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**PRODUCTION AND PRESERVATION OF WATER
RESISTANT GUM FROM CASSAVA STARCH(DEXTRIN)**

BY

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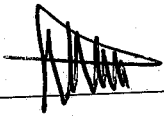
**A PROJECT REPORT SUBMITTED TO THE DEPARTMENT OF
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CERTIFICATION

This is to certify that this work was carried out by Anyadiufu Nkiruka, and as read and approved under the supervision of Engr.M.U. Garba as meeting the requirement of chemical engineering department, federal university of technology, Minna, for the award of bachelor of engineering, (B.Eng) in chemical engineering.



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DECLARATION

I hereby declare that this project work is my original work and has never to my knowledge being submitted elsewhere by anyone before.



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DATE

DEDICATION

This work is dedicated unto Him the Omnipresence, Omniscience, and Omnipotent, the Alfa and Omega who has kept me throughout these days from falling. The most High God whom without His divine guidance and mercy, this programme would not have come to reality.

To my lovely husband Mr. Joseph U. Okoh and my beautiful daughter Dabel.

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TABLE OF CONTENTS

| | |
|---|-----|
| Title Page | i |
| Certification | ii |
| Declaration | iii |
| Dedication | iv |
| Acknowledgement | v |
| Table of Contents | vi |
| Abstract | ix |
| CHAPTER ONE | |
| 1.1.0 General Introduction | 1 |
| 1.2 Aim | 1 |
| 1.3 Objectives | 1 |
| 1.4 Justification | 2 |
| CHAPTER TWO | |
| 2.0 Literature Review | 3 |
| 2.1 Sources Of Starch | 3 |
| 2.2 Molecular Structure of Starch | 4 |
| 2.3 Physical properties of Starch | 4 |
| 2.4 Chemical properties of Starch | 6 |
| 2.4.1 Condensation Reaction | 6 |
| 2.4.2 Hydrogenation | 7 |
| 2.4.3 Oxidation | 7 |
| 2.4.4 Retrogradation | 7 |
| 2.4.5 Iodization Test | 7 |
| 2.4.6 Esterification and Etherification | 7 |
| 2.4.7 Hydrolysis of Starch | 7 |

| | |
|--|----|
| 2.5.0 Uses of Starch | 8 |
| 2.6.0 Gelatinization of Starch | 8 |
| 2.6.1 Factors Affecting Gelatinization | 9 |
| 2.7.0 Dextrin | 10 |
| 2.7.1 Classification of Dextrin British Gum | 10 |
| 2.8.0 Gum Adhesive | 10 |
| 2.8.1 Water soluble polar adhesive | 10 |
| 2.8.3 Water insoluble polar adhesive | 11 |
| 2.8.3 Non polar adhesive | 11 |
| 2.9.0 Stabilization of adhesive | 11 |
| 2.9.1 Plasticizers | 11 |
| 2.9.2 Humectants and liquefying agents | 11 |
| 2.9.3 Preservatives | 11 |
| 2.9.4 Deformers | 11 |
| 2.9.5 Solubilizers | 12 |
| 2.9.6 Mineral fillers | 12 |
| 2.10.0 Evaluation of adhesive | 12 |
| 2.10.1 Adhesive formulation | 12 |
| CHAPTER THREE | |
| 3.0 Materials and methods | 14 |
| 3.1 Materials | 14 |
| 3.2 Equipment | 14 |
| 3.3 Method | 15 |
| 3.3.1 Procedures for production of adhesives | 15 |
| 3.3.2 Peeling and washing | 15 |
| 3.3.3 Grating | 15 |
| 3.3.4 Screening | 15 |
| 3.3.5 Settling and decanting | 16 |

| | |
|-------------------------------------|----|
| 3.3.6 Drying | 16 |
| 3.3.7 Frying | 16 |
| 3.3.8 Sources of experimental error | 17 |
| 3.3.9 Precautions | 18 |
| 3.3.9.1 Limitation | 18 |
| 3.4.0 Physical characterization | 19 |
| 3.4.1 Gelatinization | 19 |
| 3.4.2 Stabilization temperature | 19 |
| 3.5.0 Chemical characterization | 19 |
| 3.5.2 Concentration variation | 20 |
| CHAPTER FOUR | |
| 4.0 Result and discussion | 21 |
| 4.1 Results | 21 |
| 4.2 Discussion of Results | 27 |
| CHAPTER FIVE | |
| 5.0 Conclusion and Recommendation | 29 |
| 5.1 Conclusion | 29 |
| 5.2 Recommendation | 29 |
| References | 30 |

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ABSTRACT

This research work was carried out to produce a water resistant gum and to study its preservative ^{properties} principles through which different concentration of acid which serves as major preserving agent was used on different dextrin samples obtained from cassava plant. The gum produced was examined for both physical properties such as: viscosity, ^{pH} PH, bindability, mechanical strength and chemical properties such as: roasting acid concentration and effective solubility through which it was shown in the result that the gum produced when preservatives were added to starch has the best properties and the best dextrin sample ^{for} foe adhesive gum production is white dextrin because of its has the highest solubility which is 36.1%. Thus, the gum production should be done with effective use of chemical additive and the white dextrin is preferable to other types of dextrin.

CHAPTER ONE



1.1.0 GENERAL INTRODUCTION

This project is concerned with the production and preservation of gum known as dextrin from cassava starch which is the major raw material.

Starch from where dextrin is obtained is an organic compound found abundantly in nature, like sugar. No synthetic method has been device to manufacture them, hence, they extracted from natural product and purified for the specific need.

Dextrin are used mostly in industries, food manufacturing company due to their non-toxicity and their low price/

They are used as water soluble glues, as thickening part in food processing and as binding agent in pharmaceuticals.

We have some other sources of starch apart from cassava e.g. tapioca, potatoes, wheat, guinea corn and maize (www.wikipedia.org/wiki/dextrin).

Cassava starch is whitish, tasteless, odorless and neutral powder by nature. If there is no contamination in the cause of production or fungal bacteria infection of cassava.

Starch belong to the group of organic compounds called polysaccharides which is major example of carbohydrate, consisting of carbon, hydrogen and oxygen as the basic elemental composition, it also consists of unit arranged as an amylose (straight chain) or amylopectin (branched chain).

Information has shown that the adhesive market was dominated by imported brand. Available records indicated that in 1983, fifteen million naira worth of adhesive product was imported into the country. Hence, with the coming of this project into reality it will be able to save the country some foreign exchange and hence boost the production, capacity of dependent industries.

1.2 AIMS

The aim of the project is to produce dextrin from starch

1.3 OBJECTIVES

The objective of this project is to produce dextrin from cassava starch via the following procedure:

- 1) Clean the cassava
- 2) Reduce the size and obtain starch pulp
- 3) Peeling, washing, rasping, grabbing sieving and ^cscreening
- 4) Drying and frying which is done between 60° to 65° C

1.4 JUSTIFICATION

Dextrins apart from adhesives which are used in the industry pastes and gums, it can be used in making cooking spacies e.g. kitchen glory and beverages example jago chokollite (malta dextrin).

CHAPTER TWO

2.0 LITERATURE REVIEW

2.1 Sources Of Starch

There are various roots or tubers that starch can be obtained from, these includes cassava roots, yam tubers, sweet potatoes, sweet yam, water yam etc.

Among the fruites that we can obtain starch from includes; corn, guinea corn etc

But for this project starch from cassava is our major source of interes.

There are three major classes of cassava from which starch can be obtained for dextrin production. The classification is based on the amount of hydrogen cyanide present which determines the tastes as well as starch content.

These species are:

1. The mainihot utilissima with better taste (this is the high hydrogen cyanide content cassava.
2. The manihot esculentus with low bitter taste. (this is the medium hydrogen cyanide content cassava.
3. The low hydrogen cyanide content (manihot polmand). This is the sweet cassava with 10-20% protein, this types of cassava can be consumed directly after gelatinization where as the former and intoxicating and needs to be fermented to reduce hydrogen cyanide by the action of meltese and zymase that increase it's super content (<http://en.wikipedia.org/wiki/starch.>)

Percentage composition of hydrogen content

composition

1. Bitter cassava 98-99.5%
2. Medium hydrogen cassava 80-86.4%
3. Sweet cassava 73.6-78.9%

X ^S (Stoire K.A and Strepkor S.M gave the following percentage of hydrogen content)

Starch is an organic compound that belongs to the polyssacharide group of carbonhydrate with the general formular $(C_6H_{10}O_5)_n$ where n ranges from hundred to over one million. Starch has 2 forms to which the units are arranged in the molecules, these are amylose (straight chain) and amylopectin (branched chain).

The table below illustrates crops that starch was obtained from and studied by meyer A

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Table 2.1 Starch Crops Table

| Starch crops | Corn | Wheat | Rice | Tapioca | Potatoes | Sagd |
|----------------------------|-------|-------|-------|---------|----------|-------|
| Source | Seed | Seed | Seed | Root | Root | Pith |
| Granude sizes mn | 5-26 | 3-35 | 3-8 | 5-35 | 15-100 | 10-70 |
| Temperature oc | 62-72 | 58-64 | 68-78 | 49-70 | 59-68 | 60-67 |
| Amylase % | 28 | 25 | 19 | 20 | 25 | 26 |
| Amylase (bp) | 480 | - | - | 1050 | 850 | - |
| Amylopectin (bp) | 1450 | - | - | 1300 | 200 | - |
| Gelatinization temperature | 62-72 | 58-64 | 68-69 | | 15-1000 | 10-70 |

2.20 MOLECULAR STRUCTURE OF STARCH

The general molecular formular of starch is $(C_6H_{10}O_5)_n$ where 'n' ranges from one hundred upwards to millions of number. The starch molecula structure for amylase and amylopectin is shown below

Schematic representation of part of amylopectin molecule

2.3 PHYSICAL PROPERTIES OF STARCH

Pure starch is different from commercial starch, it is a white odourless, tasteless, neutral, powder, insoluble in cold water or organic solvent, in a well dried state it is hydroscopic, when it is air dried. Starch contains quite appreciable amount of water.

The colour of commercial starchs may vary from white to a shade of grey or brown, their reaction may either be alkaline or acidic, the color of a commerical starch is treated heat to form a brownish pastes called dextrin. The densities of the more important starch are approximately 1.625g/cm³, although the density of potato starch is seen to be the highest compared with a gram of other crop.

The study of physical properties of starch is a fascinating one and leads to the information explaining a number of perculiarities that may often be observed. Why, for example should one batch of starch be more readily convertible to dextrin than others or give stiffer paste than a preveious delivery, or why should one paste possess adhesive properly or stable mucilages than another or take longer time reach the same viscosity when converted to dextrin by enzymes. Physical chemistry always give the answer to such question.

X
Starch is insoluble in cold water but swells to some extent when mixed with water, and shrinks to its original size when dried to its previous moisture content starch whose consistency is 42% appears as fluid but the slightest pressure on it causes it to solidify and appears as a damp solid cake.

(Young)
X (A. Speich) has studied the structure of starch granules and determined the degree of swelling potatoes starch grains that had been carefully dried over phosphorus oxide when dispersed in 28 different liquids.

The volume changes varied from 68.88% increase in glycol to 7.20% decrease in dicalydehyde, for water the increase was 57.99%. in general the more hydrophilic the liquid the greater the swelling and the more lipophilic the less the swelling of the starch grains (Bailey, 1977).

The refractive indices of starch was studied by A. Frey-Wything; he considered the double refraction shown by potatoes starch granules to be due to strains, possible in a radial direction and not to the structure of the starch.

Lipophilic (non-polar) liquids were used with different sizes of grain granules of size ranging from (0.013 whole grain to 0.0124 periphery).

X He notices that, at a wave length of 589nm and temperature of 25°C the refractive index is 1.535 in the direction of the optical axis.

The determination of the physical property of starch was done as a team work, by: O.E. Samberg, who calculated the surface area of seventeen types of wheat flour from the dimension recorded by Grewe and Bailay, 1977.

He found out that the average surface area of starch granules is $200\text{cm}^2/\text{g}$ by average. Also he found out that the surface area of potatoes, wheat, corn, and rice respectively giving a rough ratio of 1:2:3:4.

X The test of hydration and the particles size was studied by many researchers and has been recorded as shown in Table 2.2.0

| STARCH SOURCE | HEAT OF HYDRATION CAL/G | REFRACTIVE INDEX | DENSITY G/CM ³ | AVERAGE GRANULAR DIAMETER M | SURFACE DIAMETER M ² /G | SURFACE AREA BY ABSORPTION |
|---------------|-------------------------|------------------|---------------------------|-----------------------------|------------------------------------|----------------------------|
| Corn | 25 | 1.5222 | 1.517 | 13.9 | 0.288 | 0.70 |
| Potato | 28 | 1.5135 | 1.511 | 41.3 | 0.0969 | 0.11 |
| Rice | 26 | 1.5219 | 1.582 | 5.25 | 0.767 | - |
| Wheat | 25 | 1.5245 | 1.542 | - | 0.20 | - |

Apart from the above data, it was also seen that dry starch is hygroscopic in nature provided, the equilibrium moisture content of the starch in question is less than that of atmospheric humidity and temperature.

Furthermore, once the critical moisture content has been established the starch will no longer absorb the moisture from the air. The moisture contents of corn, tapioca, potato and cassava were found to be 39.9%, 42.9%, 50.9% and 19.2% respectively. (Dr. M. Grace, 1971).

2.40 CHEMICAL PROPERTIES OF STARCH

The starch granules composed of hydroxyl group (OH) as well as the structure composition of amylose and amylopectin (Alexander, 1981). Due to the presence of amylose and hydroxyl group; starch undergoes various reactions such

2.4.1 CONDENSATION REACTION

When some quantity of aldehyde are introduced to a starch (under acidic condition) crosslink of structure are found between adjacent starch molecules, the product obtained becomes non-dispersible in water there by forming a low degree of product cross linking this product is the paste (dextrin).

2.4.2 HYDROGENATION

Sorbitol, polyhydric alcohols are obtained when aldehyde group present in the starch is reduced catalytically in the presence of acid this mechanism is hydrogenation.

2.4.3 OXIDATION

When starch is oxidized, aldehyde is formed in large quantity as well as ketone and carboxyl groups. The rate of oxidation is a function of the oxidant used and the prevailing temperature of the medium e.g

Alkaline hypochlorite solution oxidizes starch to yield carboxyl groups. The oxidant result in increase in solubility in water and also reduces the length of the molecules especially in dilute alkaline solution.

2.4.4 RETROGRABATION

The process of gel formation by association of the linear molecules (amylose) on cooling is termed retrogration. Retrogration is a function of molecular weight for any material and alcohol helps in retrogration. It is the formation of skin of pastes as well as those physico-chemical changes of states taking place in solutions.

2.4.5 IODINATION TEST

When iodine is reacted with starch, blue colouration is obtained. This is a result of the enzymes (amylose) that constitute fraction components of the polysaccharide cells of starch. This iodine that is test is useful in food industries in order to know whether the total starch present in complex food formulation has been fully converted. This confirms the birefringence of starch molecules

2.4.6 ESTERIFICATION AND ETHERIFICATION

The hydroxyl groups in starch undergoes the normal esterification and etherification reactions of alcohol. Thus when α - β -glucopyranose is treated with acetic anhydride, all the hydroxyl function are acetylated to yield retan-O-acetyl glucose.

2.4.7 HYDROLYSIS OF STARCH

An important property of starch is its ability to be hydrolysed. Acids or enzymes can be used to hydrolyse starch in either granules or gelatinized form. Enzymes hydrolysed starch efficiently only in dextrin form of starch. Based on efficiency acid is very effective than enzyme because it can hydrolyse starch both at granule or gelatinized form without any problem.

Some example of acids include HCl, H_3PO_4 e.t.c while enzymes include bacteria, fungi, animals and vegetable source of enzymes.

When starch is hydrolysed by any of the above method D-glucose is obtained. The ability of acid to hydrolyse starch is based on its ability to penetrate ungelatinized granules causing

hydrolyses at temperature of the starch. In the case of enzymes eg α -amylase, β -amylase and gluco-amylase at 58-65^oc being gelatination temperature ranges, most of these enzymes will die since most of them live mostly in the region of room temperature. This explains chiefly why enzymes cannot be used to hydrolyse starch properly like the acid counterpart (Grace, 1971)

2.5.0 USES OF STARCH

Starch can be used in a variety of ways based on mode of extraction operation

- 1) Starch plays a lot of role in industries such as paper industries etc
- 2) Sugar industries convert starch to obtain D-glucose
- 3) In an oil field, it is used by drillers during operation to prevent water entering the drilling channels for oil recoverability.
- 4) Textile industries use starch extensively to size yarn warps before weaving and in finishing various woven fabrics and as laundry starches.
- 5) It is used to produce dextrin, which is our major concern as far as this project is concerned(Kirk,2002).

2.6.0 GELATINIZATION OF STARCH

Starch granules gelatinize when treated in hot water between temperature ranges of 50-70^oC when the temperature of starch granules is ranged above room temperature, it swells progressively, the linear molecules (amylose) is dissolved to form paste with a loss of birefringence and disintegration of the granules fragments. Upon cooling, the viscosity continues to go higher and more paste is formed, depending on the concentration of starch used for paste production.

The production of paste employs a free radical mechanism thus, resulting in an irreversible process when paste is obtained

When the temperature of the starch granules is raised each granules continues to burst which result in swelling; thereby increasing the quantity of paste thrice the quantity of starch used.

As this bursting is continued a time is reached where the large particles have finished bursting and anymore addition of hot water will result in reduction in viscosity and consistency, this point where bursting ends is the gelatinization point; the process is gelatinization process while the critical temperature reached is called gelatinization temperature.

At this temperature the birefringence of starch is lost when viewed under a polarized microscope.

X.

When gel is formed without destruction of the starch property, dextrin is produced after the gel has cooled, since different crops cannot be gelatinized at the same temperature and at the same time. The table below shows gelatinization temperature ranges of some starching food crops

Table 2.1 gelatinization temperature

Table 2.3 Gelatinization temperature of different starches from different crops

| Starch source | Temperature °C |
|---------------|----------------|
| Wheat | 58-64 |
| Potato | 59- 6 8 |
| Corn | 67-72 |
| Sorghum | 68-100 |

By Malcoms, 1979.

2.6.1 FACTORS AFFECTING GELATINIZATION

Several factors affecting gelatinization are as follows;

Starch concentration in the slurry: the higher the consistency of the starch, the higher the concentration

Temperature: when the critical temperature of the paste is not exceeded; the higher the temperature, the faster the rate of dextrin formation.

Agitation: this implies stirring of the slurry; stirring allows for proper mixing as the process of mixing is enhanced, the kinetic energy of the medium is liberated thereby causing molecules to move randomly, as it moves in its mean straight line, they collide with one another and thereby liberating more kinetic energy which results in increase in gelatinization temperature.

pH: this is the amount of hydrogen ions (H⁺) or hydroxide (OH⁻) present in the starch slurry. If the pH is predominantly protonic (H⁺) the medium is acidic and it is highly (OH⁻) by indication it is alkaline.

Due to their presence, in exothermic reaction occur thereby raising the gelatinization temperature, the pH can be raised by adding additives such as borax, acids etc.

Retention time

This is the time by which the slurry is tested before it is gelatinized (Pascarl, 1965)

2.7.0 DEXTRINS

Dextrins were accidentally discovered in 1827 when a fire occurred in Dublin textile mill, when one of the workmen noticed that some of the unused starch had turned brown with the heat and dissolved easily in water to produce a thick adhesive paste.

Three primary group are now known and they are classified below.

2.7.1 CLASSIFICATION OF DEXTRIN BRITISH GUMS

These are formed by heating starch alone or in the presence of a small amount of alkaline buffer salts to a temperature range of about 180^o-220^oC.

The final product range in color from light to very dark brown, they give aqueous solutions whose viscosity are lower than starch.

White dextrins

Prepared by mild heating of starch with a relatively large amount of added catalyst such as HCl at a low temperature of 80-120^oC for short periods of time. The final product is almost white in color, has a limited solubility in water and retains varying degrees of the set back tendency from the original paste.

Yellow dextrins

Lower acid or catalyst levels are used with higher temperature of conversion 150-220^oC for longer conversion times. They are soluble in water, form solution of low viscosity and have a color from light yellow to brown (www.wikipedia.com).

2.8.0 (GUM) ADHESIVE

This is a material obtained by free radical mechanism, capable of holding materials together by surface attachment. Adhesive can be produced using different raw materials such as cassava, potato, corn etc, production of gum depends on it's use. Eg gum production for textile industry is different from that of paper industry.

Adhesive include cement, epoxy, butyric epoxy, araldite, glue etc.

There are various types of adhesive which gives rise to different nomenclature

2.8.1 WATER SOLUBLE POLAR ADHESIVE

These are dextrins converted to gum in which hydroxyl group is predominant, in this type, solubilization with water is predominant

2.8.2 WATER INSOLUBLE POLAR ADHESIVE

These are obtained from polymers which have a linear structure eg synthetic rubbers cellulose, acetate or nitrate, epoxy and resins, there is presence of dipoles and this provides attractive force capable of attracting or holding two surface together.

2.8.3 NON-POLAR ADHESIVE

These ones are obtained at temperatures below 40°C after emulsification of naphtha monomers eg PVC, glue, superglue mortar betec etc. they have the highest bonding capacity than any type of gum

2.9.0 STABILIZATION OF ADHESIVE

Dextrin when exposed to the atmosphere for a long time absorbs moisture from the atmosphere and loose it's bonding and thickness quality therefore it is necessary to add certain additives to modify and maintain these properties for longer periods so that the life span of the gum may be long without deterioration, and this process is known as stabilization.

The following are the necessary additive for stabilization of gum

2.9.1 PLASTICIZERS

These improves the drying rate of gum and also make it slippery, lustrous due to the alcoholic content and shining eg glucose, glycerol, sorbitol, sugar etc.

2.9.2 HUMECTANTS AND LIQUEFYING AGENTS

These reagents reduces the viscosity thus increasing the flowability of the gum eg calcium chloride, urea, sodium nitrate, acetamide etc.

2.9.3 PRESERVATIVES

This help to preserve the starch from bacterial and fungal attack thereby increase it's life span eg sodium hypochloride, phenols formaldehyde, copper sulphate etc.

2.9.4 DEFOAMERS

These are to prevent foaming of the product eg methylated spirit.

2.9.5 SOLUBILIZERS

X

These act as a solvent base for the starch powder and helps to speed reaction between the slurry and chemical additives eg water and for non-polar adhesive we have phenols hexane and petrol

2.9.6 MINERAL FILLERS

This increase penetration into porous substance by acting as a selant or barrier eg kaolin clay, calcium carbonate, titanium aroxide (www.wikipedia.org/wiki/dextrin).

2.10.0 EVALUATION OF ADHESIVE

This is the consideration of how effective a particular adhesive is, a good adhesive should be able to last at list for a year.

The choice of adhesive depends on the following

- 1.the duration of the adhesive
- 2. the performance during surface attachment
- 3. ability to resist distruction after binding
- 4. corrosivity and toxicity

2.10.1 ADHESIVE FORMULATION

The method by which each part of the additives is added to starch to yield gum

Below are the table of formulation using different additive to achive gum for different purpose.

X **Table 2.4** ^P **percentage composition of kraft gum**

| component | Part by weight |
|----------------|----------------|
| Corn starch | 80 |
| Bantonite clay | 10 |
| Borax | 17 |
| Formaldelirae | 1 |
| X Water | 880 |

By Austin,1984.

Table 2.5 Formulation of gum using starch

| component | Percentage composition by weight |
|-------------------|----------------------------------|
| Water | 79% |
| Starch | 10% |
| Glycerol | 3% |
| Urea | 2% |
| Borax | 2% |
| Formaldehyde | 25% |
| Methylated spirit | 1.5% |
| | 100% |

By Austin, 1984.

CHAPTER THREE

3.0 MATERIALS AND METHOD

3.1 MATERIALS

The materials, chemicals or reagent, used to achieve the set goal of this research work is stated below

- I. cassava starch
- II. dextrin
- III. hydrochloric acid (HCl)
- IV. distilled water
- V. trioxonitrate v acid (HNO_3)
- VI. pulp
- VII. calcium chloride (CaCl_2)
- VIII. methylated spirit
- IX. starch fibre

3.2 EQUIPMENT

The equipment/apparatus used for the production and preservation of a water resistance gum from dextrin is as follows

- X I. 200mm sieve
- II. Metallic trays
- III. Electronic Sieving machine
- X IV. 300ml beaker
- V. Thermometer (liquid in glass 900°C)
- VI. Measuring cylinder (graduated to 1000cm)
- VII. Electric oven
- VIII. Electronic weighing balance
- IX. Grinding machine
- X. Water bath
- XI. Sieve shaker
- XII. Viscometer
- XIII. Electronic stirrer
- XIV. Stop watch (timer)

3.3 METHODS

The methods used for the production of adhesive gum from cassava starch was based on experimental procedures illustrated bellow:

3.3.1 PROCEDURE FOR PRODUCTION OF ADHESIVE GUM

Production of adhesive gum from cassava starch routed from the pretreatment of cassava tuber to the final stage of gum extraction. This involves the following operations.

- a. Peeling and washing
- b. Rasping and grating
- c. Screening
- d. Sedimentation
- e. Drying
- f. Frying

3.3.2 PEELING AND WASHING

The operation began by peeling the cassava roots using a knife and after the peeling they were washed to remove any dirt that may be clinging to the body

3.3.3 GRATING

After the above operation, the size was reduced by pressing the roots against a swift moving surface provided with sharp protrusions, the cell walls were turn up and the whole root turned into a mass in which the greater part of the starch granules was released.

3.3.4 SCREENING

In separating the pulp from the free starch a liberal amount of water was added to the pulp as it is delivered by the rasper or grater and the resulting suspension was stirred vigorously and then screening was carried out using white cloth well constructed on a wood.

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3.3.5 SETTLING AND DECANTING (SEDIMENTATION)

The pulp was mixed with a liter of water and a small amount of dilute hydrochloric acid was added to enhance the whiteness of the product as well as quicken the rate of sedimentation. This HCl acid is removed after or more subsequent settling in pure water(Hossaina,2002).

After about four hours, the pulp was settled at the bottom of the container and the water phase had become clear and transparent which implies that the water phase can be decanted, after decanting a very white and fine pulp was obtained, the pulp was weighed and the weight was 311.5gram.

3.3.6 DRYING

Drying is either done in the open air or in the oven and it is necessary to dry the pulp in order to remove the relatively small moisture present in the pulp. Hence, drying was carried out in a dryer.

The wet pulp was separated into 3 ^{trays} try with

- X ^{Tray} Try A containing 123g
- X Try B containing 125g
- X Try C containing 123.5g

When the machine was switched on the temperature read 33°C and it was gradually increased after about four hours at temperatures *between* 69°C and 65°C the weight of the pulp become constant and when weighed

Tray A contained pulp weighing 95g

Tray B contained pulp weighing 97g

Tray C contained pulp weighing 95g

This temperature was increased to 70°C and the weight still remained constant then they were removed from the dryer.

3.3.7 FRYING

It was important to fry the powder of starch in order to increase the adhesive potential of the starch but a direct heating must be avoided, hence a bath containing slack oil (used or spent oil) was used to indirectly heat the starch. Though with a little success (due to the dangerous effects of the vapour given off by the boiling oil).

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Another method was then used in which the container of the powder did not have a direct contact with the hot phase used (in this case air was the medium). The frying went on for six hours and when the first change in colour was noticed (from pure white to milk colour) the frying was stopped. The temperature of the powder was taken at five different points and they ranged between 68°C and 70°C.

When the weight of the fixed powder was taken, it was just 3g

The fried powder was further guided giving it a more finer texture and when left to cool to the atmospheric temperature for some days preferably about 48 hours, and stored in an opened container (Prescal, 1998)

3.3.8 SOURCES OF EXPERIMENT ERROR

In order to obtain a good gum from cassava starch, the following precaution were strictly adhered to right from the start of the experiment

The peeling washing, grating and decanting were done the same day because the processing of cassava has to be completed within a short time as possible, this pertains particularly to the separation of the free starch from it's suspension in the 'fruit water' since in this solution processes are soon very noticeable either of a physio-chemical or enzymic nature or both. In the first instance, the process lead^s to the formation of very stable complexes, between starch proteins, fatty materials and so on from these complexes, it is almost impossible to separated the pure starch.

At a later stage the fruit water being rather rich in supers and other nutrients, micro organisms' stars to develop which eventually lead to a vigorous fermentation.

Alcohol and organic acids are produced among which butyric acid is particularly noticeable on account of its odour.

3.3.9 PRECAUTION

The roots were not left in a wet area to prevent their getting rotten

The screening was done using a well constructed sieve so that the starch fibres cannot pass through with the pulp.

Dilute HCl was used, this was to prevent any sort of reaction between the pulp and the acid and it was immediately washed out by a subsequent settling in pure water

The fried powder was not closely packed in a polythene (tightened) to prevent the formation of moisture into the powder.

Methylated spirit was not added while boiling the starch because it is highly volatile

Calcium chloride was grinded properly to a fine texture so that it dissolved quickly in the mixture.

During drying the temperature was monitored properly to avoid excessive heat which may destroy the properties of the starch powder.

Direct heating was avoided when frying so that certain properties of the dextrin was not destroyed.

While frying the powder, it was continuously agitated to enhance even distribution of heat to all the powder.

The additives were not separately heated so as not to change the composition of the various additives before adding them to the gelatinized starch.

A good precaution was also taken while observing the period for the change in colour of the powder, so that the powder was not allowed to burn.

3.3.9 LIMITATION

Water bath was not available to enhance a uniform and evenly distributed heat during frying and gelatinization. Hence, errors were encountered during the frying in the form of panlid buring of the powder and non homogenous gelatinization of the dextrin.

The use of manual stirre (only spatula was provided) did not allow for proper stirring of the powder during frying and during gelatinization process therefore there was no uniform mixing of the gel.

A limited number of additives were provided therefore a lengthy analysis was restricted.

Because of the need for a mechanical grinder to grind certain additives to their fines grain for easy solubility (eg calcium chloride) in water the grinding mechine in the laboratory has been used to grind different material, thus implying that the possibility of contamination of the material which may in turn affect the entire experiment partially and resulting in one error or the other.

3.4.0 PHYSICAL CHARACTERIZATION

On the adhesive gum produced, the following physical characterization could be further carried out to authenticate the quality of the gum produce and the validity of reagent used

- a) Gelatinization and stabilization
- b) Viscosity test

- c) pH
- d) bindability

3.4.1 GELATINIZATION

To carry out this process, a known amount of water (in weight %) was used, a measured quantity of the dextrin (powder) and known weights of other additives which include formaldehyde, methylated spirit, calcium chloride, sodium benzoate were added in a proper order.

The mixture was heated gradually and stirred properly resulting in a viscous and colloidal compound whose viscosity continued to increase with increase in temperature. When the temperature was about 70°C the gum produced was noticed to produce a slight change in its original colour (becoming less transparent and having reduced the viscosity) the heating (indirect) was stopped while the stirring continued to avoid hardening of some portions of the gum (prescal, 1998)

3.4.2 STABILIZATION TEMPERATURE

The gum produced was divided into three equal parts. Each of the samples was tested for temperature of gelatinization, this of course was determined while cooking the slurry.

Test was also carried out on tackiness, bindability and this was done by spreading each sample (of known weight) on a measured portion of a paper and then press to adhere into wood of known weight. After about twelve hours the particular wood was left with each sample randomly. Some left the wood with detaching, some detached while some did not completely. The various samples were also tested for bindability on paper to paper, paper to carton, paper to plastic and paper to bottle.

Test on flowability was also carried out on a solid material (wood), the material was marked and inclined at an angle and the rate of flow for the different sample was noted, stop watch was used to state the time taken

The sample was test for pH, in this case a pH meter was employed to obtain an accurate value, tests on spreadability was carried out on material like carton, paper, wood, glass, plastic.

The remaining portions of each sample was left to investigate the bacterial and fungal actions as well as other macroscopic organism that can possibly attack the sample.

3.5.0 CHEMICAL CHARACTERIZATION

The chemical analysis carried out on the gum sample produced is

- a. Process variable distribution
- b. Concentration variation

3.5.1 PROCESS VARIABLE DISTRIBUTION

The experimental design table that was chosen was used to ensure proper distribution of the process variables to arrive at a reasonable conclusion for the optimum condition for the production of starch with respect to the chosen variables i.e concentration of acid, roasting time and roasting temperature the eight run screening design algorithm was used.

The extremes of the ranges of the variables represent the low and high factor levels. The eight runs are useful for the estimation of main effect and interactions. The response error could be estimated by adding a third level of each factor which is known as (cp). The centre point of each factor is located at the centroid of the factor range

3.5.2 CONCENTRATION VARIATION

Fifty grams of dried cassava starch was weighed into a beaker and then sprayed with 0.3M HCl and vigorously stirred.

It was allowed to age for 24 hours

It was then heated at a temperature of 60°C and 60 minutes after which a sticky ash brown solution was formed.

The content was then cooled in a water bath until the temperature of the pasty mass was the same as the room temperature.

This same procedure was repeated for the remaining experiment but the concentration of acid, roasting temperature and roasting time were now varied for the three type of dextrin.

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CHAPTER FOUR

4.0 RESULT AND DISCUSSION

4.1 RESULTS

The results obtained from the experimental procedures on the product of destrins are presented in Table 4.1 to Table 4.5 below:

LIST OF TABLES:

Table 4.1 different formulations

| Sample composition | A wt % | A1 wt % | B wt % | B2 wt % | C wt % | D wt % | E wt % | F wt % | G wt % | J wt % | H wt % |
|--------------------|--------|---------|--------|---------|--------|--------|--------|--------|--------|--------|--------|
| Starch | 10.0 | 8.0 | 8.0 | 8.0 | 6.0 | 10.0 | 10.0 | 10.0 | 10.0 | 4.0 | 8.0 |
| Water | 78.0 | 92.0 | 78.0 | 84.0 | 78.0 | 80.0 | 82.0 | 82.0 | 81.0 | 91.0 | 81.0 |
| Calcium chloride | 4.0 | - | 4.0 | 2.0 | 5.0 | 3.0 | 3.0 | - | 4.0 | 1.0 | 1.0 |
| Methlated spirit | 3.0 | - | 3.0 | 2.0 | 3.0 | 3.0 | 3.0 | 3.0 | 3.0 | 1.0 | 1.0 |
| Sodium benzoate | 3.0 | - | 5.0 | 1.0 | 6.0 | 1.0 | 3.0 | 3.0 | - | 2.0 | 2.0 |
| Formaldehyde | 2.0 | - | 2.0 | 3.0 | 2.0 | 3.0 | 1.0 | 2.0 | 2.0 | 1.0 | 2.0 |
| Glycerol | - | - | - | - | - | - | - | - | - | - | 2.0 |
| total | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 | 100 |

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X TABLE 4.2 Physical characterization of adhesive gum

| samples | pH | Viscosity (cp) | bindability | flowability | spreadability | Strength development | %moisture content |
|---------|-----|----------------|-------------|-------------|---------------|----------------------|-------------------|
| A | 7.2 | 0.25 | v.good | Low | High | Good | 0.2 |
| A1 | 6.9 | 0.20 | Good | Fair | High | Good | 0.6 |
| B | 7.3 | 0.06 | Fair | Low | High | Fair | 1.2 |
| B2 | 7.6 | 0.18 | Good | Fair | High | Good | 0.8 |
| C | 7.2 | 0.02 | Poor | Poor | Poor | Low | 1.42 |
| D | 7.4 | 0.06 | Fair | Low | Fair | Fair | 1.32 |
| E | 7.2 | 0.05 | Fair | Fair | Fair | Fair | 1.38 |
| F | 7.3 | 0.04 | Fair | Poor | Fair | Fair | 1.72 |
| G | 7.5 | 0.18 | Good | Fair | Fair | Good | 0.8 |
| H | 7.7 | 0.16 | Good | Good | Fair | Fair | 0.85 |
| | 2 | 1 | 4 | 5 | 6 | 7 | 3 |

TABLE 4.3 Dextrinization process parameters for white dextrins

| run | Roasting temperature(°C) | Conc. Of acid used (M) | Raosting time (min.) | Relative solubility (%) |
|-----|--------------------------|------------------------|----------------------|-------------------------|
| 1 | 60 | 0.300 | 30 | 36.1 |
| 2 | 80 | 0.300 | 30 | 33.0 |
| 3 | 60 | 0.350 | 30 | 32.0 |
| 4 | 80 | 0.350 | 30 | 32.0 |
| 5 | 60 | 0.300 | 90 | 33.6 |
| 6 | 80 | 0.300 | 90 | 34.2 |
| 7 | 60 | 0.350 | 90 | 33.8 |
| 8 | 80 | 0.350 | 90 | 33.5 |
| 9 | 70 | 0.325 | 60 | 31.0 |
| 10 | 70 | 0.325 | 60 | 30.0 |
| 11 | 70 | 0.326 | 60 | 28.2 |

Estimate of response error using replicate

$$Y = \frac{\sum Y_t}{r} = \frac{31+30+28.2}{3} = 29.73\%$$

$$S = \frac{\sqrt{(31-29.73)^2 + (30-29.73)^2 + (28.2-29.73)^2}}{3-1} = \frac{\sqrt{4.0267}}{2} = \frac{2.006}{2} = 1.003\%$$

with $(r - 1) = 3 - 1 = 2$ degree^s of freedom

TABLE 4.4 Dextrinization process parameters for Yellow dextrin

| run | Roasting temperature(°C) | Conc. Of acid used (M) | Raosting time (min.) | Relative solubility (%) |
|-----|--------------------------|------------------------|----------------------|-------------------------|
| 1 | 70 | 0.250 | 60 | 12.00 |
| 2 | 90 | 0.250 | 60 | 18.00 |
| 3 | 70 | 0.300 | 60 | 16.18 |
| 4 | 90 | 0.300 | 60 | 21.80 |
| 5 | 70 | 0.250 | 180 | 18.00 |
| 6 | 90 | 0.250 | 180 | 15.60 |
| 7 | 70 | 0.300 | 180 | 19.30 |
| 8 | 90 | 0.300 | 180 | 32.16 |
| 9 | 80 | 0.275 | 120 | 21.10 |
| 10 | 80 | 0.275 | 120 | 20.00 |
| 11 | 80 | 0.276 | 120 | 18.40 |

$$Y = \frac{\sum Y_t}{r} = \frac{21.1+20+18.4}{3} = \frac{59.5}{3} = 19.83\%$$

$$S = \frac{\sqrt{(21.1-19.83)^2+(20-19.83)^2+(18.4-19.33)^2}}{3-1} = \frac{\sqrt{2.5067}}{2} = 0.79\%$$

X with $(r - 1) = 3 - 1 = 2$ degree^s of freedom
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Table 4.5 Dextrinization process parameters for British gum

| run | Roasting temperature(°C) | Conc. Of acid used (M) | Roasting time (min.) | Relative solubility (%) |
|-----|--------------------------|------------------------|----------------------|-------------------------|
| 1 | 100 | 0.230 | 60 | 18.8 |
| 2 | 120 | 0.230 | 60 | 19.2 |
| 3 | 100 | 0.280 | 60 | 18.7 |
| 4 | 120 | 0.280 | 60 | 15.8 |
| 5 | 100 | 0.280 | 180 | 14.9 |
| 6 | 120 | 0.230 | 180 | 19.7 |
| 7 | 100 | 0.280 | 180 | 15.4 |
| 8 | 120 | 0.280 | 180 | 16.4 |
| 9 | 110 | 0.285 | 180 | 15.2 |
| 10 | 110 | 0.255 | 120 | 14.8 |
| 11 | 110 | 0.255 | 120 | 18.6 |

$$Y = \frac{\sum Y_t}{r} = \frac{15.2+14.8+18.6}{3} = 16.2\%$$

$$S = \frac{\sqrt{(15.2-16.2)^2+(14.8-16.2)^2+(18.6-16.2)^2}}{3-1} = \frac{\sqrt{8.72}}{2} = \frac{2.952}{2} = 1.47\%$$

with $(r - 1) = 3 - 1 = 2$ degree^s of freedom

4.2 DISCUSSION OF RESULTS

When gum is produced without additives, a long exposure to the atmosphere causes it to deteriorate and this is accounted for as a result of bacterial and fungal attack on the gel thereby causing the starch to become less viscous, very watery and loosing it's ability to bind which is the major purpose of producing the gum.

It was noticed that the gum with additives stayed a longer time binding the materials which it was used on and when one of the materials was turn away from the other part of the material still stack to the other and this can be referred to as strength development.

Various tests were carried out trying to analyze the effect of the additives and the effectiveness of the proportion to which they were added.

From the table in Table 4.1, it will be noticed that different formulations were used some with all the additives and some without all the additives (prescal, 1998).

Tests were carried out on the viscosity of the gum and the result of each sample is as recorded in Table 4.2. Raw starch suspended in water gives rise to a more or less viscous slurries. While in some application the viscosity of these suspensions may be of some technical importance, the term viscosity of the starch flour is generally used gelatinization which is also referred to as the hot paste viscosity.

So from the point of viscosity, it was noticed that some of the formulations were good for high viscosity gum and some good for low viscosity gum, the former is good for harden materials, while the latter is good for light materials and office use.

The amount of water added to the starch flour determine the viscosity and so for the kind of gum needed, the ratio of water to starch flour should be done as in the appendix.

Water is not the only determinant of viscosity, the amount of fillar used in this case calcium chloride, sodium benzoate and if glycerol and glucose solution is added, then this can help in reducing the viscosity of a none luminous and shiny paste if not required too thick.

^{pH}
PH test is very important in the manufacturing of dextrin from cassava starch the reason being that the amount of molds or impurities in the flour and a low pH value indicating corresponds with a progressing deterioration.

From literature review, it has been found that the acid factor should be of prime importance in the of the dextrinization required the value of the acid to be between 20 and 25(Hassaina, 1981).

A gum paste is better of the factor is alkaline from the result sample had the highest alkaline factor 7.7 compared to other which make it very good for office use and remstening of envelopes in the post office, and A, without additives way very acidic and this is the reason why

after a day of production, the sample becomes very sticky to the container and after some days, started deteriorating, and becoming watery this shows a progressing deterioration by attack from bacteria due to the presence of molds, and some other impurities which were not taken care of by the preservatives.

The samples were tested for their ability to bind well on different surfaces and the following results obtained A-good, B-fair, B₂-Good, C-poor, D-poor, E-fair, F-good, G-good, H-good (table 4.1).

The result pertaining to their bindability to the different surfaces are as recorded in the appendix. Once again, these gums can serve different purposes i.e. they are made and used for the purpose for which the consumer requires it, if the consumer needs it thick for a hard material sample A suits that purpose best and if for a light material, sample B suits it best, sample B₂, J and H have similar characteristic. However, for a medium binding surface sample E is choosing.

Another analysis that was carried out to study the behavior of adhesive gum is moisture content test. This was done to remove moisture in form of water present in the starch sample used for the gum production. The dextrans with smaller moisture content have longer life span and better adhesive properties. Excess moisture in the gum allows for bacteria and fungi attack.

Flowability test was also carried out on the gum samples, through which the sample with highest viscosity flow, more reluctantly compare with the ones with smaller viscosity.

The wider the area of application the greater the spreadability sample with low viscosity were noticed to spread easily and the result is as in the appendix.

Tackiness

This is the ability for the gum to be able to adhere to the fingers when pressed together the result showed that sample A, A₁ B and B₂ were very high and D, E, F, G, H, J samples were fair while sample C was poor

Analysis of the samples for water resistance also indicated that, most of the samples are highly resistant to water except samples B and C as well as sample F whose moisture contents are extremely high and may easily get diluted on exposure to more water to lose their adhesive properties thereby, harbour fungi and bacterial. The concentration of acid added on each case was seen to have a corresponding effect on the roasting time and relative solubility of the dextrin samples used in each case through which the best solubility of 36.1% was obtained with addition of 0.3M to a white dextrans and 99.2% on degree of freedom of 2.0 in each case as shown in table 4.3.

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CHAPTER FIVE

5.0 CONCLUSION AND RECOMMENDATION

5.1 CONCLUSION

X The aim of this research work was achieved since the production and preservation of water resistance gum was successfully done. The result of experimental procedure on different gum produced from different types of dextrins: white dextrins, yellow dextrins and British dextrin yielded.

Also, analysis of various gum sample, produced made it evidential that, the higher the moisture content, the lower the viscosity and the lower the bindability of the gum produced. A gum sample labeled A was found to yield the best bindability due to relatively lower moisture content and high viscosity.

The gum sample produces was better preserved when its starch c composition relations to water (starch: water) is higher and the preservations are used in absence of glycerol.

5.2 RECOMMENDATION

The following recommendations are made for the validity of this research work:

A further research should be made on the effect of preservative chemical, on the gum samples produced.

An alternative source of starch used as raw material for gum productivity should be tested (other than cassava) to compare the effectiveness and efficiency of product.

Materials for gum production should be made available for effective research-making on the subject matter.

There should be a governmental sector that sees to the development of adhesive gum production principles in order to support the project (research) financially.

It is also recommended that, a liquid starch should be used as a starching material for gum production to study the variation of moisture content of the raw material and the product.

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