equilibrium curve.
- Although the researcher found out that the use of commercial saw-dust (cellulose) is more profitable and less expensive, and also more faster and easier to process and extract unlike other cellulose from rice tusks e.t.c.
- I recommend that the application of the acid hydrolysis of cellulose should be employed in large-scale sugar/glucose manufacturing in Nigeria as it will save our country a great deal of foreign exchange.
- Lastly, the government, industrialists, investors, business individuals and others should take consideration on the capital to invest into this new method of glucose hydrolysis. Through acid hydrolysis, for it will help to increase the availability of

cellulose in the market, provide employment, reduce the cost of sugar and improve the exportation of glucose in Nigeria, hence making our economy an industrialized economy.

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