THE PHYSICO-CHEMICAL PROPERTIES AID EFFICIESCIES OF STARCHES EXTRACTED FROM SOME LOCAL CEREALS ASP TAKERS

BY

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A Thesis submitted to the Department of Textile Science and Technology, Ahmadu Bello University, Zaria in partial fulfillment of the requirements for the award of the degree of Master of Science in Textile Science and Technology.

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CERTIFICATION

This dissertation entitled "THE PHYSICO-CHEMICAL PROPERTIES AND EFFICIENCIES OF STARCHES EXTRACTED FROM SOME LOCAL CEREALS AND TUBERS", by Abiodun Ogunribido, meets the regulations governing the award of the degree of Master of Science in Textile Science and Technology of Ahmadu Bello University, and is approved for its contribution to knowledge and literary presentation.

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DEDICATION

This project is dedicated to the Lord Jesus Christ who died that I might live and rose that I may be justified.

Ogunribido, A.

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I am greatly indebted to my supervisor DR. O.K. SUNMONU whose advice and commitment to this work saw me through this work. My sincere appreciation goes to my colleague, Benjamin, whose help and patience enabled me to perform certain experiments which would have been difficult for me to perform.

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ABSTRACT

A comparative study is carried out on the physical and chemical properties of starches extracted from maize corn, guinea-corn, potato, cassava and rice and modified starches of corn and cassava. These starches were used to size cotton yarns and to stiffen cotton fabrics. Effects of the starches on the air permeability and thickness of fabric was also studied.

From the study, it is apparent that starches extracted using the traditional method of extraction could be of relatively good grade and of some industrial importance if care is taken during extraction. It is also evident that the viscosity of the starches is directly linked to the sizes of the granules of the starches.

The efficiency of the starches as sizing and stiffening agents was found to be directly related to the ability of the starch granules to penetrate the fabric or yarn as the case may be. Cereal starches gave better result than the tuber starches.

Modified starches gave very good results when employed as a size or stiffening agent and in some cases gave better results than for unmodified starches.

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CHAPTER ONE

1.0 Introduction and Literature Survey

Pure extracted starch is a white, amorphous polymer having no taste. Starch is insoluble in cold water, alcohol and chloroform but soluble in hot water[1]. On cooking, provided the starch is of sufficiently high concentrations, all free flow of water will be prevented and a gel results. Under a microscope, starch is seen to consist of granules whose general size and shape is specific for each variety of starch[2] (see table 1.2). The granules are made up of cells, each having a dark spot, central in some, eccentric in others, known as the hilum[3].

1.1 History

The first starch was probably obtained from wheat by the Egyptians as food and for binding fibres to make papyrus as early as 4000-3500 B.C[4]. Between A.D. 700 and A.D. 1300 most paper were heavily coated with starch, but the practice was abandoned towards the end of the 14th century and was not revived until modern times[4]. The use of starch in textiles began during the middle ages, when it was a common stiffening agent. By 1744 the English were using it in sizing and warp glazing[5]. Textile demands soon brought about the introduction of potato starch to supplement the starch. In 1811, the discoveries of Kirchoff with respect to glucose and the thinning of starches by enzymic action

gave great impetus to starch manufacture[4]. In Nigeria, cereals and tubers, (the major sources of starches) have been major food crops for a long time. When the cooking of whole grains was carried out, it was discovered that starch gels formed, tended to remain intact giving the texture typical of such foods[5].

1.2 SOURCES OF STARCH

Starch is a widely distributed polysaccharide in nature which is found in cereals, root crops (tubers), and to some extent in stem and fruits of certain crops[2]. It constitutes from 60-70% of the dry weight of cereal seeds and as high as 80% of the dry matter of potato, cocoyam and other tubers[2].

Table 1.1 Names of Some Nigerian Crops which contain starch[19]

Common name	Botanical Name
Corn (Maize)	Zea mays
Guinea-corn	Sorghum vulgare
Rice	Oryza sativinum
Millet	Pennisetum typhoideum
Barley	Hordeum sativum
Wheat	Tricticum sativum
Fonio (acha)	Digitaria exilis
Cassava	Manihot utilissima
Irish Potato	Solanum tuberosum

Sweet potato	Ipomoea batatas
Bitter yam	Discorea dumetorum
White yam	Discorea rotundata
Cocoyam (taro)	Colocasia esculenta
Cocoyam (tannia)	Xanthosoma sagi hifolum
Arrowroot	Maranta arroundinacea

1.3 Chemical Properties of Starch

One of the most important advances in the chemistry of starch was the realisation that it was not homogenous. It consists of a mixture of two types of polymers which differ in molecular weight and chemical structure. These polymers are amylose and amylopectin.

1.3.1 Amylose

This is the linear polymer made up of long chains of D-glucose units joined together by α-1,4-glucosidic bonds (see fig. 1.1). Common starches contain 20-30% amylose.

1.3.2 Amylopectin

This is the branched chain polymer, which in addition to normal chain of glucose units joined by α-1,4-glucosidic linkages have side chains joined to the main chain by α-1,6-glucosidic bonds. The side chains may in turn have other side chains, resulting in a network of molecules. Most starches contain about

75-80% amylopectin. Waxy type starches contain almost pure amylopectin with a very small fraction of amylose.

Fig 1.1 Amylose Molecule

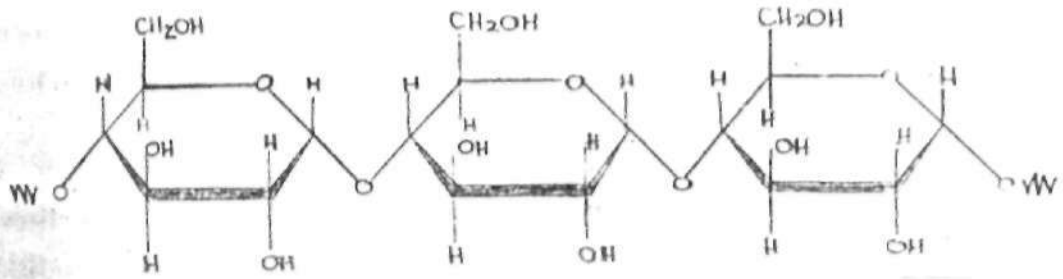
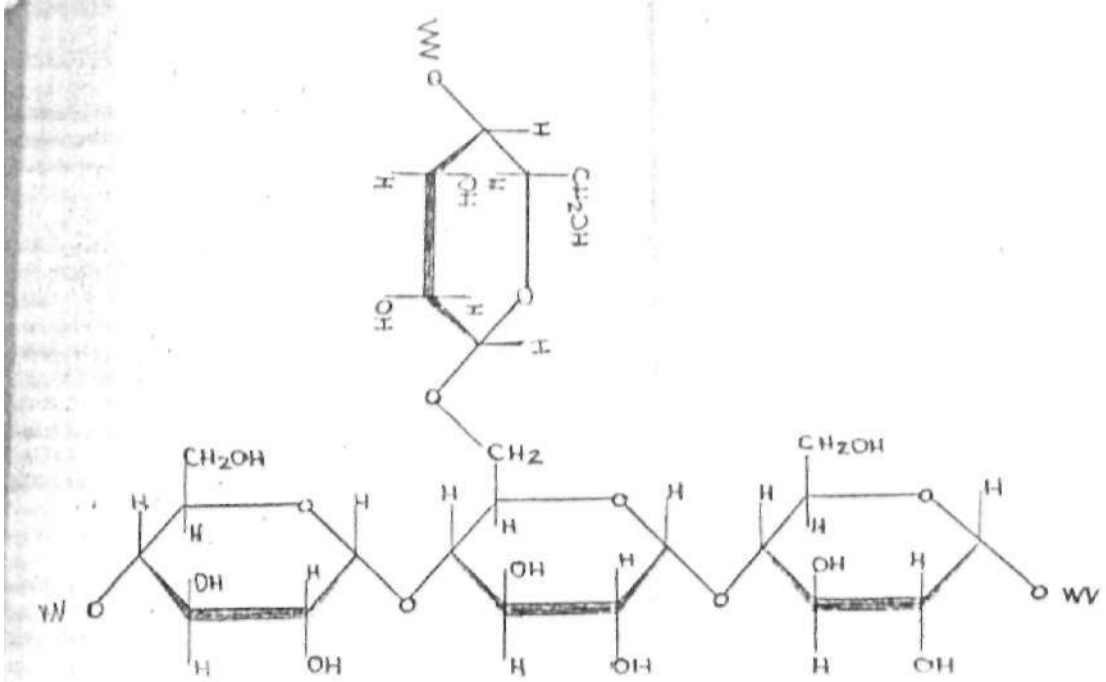


Fig 1.2 Amylopectin Molecule



1.3.3 Ash Content

All starches from cereal and tubers contain minor quantities of uncombined inorganic materials. The bulk of these originates in the crop from which the starch is isolated but a small fraction may be contributed by the water used in proc[illegible]. The approximate concentration of these salts is customarily determined as a residue after ignition at a specfied temperature [13].

1.3.4 pH of Starch

Since the pH of starch affects the physical p[illegible]ies of pastes, most unmodified starches are adjusted to a pH level of about 5.0 during the final stages of processing. Some modified and derived starches on the other hand may be adjusted to a somewhat higher pH value.

1.3.5 Nitrogen (Protein) Content

Commercial starches are recovered from cereal grains and tubers containing various concentrations of protein fibre and fat in addition to starch, together with other minor components. Recovered starch contains minor or trace amounts of protein together with trace quantities of other components in the starting material.

Protein content of starches are estimated from the determined nitrogen contents using conversion factors appropriate for the

particular crop. The Kjeldahl procedure is the most commonly used for Nitrogen content determination[12].

1.3.6 Fatty Matter Content

Cereal starches contain trace amounts of fatty acid glycerides usually less than 0.1%, these can be extracted with hydrophobic solvents such as ether, hexane or carbon tetrachloride, using conventional soxhlet or butt-type extraction equipments. Most starches also contain about 0.5-0.6% of free fatty acids which appear to be complexed with linear fraction (amylose) of the native starch. Since these complexed fatty acids alter the physical and chemical prperties of the starch it is often of interest to know their concentration and sometimes their removal is a pre-requisite for accurate analysis such as the determination of iodine affinity[12].

1.3.7 Reaction of Starches

Hydroxyl groups in starch can be oxidized by hypochlorites of sodium and potassium to form aldehydes, ketones and carboxyl groups. Oxidation is pH-dependent[5]. Oxidation does not change the appearance of the starch. The granules retain their shape and give a normal reaction with iodine.

Hydroxyl groups in starch also undergo esterification and etherification reactions. Esterification and etherification of

starches reduces the tendency of starches to retrograde and this is of interest to the textile industry[19].

1.4 Physical Properties of Starch

1.4.1 Granule Size and Shape

Starch granules from cereal grains vary between 3 and 30μm with respect to the average size of their largest diameter, and those from the roots and tubers between 10-100μm, palm (sago) starches are medium in size having a diameter of about 50μm[9].

Table 1.2 Physiological Sizes and Shapes[19]

Starch Source	Size (μm)	Shape
Maize or Corn	5-10-15-25	Polyhedral, spherical
Potato	Rounded 10 - 35	Ovoid, irregularly
	Ovoid 30 - 100	Ovoid, subspherical somewhat flattened
Rice	3-5-8-12	Polyhedral
Cassava	L = 25 - 35	Subspherical
	B = 15 - 22	
Guinea-Corn	5-15-25	Rounded shape and covered with a thin sheet of protein, granule are polygonal

1.4.2 Solubilities

Starch is a relatively inert polymer, insoluble in cold water, alcohol and ether but dissolves in potassium hydroxide solution, forming a potassium derivative. It is soluble in hot water and it's abundant hydroxyl groups make it strongly hydrophilic[20]. It is very hygroscopic, retaining when air dried, 16-18% water and when dried in a vacuum, it retains about 10% water.

1.4.3 Gelatinization

When dry starch first comes into contact with cold water it swells with no apparent change in appearance of the granules. When the suspension is heated no change is apparent until a certain critical temperature is reached. This critical temperature differs according to the source of the starch. At this temperature, some of the granules swell considerably and as the temperature rises further, more and more granules swell until finally a thick paste is formed. At this stage, gelatinization is said to be complete. The temperature range between the start of swelling and formation of paste is called gelatinization temperature range.

1.4.4 Swelling Power

When starch is pasted in hot water, the individual granules swell and a portion of the starch substance dissolves

into the surrounding aqueous medium. According to Leach[112], the swelling behaviour of starch depends primarily on the strength and character of the micellar network within the granule. The initial swelling takes place in the amorphous region of the granule. This occurs at the gelatinization temperature.

Swelling power of some starches have been shown in Table 1.3. The gelatinization properties are of considerable importance in the textile and paper industries where ease of cooking and solubility are of importance.

Table 1.3 Gelatinization temperature and swelling power of some starches[19]

Starch source	Gelatinization temperature range (°C)	Swelling Power at 95°C
Maize	62 - 72	24
Waxy maize	63 - 72	64
High amylose corn	67 - > 100	6
Wheat	52 - 63	21
Rice	61 - 78	19
Sorghum	68 - 73	22
Waxy sorghum	67.5- 74	49

1.4.5 Moisture Content

Low moisture content is important for a good storage in order to prevent decay of starch. Dry starch should be kept away from moisture to prevent the growth of fungi and decay-causing bacteria. Under average ambient temperature and humidity conditions, the equilibrium moisture content of most cereal starches is about 12%; whereas that of some potato starch is up to 18%.

1.4.6 Colour

In the food and pharmaceutical industries, pure white colour of the starch is important. Such starches are used in the formulation of drugs forms[19] and cosmetic preparations. Colour is also important in paper sizing where paper is coated with starch to give it a shiny face. Colour, particularly on stamps and pale envelopes is also an important property of starch.

1.4.7 Refractive Index

The refractive indices of certain starches are shown in Table 1.4.

Table 1.4 Refractive Indices of Starches[2]

Starch Source	Refractive Indices
Maize	1.5222
Wheat	1.5245
Potato	1.5135
Rice	1.5219

1.4.8 Gel Strength

Advances in starch gel strength have been reviewed by many workers[12,13,14]. The corn industries gelometer[13] is used for an accurate measurement of the gel strength.

Certain gel strengths at given starch concentrations are given in Table 1.5.

Table 1.5 Gel Strength of Starch-Water Paste

Starch Source	Concentration (% dry substance)	Gel Strength (g-cm)
Sweet potato	1.96	0
Sorghum	4.66	76
Rice	5.40	31
Millet	6.44	345

1.5 Uses of Starch

The most important uses of strach are industrial uses(6).

1.5.1 Food and Beverages

Although cooked starch is a good source of carbohydrate in the diet, it is rarely included in food product for this purpose. Some of the uses of starch in the food industry are;

(i) Canned and Powdered Soups

Starch is used in large quantities in the soup industry as a thickener. Here, the paste and texture of starch is important. The starch must give adequate thickening power and have a bland taste as well as the right texture to be palatable. Starches which have high amylopectin content e.g waxy maize starch, are used in condensed soups because of their ability to be diluted satisfactorily.

(ii) Ice-Cream

In ice-cream, fillers are required to increase the total solids needed for their high water holding capacity. Starch fulfils this double requirements.

(iii) Custard Powder

Starch with good colour and flavour is used to make custard powder. Custard is essentially a coloured and flavoured starch with milk as the liquid rather than water.

(iv) Bakery Products

Wheat starch has been useful in biscuit making to increase spread and crispiness. By replacing 30% of the flour with wheat starch, better working and handling characteristics are obtained in the dough and the finished pastry is more flaky and requires less shortening because of the reduced protein content.

(v) Baking powder

Baking powder consists of an acidic substance and a carbonate in dry form. When water is added, these react to form CO_2 gas which when liberated within a warm flour dough gives the leavening effect in a good loaf of bread. Starch (e.g wheat and maize starches) are used as diluent in such cases. A range of products which are called "Wort syrups" are manufactured and supplied as adjuncts to brewing industry.

1.5.3 Paper Industry

Starch is used in the paper industry in large quantities although it only represents a small fraction of the total weight of paper and board manufactured (6).

It is employed at three points during the process.

(a) At the wet end where the basic cellulosic fibre is beaten to a suitable pulp.

(b) At the size press or calenders. This is the stage at which the paper sheet or board has been formed and partially dried.

(c) At stage (b) above, or later when the pigment coatings are applied to the paper.

1.5.4 Textile Industry

Starch is being utilized in the textile industry in numerous ways. As earlier stated in 1.3.4, starch and starch derivatives are used in four general areas. These are;

(a) As a size for warp yarns

(b) As a finishing agent to change the hand and appearance of fabrics

(c) In printing paste as a thickening agent

(d) As a component in finishes to glaze and polish sewing threads

Starch is also widely used as a stiffening agent for bafrics.

(a) Sizing of warp yarns

Sizing operations are confined to the warp yarns to improve their ability to withstand the mechanical stresses and strains of the weaving process, as well as add weight to the yarns in some instances. The process of sizing is sometimes referred to as slashing (33).

Yarn spun from staple fibres such as cotton are sized with starch to increase the yarn strength 10-30% and to reduce the

fuzz of the yarn by cementing the protruding surface fibres to the body of the yarn.

To accomplish these, the size film is added as a thin coating on the surface of the spun yarn and should penetrate the yarns only far enough to provide satisfatory adhesion. If the size penetrates the spun yarn, the yarn will be too stiff. The amount of size added to the spun yarn is usually 10-15% of the weight of the yarn, though smaller amounts can be used (13).

(i) Properties of Size film

Numerous attempts have been made to relate the properties of size films to weaving performance. These screening investigations have been helpful in eliminating unsuitable sizes. However, it has not been able to select from satisfactory sizes the optimum material for weaving a particular style of fabric by testing size films, sized yarns or by small scale weaving tests (13).

Weaving performance is calculated as a percentage of the amount of fabric that could be produced when looms run continuously. In weaving, many fabric styles, weaving efficiencies of 90% to 95% are common and a difference of 1% in weaving efficiency can result in a loss or gain by the mill of thousands of naira over a one year period.

The general properties of size films needed for satisfactory weaving performance are described below.

Adhesion to fibres

This is probably the most important characteristic of a size film. If good adhesion between film and fibre is not attained, the size fim is quickly shed and does not protect the warp as it is braided during weaving. Starch film adhere well to cellulosic fibres because the two are chemically similar and hydrogen bonds probably occur between them (13). The extent to which the size is coated affects its film forming properties and adhesive properties. The viscosity of the solution influences the extent to which the size film penetrates the yarn so that poor penetration of the fibre is associated with excess shedding during weaving.

Abrasion Resistance

As previously mentioned, the primary purpose for sizing is to provide a smooth abrasion-resistant surface to protect the yarn during weavinng. The size film must be hard enough to resist abrasion but soft enough to flex and stretch as the yarns are processed. The type of starch used and the material added during cooking are selected to achieve the proper balance of properties to ensure abrasion resistance of sized yarns.

Ease of removal

Another important property of size film is its ease of removal during wet-processing. Starch films are moderately easy to remove with enzyme or acids and usually cause little

difficulty in wet processing. Hard size deposits created when starch is coagulated during sizing can create areas that resist size removal as can over-drying on the steam cylinders after the size solution is applied. Modified starches are more readily removed than unmodified starch and are sometimes used for this reason.

(ii) Preparation of Warp Sizes

Warp sizes usually contain three ingredients;

(a) Film forming materials, usually starch or a mixture of starch and derivatives, or a synthetic polymer.

(b) Film modifiers such as fats used as softeners or glycerine used as a humectant.

(c) Lubricant, such as paraffin wax.

Various methods of size preparation exists. These are: Kettle cooking, sizeometer cooking, Homogenizer cooking and Jet cooking (13).

(b) Finishing with Starch Solutions

Starch and starch derivatives are used in textile finishing operations to change the stiffness or hand of the fabric, to modify the appearance by filling the interstice, and to add weight (9). Since the finish provided by starch is temporary, its use is confined to inexpensive fabrics or to materials such as shade cloth or book bindings that are not to be washed.

Fabric finished with starch alone are quite stiff and brittle, and the finishing solutions usually contain relatively large amounts of film-modifying materials. Starch is also used in conjunction with thermoplastic or thermosetting resins to obtain a stiffened finish. When used in this fashion, it provides a permanent change in the fabric properties (13).

(c) <u>Use of starch in printing and thread glazing</u>

Thickeners are used in printing to give the printing paste the consistency necessary to produce a clean, sharply defined pattern.

Vegetable gums and derivatives of cellulose and starch are more suitable for use in printing than unmodified starch. To be useful, a thickener should not be affected by the other chemicals in the printing paste, should not affect colour or cause pigment agglomeration and should be readily removed after colour is fixed.

Starch is also used as an ingredient in the solutions applied to sewing thread in the final finishing operations. The exact composition of the solutions used are not readily available but they often contain starch, glue and wax. The solution is applied by passing the thread over a roller partially immersed in a trough containing hot solution. The thread then passes over rotating brushes as it is dried by a stream of hot air. The resulting finish improves the lustre of the thread and reduces friction during sewing operation.

1.5.5 Pharmaceutical Industry

Drugs and medicaments are often required to be taken in small but accurate doses. This is conveniently done by their adminstration in the forms of pills and tablets which contains a quantity of filler. Starch is used as a filler because it is bland, odourless and capable of digestion. Aspirin tablets are good examples of this. Starch is also used as an attractant in insecticides; as an absorbent due to it's hygroscopic characteristics; as a disintegrant to enable drugs to be taken in bits; and as face powder.

1.5.6 Adhesives

By it's nature, starch makes a good adhesive in various forms; it is used in a great variety of ways for this purpose. It is used in the manufacture of corrugated and laminated paper boards, remoistening gums and wall papers for home uses.

1.5.7 Oil Industry

When bores are being drilled for the establishment of oil wells, a composite drilling mud is pumped down through the hollow drill. The purpose of the mud is to lubricate and cool the bit. Modified starches are used in the mud to give the correct viscosity and water holding capacity.

1.6 Properties of Starches that qualify them for specific applications

Like all systems in nature, the characteristics and properties of a particular starch generally measures it's effectiveness in a particular application.

1.6.1 Rice Starch

It is used as a cosmetic dusting powder due to it's small granule size and strong covering power. It's use has been particularly recommended for infants because of it's non-toxicity and absence of irritant effect if inhaled. Due to its small granule size, it penetrates into the interstices of the fabric when used as a laundry stiffening agent.

It is used as a custard or pudding starch due to the bland flavour of it's paste and as an attractant in insecticides for the same reason.

1.6.2 Potato Starch

Potato starch pastes penetrates the fibres deep before congealing than cereal starch pastes do and consequently impart added strength. The toughness and flexibility of films of potato starch relative to other starches permits warps to be woven at lower humidity than would otherwise be possible.

The smooth clear appearance obtained with potato starch have other advantages in warp sizing. For instance, mechanical

strength (behaviour of the size during storage and usage). Potato starch has a low tendency to set-back or retrograde to a gel which is essential during plant shut down.

1.6.3 Cassava Starch

Due to it's high viscosity, ease of cooking and colour; cassava starch is used as a diluent, thickener and disintegrant in food and pharmaceutical industries.

1.6.4 Maize (Corn) and Guinea-Corn Starches

Like most cereal starches, maize and guinea-corn starches have small granule sizes, white colour (except for red guinea-corn), water resistance and smooth appearance when used in paper industry as a coating. They also give moderate penetration, and hence give the correct degree of surface loading in paper press.

1.7 Effect of Starch modfication of starch on certain properties

(a) Pre-gelatinized starches

This is the "cold water soluble" starches that have been cooked, (that is gelatinized and dried). It has been observed that the paste of the pre-gelatinized starch is not as viscous, nor as smooth in texture and is less transparent to light. This is because there is loss of some swelling power in the processing as well as a significant retrogradation.

(b) Oxidized starches

Starch can be oxidized by a number of chemical agents but commercially sodium hypochlorite is usually employed. During oxidation, aldehyde and keto groups are formed depending on the pH value at which the oxidation is carried out (15). Oxidation causes rupture of the α-glucosidic bonds between the glucopyranose units and this rapidly changes the starch to a thin boiling type. The presence of the carbonyl and carboxyl groups along the linear chains of starch prevents the association and bundling of these chains which is characteristic of amylose fraction. Therefore, the paste of an oxidized starch has lost some of it's tendency to set-back and more translucent than the parent starch. Thus, viscosity of the starch is lowered.

(c) Acid Modified Starch

An acid treatment of starch breaks the starch chains by hydrolysis at a number of points. The important effect is the breaking of the amylopectin chains in it's branched structure to give fragments which are sufficiently linear and of an effective size to form bundles and thereby increasing the tendency of the starch paste to form gel. The overall effect is that there is a reduction in the hot paste viscosity and set-back.

(d) Cationic starches

The simple reaction between 2-di-ethylamino ethyl chloride ($C_2H_5)_2N.CH_2.CH_2.Cl$) and starch under alkaline conditions

produces a starch which is a good example of an important group of cationic starches. Introducing the tertiary amino group into the starch molecule gives it the property of carrying a significant positive charge when dispersed into water.

Apart from carrying a positive charge, cationic starch ethers show marked changes in paste properties when compared with the original starch (6). The paste has a higher light transmittency and less tendency to retrograde. It also has improved water retention properties, giving a higher viscosity than the original starch.

(e) Cross-linked starches

It is possible to form bridges across the large number of chain contained in starch. Cross-linking decreases the swelling power of the starch and consequently the viscosity changes. A very small amount of cross-linking reinforces the granule and subsequent paste structure and this results in higher viscosity and increases resistance to break down.

The major disadvantages of cross-linking starches are:

(i) Stability of the starch paste at low temperatures is diminished.

(ii) Loss of light transmittency in the pastes, particularly on storage.

(f) Starch ethers

Starch ethers are prepared by the reaction of starch with ethylene oxide (6). Although the physical appearance of the starch granules is unchanged and the degree of substitution is quite low, the basic properties of the starch are altered. The pastes of the starch ethers show a greatly reduced tendency to retrograde and form a solid gel. Starch ethers have a lower pasting temperature than the parent starch.

(g) Others

Other modified starches include starch esters, dextrins, glucose syrups, and dextrose, where the chemical nature of the starch is in one way or the other altered.

1.8 Extraction of starch (standard procedure)

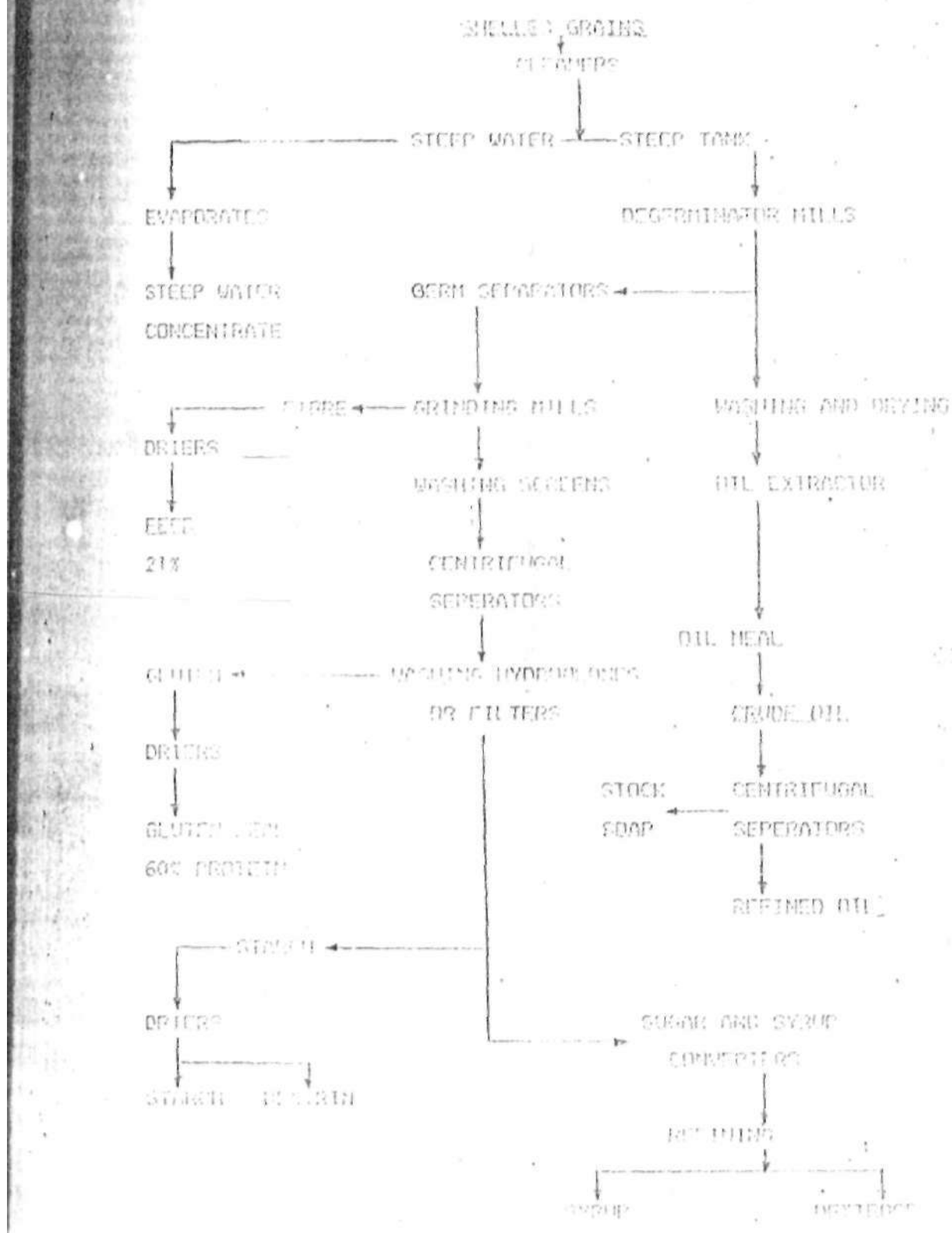
Starch from various crops are extracted in different ways.

1.8.1 Maize starch

Standard procedure for the extraction of corn and guinea-corn is the "Wet Milling Process" (13). A flow diagram indicating all the processes and end products in the wet milling process is shown in Fig 2.1.

The general steps involved in the wet milling process are outlined below;

Fig 1.3 FLOW DIAGRAM OF THE WET MILLING PROCESS (13)



(i) [illegible] GRAINS

Large and small pieces of cob, chaff and other undesirable foreign matter are removed by screening. While dust and light chaff are removed by aspiration.

(ii) STEEPING

Softening of grain to optimize milling and separation of maize components with the aid of water is referred to as steeping. To optimize starch recovery, a correct balance of water flow, temperature, SO_2 concentration and an adequate population of lactic acid bacteria must be maintained. After steeping, the grains should have absorbed 45% (wet basis) of water, released 6.0-6.5% of its dry substance solubles into the steep water, absorbed 0.2g-0.4g per kg and become sufficiently soft to yield when squeezed between fingers (13).

(iii) MILLING AND FRACTION SEPARATION

The object of milling process is to obtain as much as possible a complete separation of component parts of the maize kernels. While commercial results are unavailable, laboratory tests give the following results shown in tables 1.6a and 1.6b. The grains were steeped for 48 hrs at 52°C in 0.1% SO_2 at pH 4.

Table 1.6a LABORATORY WET-MILLING RESULTS OBTAINED WITH REGULAR AND HIGH OIL DENT MAIZE AND REGULAR RED MILO (13)

Fraction	Regular dent	High Oil dent corn (%)	Red Milo (%)
Whole grain analysis			
Moisture	14.3	13.6	14.9
starch	71.5	67.0	73.1
Protein	10.5	10.4	13.0
Fat	5.10	7.96	3.6
Wax	trace	trace	0.32
Solubles			
Yield	7.6	10.8	7.20
Protein	46.1	46.9	41.5
Starch			
Yield	63.7	59.7	60.17
Protein	0.30	0.26	0.32
Fat	0.02	0.03	0.03

Table 1.6b: LABORATORY WET-MILLING RESULTS OBTAINED WITH REGULAR AND HIGH OIL DENT MAIZE AND REGULAR RED MILD (13)

Germ			
Yield	7.3	10.9	6.17
Starch	7.6	7.2	19.1
Protein	10.7	7.2	11.9
Fat	58.9	65.5	36.7
Oil yield	4.30	7.14	2.44
Gluten			
Yield	9.5	9.8	9.30
Starch	11.4	12.3	36.7
Protein	11.3	11.0	19.7
Fat	1.8	2.7	3.8
Fibre			
Yield	9.5	9.8	9.30
Starch	11.4	12.3	36.7
Protein	11.3	11.0	19.7
Fat	1.8	2.7	3.8
Squeeses			
Yield	3.9	3.6	5.57
Starch	91.7	93.8	74.8
Protein	6.1	3.6	20.7
Fat	6.3	0.4	1.6
Total dry Substance	99.4	101.0	98.0

After steeping, the grains are coarsely ground or pulped with water in an attrition mill. The bulk of the germ is freed in the first pass but a second pass is usually provided after the free germ has been removed. Most wet milling plants have converted to the liquid cyclone method of germ recovery. The liquid cyclone or hydroclone is a conical tube about 6 inches in diameter at the top of its 3ft length. The heavier endosperm and fibre particles pass out through the bottom of the tube while the lighter germ is drawn off the vortex.

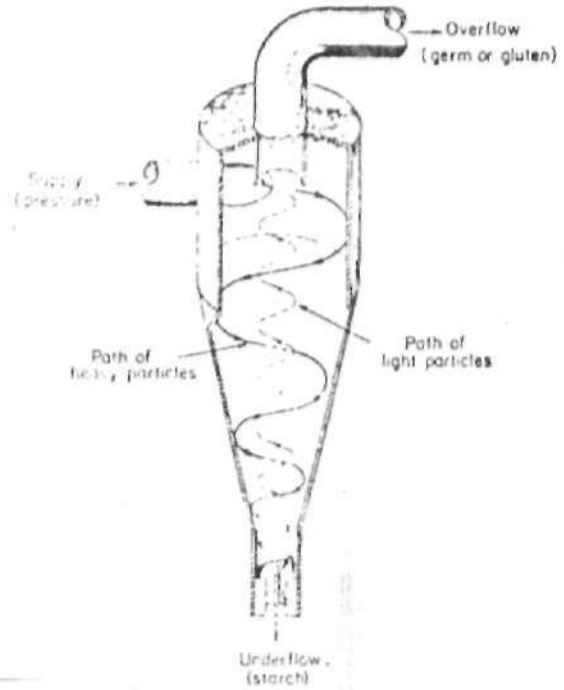


Fig 1.4 CUTAWAY DIAGRAM OF HYDROCLONE SEPARATOR (13)

The defibred mixture of starch and protein is known as "Mill starch". Protein contents varies from 5-8%. The low density of the gluten (1.1) particles as compared with starch (1.5) allows for their separation. The historic method of setting starch using tables (17) has virtually dissappeared. Centrifuges are used now in most industries. The heavier starch granules are thrown to the periphery of the centrifuge bowl and are ejected through nozzles, while the ligher are swept up by a counter current stream of water between angled separator plates and out along the central axis.

(iv) DRYING

The history of drying has been one of continued reduction in drying time. New drying procedures with reduced product retention time have given improved quality. Refined starch slurry hydroclones are first dewatered on a moving belt filter or in a basket centrifuge prior to drying. One popular starch drier employs a moving belt of metal fabric that carries the starch cake through chambers of circulated air heated to desired temperature and finally thr[illegible] cooling chamber. The entire cycle requires 20-60 minutes, depending on the nature of product being dried (13).

1.8.2 RICE STARCH

The manufacture of rice starch has been going on for many years in Europe, principally Germany, Belgium, and the Nether-

lands. From about 1932 until 1943 it was manufactured commercially in the United States.

Rice starch granules are bound into a rigid structure by protein in close association with the starch. Chemical treatment is necessary to separate this protein fraction from the starch during manufacturing process (13).

Table 1.7 CHEMICAL COMPOSITION OF BROKEN RICE (13)

CONSTITUENT	RANGE (%)	AVERAGE (%)
Ash	0.36 - 0.61	0.48
Protein	6.0 - 10.0	8.0
Lipids	0.26 - 0.95	0.54
Starch	87.2 - 93.5	90.2

Because rice starch granules are very small and are surrounded by a water insoluble protein, matrix, it is necessary to soften granules by chemical means first. Orlando Jones who operated a starch factory in London, in 1840, patented a process which embodies the general principles of the processes now in use (13).

A process now in common use is known as the ALKALI STEEP process. This involves steeping rice for 24 hrs in five times it's weight of sodium hydroxide solution which consists of 1 part of NaOH to 350 parts of water to soften and partially disintegrate the kernels. The sodium hydroxide solution is drawn

off and the kernels are then washed at least twice by vigorous stirring in excess water. The water is drawn off and the rice is dried in bags. The dried rice is then ground in an attrition mill and the finely ground flour is stirred in 10 times its weight of sodium hydroxide solution. After 20 hrs the starch is permitted to settle down for 20hrs. The major portion of the protein is contained in the supernatant sodium hydroxide solution which is carefully siphoned off. The starch is then suspended in 20 times its quantity of water and vigorously stirred. The diluted starch milk is screened off to separate whatever fibre is present. Washing, settling and siphoning are repeated and the refined starch milk is allowed to settle. The starch is then recovered and dewatered, the mass is then dried. The dried starch is crushed into lumps, packaged and shipped (13).

Other processes used in the manufacture of rice starch are the American process, village method, and the European process. All these methods make use of sodium hydroxide solution during the manufacturing process.

1.8.3 POTATO STARCH

It is difficult to define a typical composition for potatoes, however, it has been discovered from analysis of potatoes grown in Maine (U.S.A) that eastern potatoes have 18-20% of total [illegible] and Russet potatoes typically about 22%. The following may be considered as illustrative of potato composition

expressed in percentage on the dry basis; starch 75, nitrogenous compounds, 10; inorganic compounds, 15; sugars (freshly harvested potatoes), 2.5; organic acids (mainly citric), 2.5; crude fibre, 2; pectic substances, 10; fatty and waxy substances, 0.3; minor constituents, 2.0 (13).

To manufacture starch from potatoes, the potatoes are milled in water. The cells are distintegrated to liberate starch, skin and fibre are then separated from starch by screening the water slurry. Removal of water solubles is effected by washing and the remaining insoluble impurities are removed from the starch by means of gravity separation. The starch is then dewatered and dried. Time taken in the processing of potato starch is as short as 1hr or even less (13).

1.8.4 CASSAVA STARCH

Cassava (Tapioca) tubers are valuable for its large tuberous roots that are rich in starch. Most modern cassava starch manufacturing industries use fresh cassava roots grown by proven agricultural methods, however, a good quality starch has been made in some of the potato starch plants in Europe using peeled, dried cassava roots imported from Africa. This process is costly and starch from this process have different viscosities from those that are fresh.

Industrial grade cassava starch is produced in Thailand by hand labour using wooden tubs for settling the starch. The roots

are w[illegible] and partially peeled as they pass through a rotary washer-peeler. From the washer the roots fall into a grinder. The roots are torn apart to form a coarse pulp which is mixed with a little water and allowed to ferment in a cement tank or wooden tub for a day or two in order to break up the root cells and release more starch.

The fermented pulp is then poured by hand unto a shaking [illegible]th fabric that resembles a mosquito net. A spray of water over the screen washes the starch and some fibre. The starch milk is then allowed to settle in a wooden or cement tank. The su[illegible] liquid is drawn off and the starch cake dry up with wooden paddles. This starch is mixed with fresh water, usually in wooden tubs and stirred by hand with wooden paddles and allowed to settle again and the supernatant liquid is drawn off. Starch cake dug up is crumpled and spread on bamboo mats to sun-dry.

The product is not suitable for food but for certain textile sizing and for manufacturing corrugated paper boards (13).

1.9 PHYSICAL AND CHEMICAL PROPERTIES

1.9.1 pH of STARCH

pH of a starch product affects the physical properties of starch pastes. Normal pH of fresh cassava roots is 6.3-6.5 while the process water has a range of 5-6. Any cassava starch having

pH outside this range may be as a result of old or spoiled raw materials and/or other factors in the processing. On the contrary, rice starch tend to possess high pH values. This is attributed to the fact that all commercial rice starch manufacturing process involves the use of NaOH for steeping. In general, excess alkali is removed by washing with water and no attempt is made to neutralize the final product. Hence the pH of rice starch is generally around 8 (13).

Most unmodified starches are adjusted to a pH level of about 5.0 during the final stages of processing, some modified and derivatized starches on the other hand may be adjusted to a somewhat higher pH value.

1.9.2 GELATINIZATION OF STARCH

(i) THERMAL GELATINIZATION

When in water at progressively higher temperatures, the granules of starch first gelatinize and lose their polarization crosses and thereafter undergo continued swelling. Gelatinization may be defined as the loss of the interference cross visible within the granule under polarized light, and the gelatinization temperature is that point at which this transition occurs (13). While each individual granule gelatinizes quite sharly, not all the granules in a starch sample gelatinizes at the same temperature but rather over a range of some 8-10°C.

This reflects differences in the internal bonding forces within individual granules. The resulting gelatinization range is characteristic of each starch species or variety of modified starch.

Normally, the gelatinization temperature is unaffected by linear starch fraction, and hence normal and waxy starches of the same species usually gelatinize over the same temperature range (11).

See Table 1.4 for gelatinization temperature range of some starches.

Three different criteria have been used to detect the gelatinization temperature, loss of birefringence, increase in optical transmittency and rise in viscosity. The measurement of the loss of birefringence is the most sensitive, accurate and reproducible technique (11).

The Kofler microscope hot-stage, provides a simple and rapid means for detecting the loss of birefringence. With this instrument sigmoid gelatinization curves may be obtained by graphing the percentage of granules that have lost birefrigence against the temperature. In most cases it is not necessary to run the entire gelatinization curve; however, the initial and final points should always be determined.

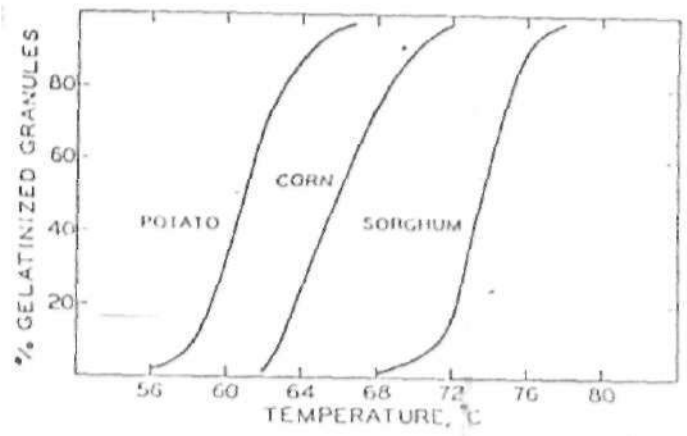


Fig 1.5. Graph of the gelatinization temperature ranges of various native starches (11)

The use of heated water bath is not often recommended. This is because temperature at the walls of the vessel will be higher than the remaining part of the vessel leading to pregelatinization of the starch granules in the vicinity of the wall.

Gelatinization temperature range may also be determined using a visco/amylo/graph. This is an instrument which plots the increase in viscosity during gelatinization. The instrument consists of a rotating stainless steel cylinder cup containing vertical pins. The cup is surrounded by a heater which transmits heat by radiation and it is by this means that the starch slurry placed in the cup is converied into a paste. A stainless steel feeler unit which dips into the paste from above when the cup is rotating, twists by an amount depending on the viscosity of the

starch under test. The movement of the feeler unit is transmitted to a balance system and recording pen. The temperature of the paste during the automatic heating phase increases at 1.5°C per minute and after a holding period at a suitable high temperature (usually 92-95°C) is cooled at the same rate to 50°C. The resulting curve which is traced by pen shows the behaviour of starch in water at various temperatures when it is subjected to a mild shearing action (11).

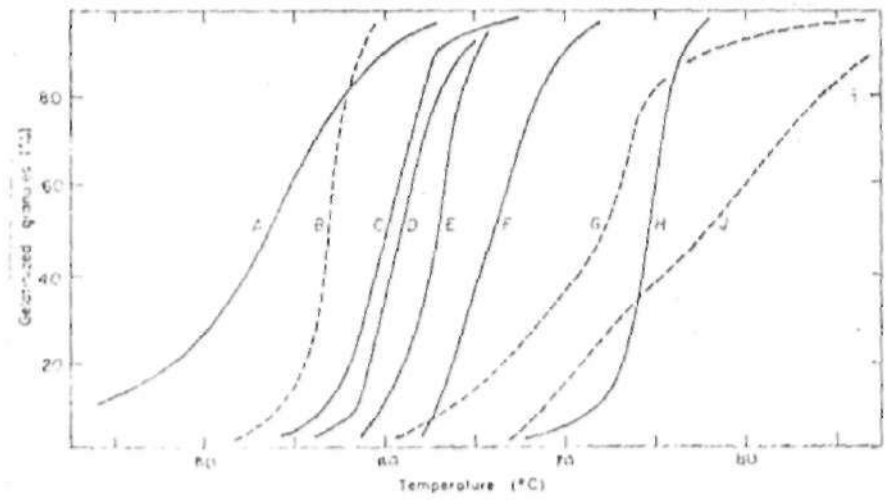


Fig 1.6 Kofler gelatinization temperature ranges of various starches (11)

A, Sulphodicarboxylic ester of corn starch; B, Barley starch; C, cationic corn starch, 0.05 D.S; D, Potato starch; E, hydroxyl ethyl sorghum, 0.1 D.S; F, corn starch; G, Cationic high amylose corn starch; H, Canadian starch; J, High amylose corn starch, 55% amylose (11)

(N.B, D.S - Degree of substitution).

Table 1.8a Kofler Gelatinization temperature of various starches (Initiation, mid-point and completion) (11)

Unmodified starches (°C)	
Maize 62-67-72	: Waxy maize 63-68-72
Sorghum 69-73.5-78	: Waxy sorghum 67.5-70.5-74
Wheat 58-61-64	: Barley 51.5-57-59.5
Tapioca (Cassava)	: Rye 57-61-70
(Brazillian) 49-57-64.5	: Pea (green garden) 57-74.5-78
(Samese) 62-68-73	: Rice 68-74.5-79
(Dominican) 58.5-64.5-70	: High amylose corn 67-80-?
Potato 59-63-68	:

Table 1.8b

Acid modified starch

40 fluidity 62-67-72

70 fluidity 63.5-69-73.5

80 fluidity 68-72-77

Oxidised maize Starch

Low converted 55-64-73

Medium converted 54-60-69

High converted 52-60-69

Cross-bonded starches

Waxy sorghum 67-75-77

Waxy maize 63-70-76

Corn 62-69-74

Cationic maize starch, high viscosity

0.04 D.S 52-58-65

0.11 D.S Room temperature

(ii) Chemical gelatinization

Aqueous solutions of certain substances gelatinize starch at room temperature. Mangels and Bailey (18) studied cold gelatinization of wheat starch in aqueous solutions of various reagents, as followed by measuring the relative viscosity of 2% starch dispersion after standing for 3 hr as 30°C. Microscopic observation indicated that gelatinization was directly correlated with granule swelling. Marked differences were observed in the reagent concentration required to induce gelatinization and in the subsequent extent of gelatinization as the reagent concentration was progressively increased. Sufficient swelling to increase the relative viscosity to a value of 4 was obtained with the following reagents at the indicated molarities. NaOH (0.09M), KOH (0.17M), sodium salicylate (0.8M), sodium, Ammonium or potassium thiocyanate (1.9-2.0M), sodium or potassium iodide (2.4M) and urea (6.2M). $CaCl_2$ solutions up to 2.4M caused pronounced granule swelling if they were allowed to act for a sufficient length of time. Also, partial gelatinization was realised when sodium or potassium bromide was used in concentrations up to 4.5M. No detectable swelling occured in 5M NaCl even after prolonged standing at 30°C. Activity of sodium compounds corresponds to the Hofmeister lyotropic series with the following anions listed in decreasing order of gelatinization power; hydroxide, salicylate, thiocyanate, iodine, bromide and chloride.

Cold gelatinization of starch is also achieved with the use of other chemicals such as aqueous solutions of Ammonium nitrate, chloral hydrate, and sodium xylene sulphonate. Aqueous alkali such as lithium hydroxide, Barium hydroxide etc are also reported to cause cold gelatinization of starch (11).

1.9.3 Viscosity of Starch Paste

The ability of starch to swell and produce a viscous paste when heated in water (or treated with certain chemicals) is its most important physical property in practical application. Consequently starch is widely used in the pasted form for a variety of purposes.

There are many methods for determining viscosity of a starch paste and the selection of the proper method will depend on the purpose of measurement.

(i) Inherent Viscosity

Treatment with HCl and oxidation with hypochlorite are the two oldest and most commonly practiced methods for starch modification. This treatment brings about significant changes in both physical and chemical properties, and changes in the former are attributed largely to reduction of molecular size. Although such changes are often detected by means of intrinsic or limiting viscosity measurements, the method is not suitable for control work nor can it be recommended for accurate appraisal of molecu-

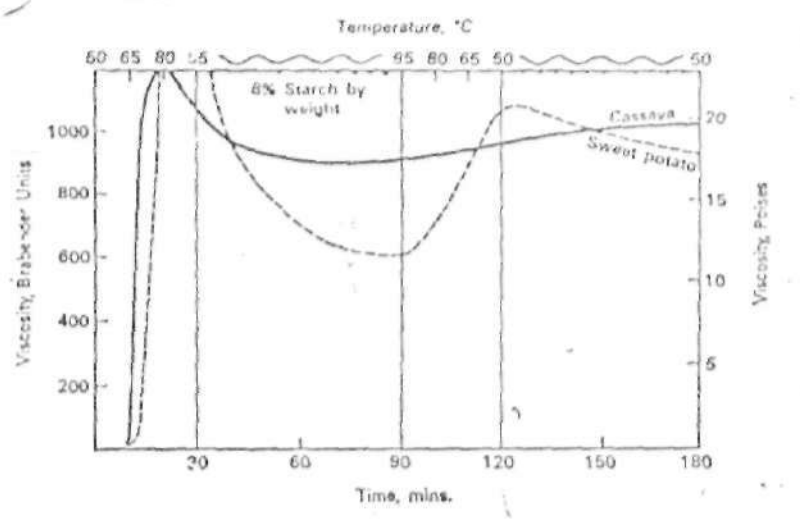


Fig 1.7 Pasting curves for starches (G)

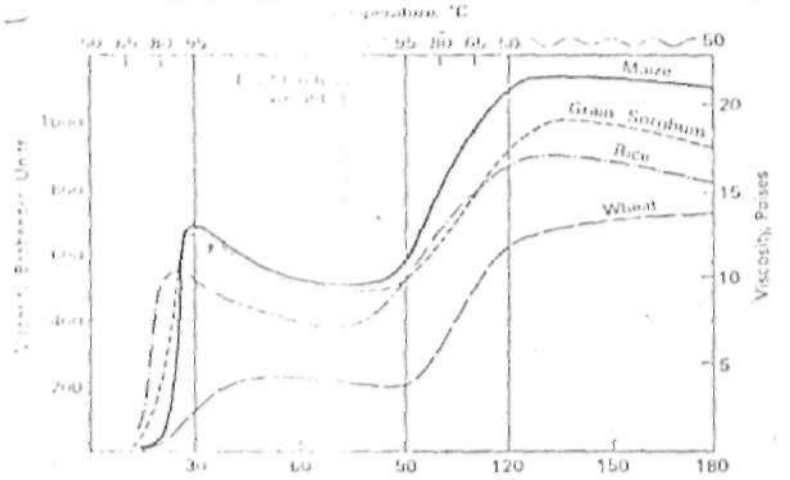


Fig 1.8 Pasting curves for starches (G)

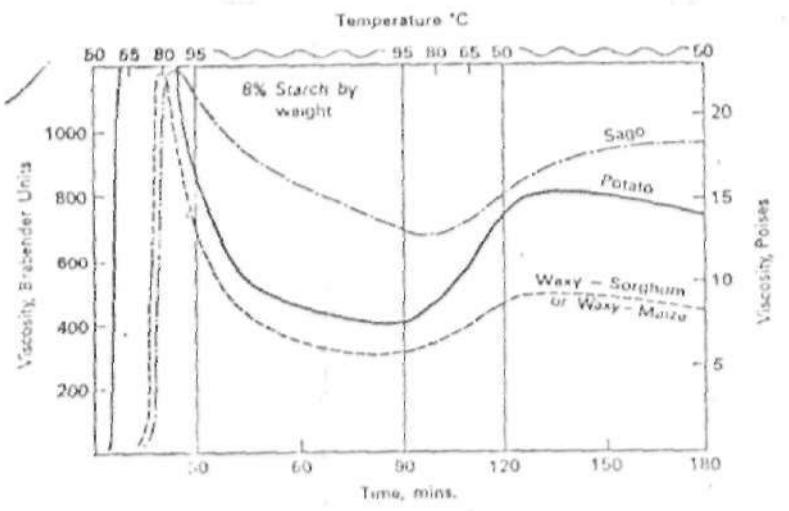


Fig 1.9 Pasting curves for starches (G)

lar size of most starches containing mixtures of amylose and amylopectin which differ in both molecular size and structure.

Limiting viscosity is useful in determining the extent of degradation occuring during starch modification involving hydrolysis. However, since the method is time consuming and not suited for control analysis, simplified procedures have been employed. One of the earliest one - point procedures was the alkali fluidity method introduced by Buel in 1912. Fluidity is the reciprocal of viscosity. Modifications of the Buel procedure are still in use. One of the most significant being that of Fetzer and Kirst (16) who selected sample concentrations related to the extent of molecular degradation. The standard procedure which was sponsored by the corn industries research foundation inc. in cooperation with the Foundation Analytical procedures sub-committee was developed so as to overcome deficiences of the former procedures. The resulting inherent viscosity method has been adopted as a standard procedure in the corn wet-milling industry.

(ii) Hot Paste Viscosity

Substantially all starch modifications involve the use of cooked pastes. Heating or cooking aqueous suspension of granular starches cause these granules to become highly hydrated and swell many times their own original volume (12). Continued cooking particularly in the presence of shear produces cooked pastes

which are mixtures of swollen granules, granule fragments and molecularly dispersed starch leached from the granules.

Changes in granule dimensions and structure during the cooking process is accompanied by significant rheological properties.

Mehods of determination of starch paste viscosities have been the subject of controversy. At present a variety of procedures based on rotational devices and flow through orifices exist. Most methods in common use measure consistency of which viscosity is the principal component (13).

Methods used by manufacturers of corn and sorghum starches are those that employ the Brabender amylograph, the corn industries viscometer, the Scott viscometer, and the brookfield viscometer. Others include the Roto visco, the Ferranti-Shirley cone-plate viscometer, the Hercules Hi-shear instrument e.t.c (13).

However, the Brabender amylograph and the corn industries viscometer are the most widely used instruments for this purpose. Each of these instruments is designed to measure the viscosity of starch paste prepared at concentration levels approaching those used in industrial applications. Viscosity curves obtained with these instruments are similar but not comparable due to differences in instrument characteristics and pasting procedures.

Proper interpretation of paste viscosity curves requires a thorough understanding of the events that occur during gelatini-

zation. When a concentrated aqueous suspension of starch is heated, the individual granules gelatinize and swell freely until they imbibe virtually all the available water. As a result, the swollen granules become increasngly susceptible to disintegration by mechanical shear. In addition, the bonding forces within the granules become tenuous, and hence, the fragility of the granule toward thermal or mechanical breakdown increases during the entire cooking cycle (11).

See fig 1.7, 1.8, and 1.9 for viscosity curves of starch from various sources at different temperatures and time (6).

The paste viscosity normally rises rapidly to a peak value (which is a measure of thickening power) and the subsequent rate of fall in viscosity is a measure of the ability of the swollen granules in the starch paste to resist thinning by prolonged heating and mechanical shear (6). The further alteration in viscosity over the holding period at the high temperature gives further information on this point. As a rule, starches that are capable of swelling to a high degree are more affected by breakdown on cooking and stirring (11). For example, maize starch exhibits a much lower viscosity than potato starch but is more stable on further heating and stirring. The maize paste consist of granules that are not very swollen in contrast to the potato in which the granules are fully stretched and swollen. Therefore, in addition to viscosity differences there are texture differences with the potato paste being long textured and stringy whereas the maize paste is short textured and heavy bodied (6).

The latter part of the Brabender viscosity curve shows the behaviour of the pastes upon cooling. Starches that exhibit set-back (or retrogradation of amylose chain) increase sharply in viscosity upon cooling. A good example of this is maize starch (6).

1.9.4 Free Swelling and Solubilities of Starch

As the temperature of an aqueous suspension of starch is raised above the gelatinization range, hydrogen bonds continue to be disrupted, water molecules become attached to the liberated hydroxyl groups, and the granule continue to swell. As a direct result of granule swelling, there is a parallel increase in starch solubility, paste clarity and paste viscosity. It is well known that the degree of swelling, the extent of solubilization, and increase in clarity are each directly dependent on pasting temperature.

To obtain fundamental information about swelling of granule, an excess of water must necessarily be present during gelatinization so that granule can swell freely without mechanical disintegration. A method that meets this requirement was used by Leah, McCown and Schoch (12) for measuring the swelling and also solubility behaviour of various types of starch.

The above method is hereby described here.

A weighed starch sample is suspended in distilled water in a centrifuge bottle. The latter is heated for 30 minutes at a

specified temperature by immersion in a thermostatically controlled bath, meanwhile stirring gently to keep starch granule suspended. The sample is then centrifuged, the aqueous supernate is removed. The swelling power is calculated as the weight of sediment per gram of dry starch, and this value is then corrected for solubles to provide a measure of swelling of the undissolved portion of the starch. Dissolved starch substance is determined directly by drying and weighing an aliquot of the supernate (11).

Swelling and solubility patterns are obtained by plotting data measured at 5°C interval over the pasting range.

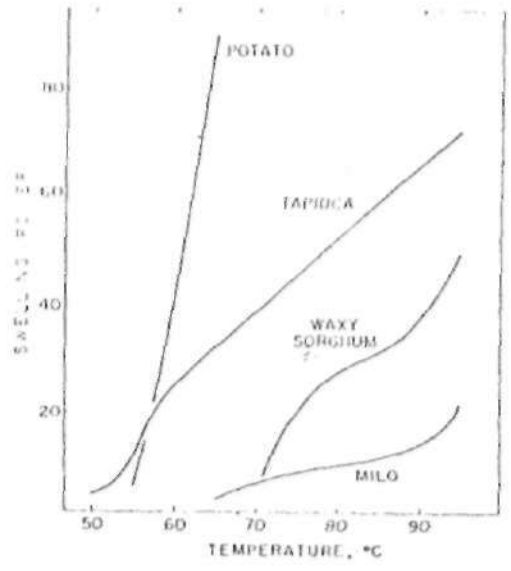


Fig 2.10 Graph of Swelling Pattern of Various unmodified starches

(11)

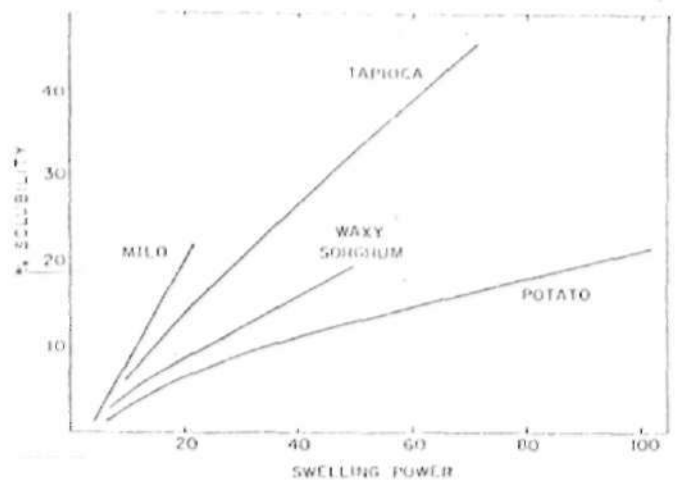


Fig 1.11 Graph of the relationship between swelling power and solubility of various unmodified starches (11)

1.9.5 Paste Clarity

On thermal gelatinization, the initial opaciity of an aqueous suspension of starch decreases and the paste becomes progressively more transparent. The quantitative relationship between extent of granule swelling and paste transmittency has been established. This was done by pasting 1% aqueous dispersion of starch at various temperatures in an identical manner to that described earlier for swelling power determination. Duplicate samples are prepared at each temperature; one for swelling power determination and the other for optical measurements. Both light transmission and light reflectance measurements are made after the paste had aged 24 hrs at 10°C. When light transmission is

plotted against swelling power, the same linear correlation was obtained for all the following unmodified starches: Arrowroot, corn, potato, waxy sorghum, rice, sago and tapioca. Hence light transmission is a direct measure of granule swelling independent of starch species, which confirms the optical method for evaluating starch pasting. Visual estimation of paste clarity is not related to light transmission but light reflectance, an entirely different property. Paste reflectance shows no correlation with granule swelling but may reflect difference in optical homogeneity within the granules with respect to uniform hydration.

Paste clarity is directly related to the state of dispersion and retrogradation tendency of the starch (11).

1.10 Starch Economics

Industrial consumption of starch has increased in recent years. In Nigeria the annual consumption of starch by industries is in the region of hunfreds of thousand of tonnes which is worth hundreds of millions of naira.

Table 1.9 Some Sectorial Requirement of Starch Products in Nigeria in 1988 (21)

	Native starch	Dextrose Anhydrous	Dextrin	Glucose	Lactose	Sucrose
Food and Beverages	15,000	15,000		10,215		
Automotive: Dry cell Batteries	702					
Cosmetrics: & toiletries	8.4					
Pharmaceuticals	800	5,470	10	30,000	865	1,110
Pulp and Paper	1,036		10			
Textiles	5,000					
Total	22,548.4	20,470	20	40,215	865	1,110

1.11 Aims and Objectives

The primary aims and objectives of this project are:

(a) To extract starch from cereals such as maize, rice and guinea corn (red), and tubers such as cassava and potato, as well as determine the percentage of starch recovered.

(b) To determine the following physical properties of the straches. (i) Colour (ii) odour (iii) moisture (iv) ash content (v) gelatinization temperature ranges (vi) description of starch paste (vii) viscosity of starch pastes.

(c) To determine the following chemical properties; (i) protein and fatty matter content (ii) pH of starch pastes.

(d) To apply starch pastes to yarn in order to determine the percentage increase in yarn strength for each starch sample.

(e) To apply starch pastes to fabrics in order to determine the increase or decrease in the following parameters.

(i) Stiffness of fabrics

(ii) Thickness of fabrics

(iii) Air permeability of the fabrics.

(f) To apply starch pastes to fabric such as "guinea brocade", Army khaki and "ankara" and carry out subjective assessment on the quality of the fabrics. The following parameters are assessed.

(i) Stiffness of fabrics

(ii) Odour of fabrics

(iii) Colour of fabrics

(g) To modify corn and cassava starches by acid-modification; and finally

(h) To determine some physical properties of the modified starches before subjecting the starches to the tests in (e) and (f).

At the end of the project it should be possible to have a clear picture of the properties of these starches and their relative efficiencies when used as a stiffening agent and for sizing warp yarns. It may also be possible to make a proper comparison between the various starches.

CHAPTER TWO

2.0 Experimental Procedures

2.1 Instrumentation and Equipments

The following equipments and instruments were used for the project;

(i) Commercial grinding machine

(ii) Nylon cloth

(iii) Weighing scale and balance

(iv) Plastic buckets and drying trays

(v) Porcelain dishes

(vi) Dessicator

(vii) Vacuum oven

(viii) Muffle furnace

(ix) Kjeldahl digestion and distillation unit

(x) Seperatory funnel

(xi) pH meter

(xii) Brookfield viscometer

(xiii) Thermometer

(xiv) Instron strength tester

(xv) Shirley bending length tester

(xvi) "Essdiel" Thickness gauge

(xvii) Shirley air permeability tester

(xviii) Stirrer

(xix) Heating mantle

(xx) Soxhlet extractor

2.2 Starch extraction

(a) Cassava

1kg of cassava flour was soaked in 10 litres of water and stirred vigorously for 10 minutes, after which the slurry was sieved using a nylon cloth. The resulting mixture was allowed to settle down for 12 hours.

The upper layer was decanted leaving the lower (starch) layer. The starch was air-dried till it was thoroughly dried, after which it was weighed, and ground to fine powder.

The percentage of starch recovered was calculated using the following equation.

$$\% \text{ of starch recovered} = \frac{\text{Wt. of starch (g)}}{\text{Wt of sample (g)}} \times 100 \quad \ldots\ldots (3.1)$$

(b) Potato

1kg of washed, and peeled potato was ground into a slurry. The slurry was filtered using a nylon cloth and the fibre washed with water to remove any remaining starch in the fibres.

The mixture was allowed to stand for 6hrs after which the upper layer was decanted leaving the starch below. The starch was thoroughly air-dried, weighed and ground into fine powder.

The percentage of starch recovered was calculated using equation 3.1.

(c) Maizeorn and Guinea-corn

1kg of maize and guinea corn grains which had been cleaned, was steeped in water containing SO_2. The maize and guinea corn

was steeped for 24 hours after which it was ground into a slurry. The slurry was filtered using a nylon cloth and the resultant mixture was allowed to stand for 12 hours to allow for the complete settling of the starch. After this, the upper protein-containing layer was decanted off leaving the starch below and the starch was dried in a tray under the sun. The starch was weighed and ground into fine powder. The same procedure was repeated for grains steeped for 48 hrs, 72hrs, 95 hrs and 115 hrs respectively.

The percentage of starch recovered was calculated using equation 3.1.

(d) Rice

1kg of rice was steeped in 5 times its weight of caustic soda solution consisting of 1 part of caustic soda to 350 parts of water to soften and partially disintegrate the kernels. The caustic soda solution was drawn off and the kernels washed twice and dried thoroughly.

The dried rice was ground to flur and the flour was stirred into 10 times its quantity of caustic soda solution. The mixture was allowed to stand for 24 hrs with stirring at intervals of between 30 minutes to 1 hr.

After 24hrs, the starch was allowed to settle for 20 hrs and the caustic soda solution siphoned off. The starch was resuspended in 20 times its quantity of water and vigorously stirred. The mixture was seperated by filtering through a nylon

cloth and the resultant mixture allowed to settle for 12 hrs before the upper layer was siphoned off and the starch was air-dried, weighed and ground into fine powder.

The percentage of starch recovered was calculated using equation 3.1.

2.3 Moisture content determination

5g of the starch sample was weighed in a pre-dried porcelain dish and placed in an oven pre-set to 120°C for 4 hrs. After this period, the dish and its starch content was weighed and the moisture content determined using the following equation;

$$\text{Moisture content (\%)} = \frac{\text{Wt. loss}}{\text{Sample Wt.}} \times 100 \quad \ldots\ldots \text{(3.2)}$$

2.4 Ash content

5g of starch sample was placed in a pre-dried, and weighed porcelain dish and heated over a hot plate until the content was thoroughly carbonized. The partially decomposed sample was placed in a muffle furnace at 525°C for 2 hours. The residue in the dish was cooled in a dessicator and weighed.

The ash content was determined using equation 3.3.

$$\text{Ash Content \%} = \frac{\text{Residue Wt.}}{\text{Sample Wt.}} \times 100 \quad \ldots\ldots \text{(3.3)}$$

2.5 Protein content determination

10g of sample was placed in a 800ml kjeldahl flask. 10g of K_2SO_4 and 60ml of conc. H_2SO_4 were added to the sample. The flask was placed in an inclined position on the kjeldahl digestion unit and heated carefully until foaming stopped. Heating was then increased briskly and the digestion was continued for 1 hour.

10ml of 1 decinormal sulphuric acid was measured into a 500ml erlenmeyer flask which served as a receiver. Distilled water was added, and the flask was connected to the kjeldahl distillation assembly so that the condender delivery tube was immersed in the absorbing acid.

After the digest had cooled, it was diluted carefully with 300ml of distilled water and mixed thoroughly. Concentrated sodium hydroxide solution was added to make the solution alkaline.

The flask was connected to the distilling unit condenser through a kjeldahl connecting bulb to prevent NaOH-containing spray from entering the condenser; the heater was turned on and the contents mixed by gentle swirling. After the reaction mixture had reached the boiling point, distillation was continued until all the ammonia had passed into the absorbing solution.

The receiving flask was removed and the excess acid titrated with 1 decinormal sodium hydroxide solution, using methyl red-bromocresol green mixed indicator.

A blank determination was done using pure sucrose instead of the starch sample. Nitrogen content was determined using the following equation;

$$\% \text{ of } N_2 = \frac{(B-A) \times \text{NaOH normality} \times 0.014 \times 100}{\text{Sample weight (g)}} \quad \ldots\ldots (3.4)$$

where

A = Sample titer value (millilitres of standard NaOH)

B = blank titer value (millilitres of standard NaOH solution)

$$\% \text{ of protein} = \% \text{ of } N_2 \times 6.25 \quad \ldots\ldots\ldots (3.5)$$

2.6 Fatty matter determination

25g of starch sample was weighed into a 600ml beaker, and this was suspended in 100ml of distilled water. When the mixture was homogenous, 300mls of boiling 4N HCl was added; the mixture was heated to boiling on a hot plate and boiled for 5 minutes. The beaker was placed in an ice-bath and cooled for 45 minutes to allow complete precipitation of fatty acids.

All the precipitate was recovered by filtering using a pre-weighed Whatman number 1 filter paper. The residue in the gravity funnel was then washed with distilled water until the washing became neutral to methyl orange indicator. The filter

paper containing the residue was folded and kept on a watch glass to dry overnight in the oven.

The folded filter paper was then extracted in a soxhlet extractor using petroleum ether. The residue was extracted for 3 hrs.

The soxhlet extraction unit was disconnected and placed in an oven for 1 hr at 100°C. The extraction unit was then cooled in a dessicator after which the filter paper containing the residue was removed from the unit and weighed. The amount of residue was then calculated by substracting the weight of the filter paper from the weight of the filter paper and residue.

The total fat content was calculated using the following equation.

$$\% \text{ total fat} = \frac{\text{Residue Wt (g)}}{\text{Sample wt (g)}} \times 100 \quad \ldots\ldots\ldots (3.6)$$

2.7 Gelatinization temperature range

10g of starch was dispersed in 200ml of water in a 500ml beaker. The mixture was heated gently in a water bath while continual stirri[illegible] maintained using the bottom of a thermometer.

The commencement of gelatinization was noticed when little bead-like lumps of gelatinized starch started to appear and the temperature at this point was noted. At the end of gelatinization

no apparent increase in viscosity was noticed and the temperature when this occurred was also noted.

The two temperature measurements were recorded as the gelatinization temperature range. this procedure was repeated for each starch sample and an average range was recorded.

2.8 pH Measurement

10g of starch sample was placed in a 400mls beaker and 200ml of distilled water was added. The mixture was stirred thoroughly and placed on a hot plate. The stirring was maintained till complete gelatinization had taken place. The starch paste was allowed to cool down to room temperature.

A pH meter that has been standardized using a buffer solution of pH 4 was used to measure the pH of the starch paste. This was repeated for all the starch samples.

2.9 Viscosity of unmodified starches

10g of starch sample was placed in a 400ml beaker and dispersed with 200ml of distilled water. The mixture was heated using a hot plate while stirring was maintained until pasting occured. The starch paste was allowed to cool down to room temperature, the hot plate was then regulated to 50°C and the mixture placed on the hot plate. On attainment of 50°C the viscosity of the hot paste was measured using a brookfield synchrolectric viscometer. The viscometer was set to 20rpm and

spindles 6 and 7 were used depending on the viscosity of the starch paste. The reading was converted to centipoise (cps) by multiplying with a factor of 500 or 100 depending on whether spindle 6 or 7 was used. Viscosity readings were also taken at 65°C, 75°C, 85°C and 95°C. This was done for all the unmodified starch samples.

2.10 Yarn Strength Measurement

A 5% wt/vol starch paste of each of the samples was used to size cotton yarns. The sized yarns were thoroughly dried and the yarn strength of the sized and unsized yarn were determined using the Instron strength tester. A number of peaks were obtained for each yarn and the average was calculated. The yarn strength was expressed in g/tex where tex is the unit of the yarn count.

The percentage increase in yarn strength was determined using the equation below;

$$\% \text{ increase in yarn strength} = \frac{(\text{yarn strength of sized yarn} - \text{yarn strength of unsized yarn})}{\text{yarn strength of unsized yarn}} \times 100$$

..... (3.6)

2.11 Fabric thickness measurements

A cotton fabric of dimension 10cm x 10cm was weighed. The thickness of the fabric was also determined using an "Essdiel thickness gauge". A number of readings were made and the average calculated.

The starch pastes in 3.10 were applied evenly on the fabric and the excess starch on the surface of the fabric was scraped off using a ruler. The fabric was dried, ironed and weighed. The thickness of the fabric was determined again using the thickness gauge.

The increase in thickness of the fabric was calculated by substracting the thickness of the starched fabric from the unstarched fabric.

2.12 Air Permeability of Fabric

The fabric used in 3.11 had its air permeability measured before the application of starch. The Shirley air permeability tester was used at 10mm H_2O. A number of readings were taken and the average calculated.

After the application of starch, the fabrics were dried, ironed and weighed. The air permeability of the starched fabrics were measured.

The decrease in air permeability was calculated by substracting the air permeaility of the starched fabric from the unstarched fabric.

2.13 Fabric Stiffness Measurement

The bending of a fabric has a direct influence on the fabric stiffness and handle. Thus, the bending length of a

fabric can be described as a measure of stiffness determining the draping qualities of a fabric (20).

On application of starch paste onto a fabric, the starch forms an intermolecular film within the fabric. This film possesses some measure of stiffness thereby resulting in the stiffness of the fabric.

In order to compare the efficiency of various starches as a stiffening agent under the same conditions, the following experiment was carried out.

Strips of cotton fabric having dimension 2.5 x 13.5cm were weighed. The bending length characteristic of the fabrics was measured using a "Shirley bending length tester". The starch paste prepared in 4.9 was applied evenly on the fabric and the excess paste was scraped off using a ruler.

The bending length of the starched fabric was also measured. (The inclined plane of the shirley bending length tester was set to an angle of 10°C).

The increase in the bending length of the fabric was calculated by substracting the bending length of the unstarched fabric from that of the starched fabric.

2.14 **Acid Modification** of Starch

30g of corn starch was dispersed in 300mls of 1.5% HCl solution, and the mixture was poured into a 500ml round-bottom flask and heated in a heating mantle at 45-50°C for 4hrs while a

stirrer continually stirred the content of the flask. At the end of this, the slurry was neutralised, filtered, washed, dried and ground into powder.

The experiment was repeated using 3.0% HCl. The whole procedure was also repeated using cassava starch.

2.15 Viscosity of Modified Starches

The viscosity of the modified starch pastes could not be measured using the brookfield viscometer because the concentration of the starch paste was too low. As a result of this, a Redwood viscometer was used to measure the viscosity of the starch paste. This viscometer uses a "flow through technique". In this apparatus, 50mls of starch paste was placed in a container in the viscometer. At the bottom of the container, there is a very small hole of known radius. The time taken for the whole liquid to pass through this hole is measured using a stop watch. Since the viscosity of the starch paste is directly related to the time taken for the liquid to pass through the small hole, it was possible to calculate the viscosity of the paste using the following equations: The whole procedure is outlined in the British Standards (B.S) No. 188, 1957.

$$\eta/\rho = At - B/t \quad \ldots\ldots\ldots\ldots (3.7)$$

where n/ρ = kinematic viscosity in stokes

n = dynamic viscosity in poise

A = 0.0026, B = 1.72,

t = time taken for the starch paste to run out of the hole in seconds

ρ = density of starch paste

The density of the starch paste was determined by using a density bottle. A pre-weighed density bottle was filled with the starch solution and all excess starch wiped off the body of the bottle. The bottle and the paste was then weighed. The density of the bottle was then calculated in g/cm^3.

2.16 pH of modified starch paste

The pH of the modified starch paste was determined using a pH meter. The experiment was similar to that carried out in section 3.8. The pH of both the neutralised and unneutralised modified starch paste was determined.

2.17 Application of Modified Starch

The modified starches were used in place of the unmodified starches to perform experiments 3.10, 3.11, 3.12, and 3.13.

2.18 Subjective Assessment of Starched fabrics

20g of starch sample was dispersed in 200ml of water in a 500ml beaker and the mixture heated until gelatinization takes place. The paste was then allowed to cool down.

Samples of "guinea brocade", army khaki and "ankara" fabrics were starched with the paste and allowed to cool down.

This was carried out using all the starch samples (modified and unmodified). The fabrics were then subjectively assessed by various people for the following parameters;

(i) Change in colour of fabric

(ii) Stiffness of fabric

(iii) Odour.

Chapter Three

3.0 Results and Discussion

Results shown in Tables 3.1 to 3.18 are for unmodified starches.

Table 3.1: Description of starch powder

Starch Source	Colour	Odour	Appearance of powder
Maize	white	odourless	fine
Cassava	white	faint odour	fine
Guinea corn	light brown: turns brown during drying	faint odour	fine
Potato	white but top layer turns brown during drying	odourless	fine (but coarse if not properly ground)
Rice	White but top layer turns light brown during drying		extremely fine

Table 3.2 Description of Starch Paste

Starch Source	Colour	Viscosity	Odour
Maize	White	high	odourless
Cassava	White	very high	Almost
Potato	Almost transparent	extremely high	Odourless
Guinea corn	Light brown	high	Odourless
Rice	White	very high	Almost odourless

Table 3.3 Percentage of starch recovered

Starch source	% of starch recovered
Maize	61%
Guinea corn	57%
Cassava-flour	60%
Potato	31%
Rice	38%

Table 3.4 Steeping time and percentage of starch recovered

	% of starch recovered	
Steeping time	Corn	Guinea-corn
24 hrs	52	54
48hrs	61	53
72hrs	57	57
95hrs	53	47
115hrs	54	50

Tale 3.5 Ash content

Starch source	Ash content (%)
Maize	0.63
Guinea corn	0.67
Cassava corn	0.67
Rice	0.47
Potato	0.37

Table 3.6 Moisture content

Starch source	Moisture content (%)
Maize	12.72
Guinea corn	10.42
Cassava	10.04
Rice	9.2
Potato	11.61

Tale 3.7 Fatty matter and Protein content

Starch source	Fatty matter	Protein content (%)
Maize	0.56	1.4
Potato	0.20	0.31
Cassava	0.35	0.82
Rice	0.63	0.94
Guinea-corn	0.67	1.04

Table 3.8 Gelatinization temperature range

Starch source	Gelatinization temperature range (°C)
Maize	64-75
Cassava	53-60
Rice	70-77
Guinea-corn	69-77
Potato	65-74

Table 3.9 pH of starch paste

Starch paste	:	pH
Maize	:	3.8
Cassava	:	4.6
Guinea-corn	:	4.6
Rice	:	8.3
Potato	:	5.8

Tale 3.10 Viscosity of Guinea-corn starch paste

% wt/vol	Temp (°C)	Viscometer dial reading	Multiplication factor	Viscosity (in cps)
5	50	39.75	100	3,975
	65	32.0	100	3,200
	75	29.5	100	2,950
	85	28.5	100	2,850
	95	27.5	100	2,750

Table 3.11 Viscosity of potato starch paste

% wt/Vol	Temp (°C)	Viscometer dial reading	Multiplication factor	Viscosity (in cps)
5	50	32.50	500	16,250
	65	29.00	500	14,500
	75	26.00	500	13,000
	85	21.00	500	10,500
	95	18.75	500	9,375

Table 3.12 Viscosity of Maize Starch paste

% Wt/Vol	Temp (°C)	Viscometer dial reading	Multiplication factor	Viscosity (in cps)
5	50	40.0	100	4,000
	65	37.65	100	3,765
	75	37.15	100	3,715
	85	34.75	100	3,475
	95	33.00	100	3,300

Table 3.13 Viscosity of Rice Starch Paste

% Wt/Vol	Temp (°C)	Viscometer dial reading	Multiplication factor	Viscosity (in cps)
5	50	5.0	500	2,500
	65	3.5	500	1,750
	75	3.0	500	1,500
	85	2.0	500	1,000
	95	1.75	500	850

Tale 3.14 Viscosity of cassava starch paste

% Wt/Vol	Temp (°C)	Viscometr dial reading	Multiplication factor	Viscosity (in cps
5	50	14.90	500	7,050
	65	12.00	500	6,000
	75	10.00	500	5,000
	85	8.40	500	4,200
	95	7.10	500	3,550

Table 3.15 Fabric Thickness Measurements

Unstarched fabric thickness = 0.36 x 0.01mm

Starch used	Thickness of starch fabric (x 0.01mm)	Increase in Thickness of starched fabrics (x 0.01mm)
Cassava	0.4325	0.072
Potato	0.460	0.100
Guinea corn	0.450	0.090
Maize	0.438	0.078
Rice	0.550	0.170

Table 3.16 Yarn strength measurement

Yarn count = 52 tex

Breaking load for unsized yarns = 400g

Yarn strength for unsized yarns = 7.69g/tex

Starch used	Breaking load (g)	Yarn strength (g/tex)	Increase in yarn strength (%)
Cassava	600	11.54	50.1
Potato	530	10.19	32.5
Maize	650	12.5	62.5
Guinea-corn	830	15.96	107.5
Rice	850	16.42	113.6

Table 3.17 Bending length measurement

Bending length of unstarched fabric = 30mm

Starch used	Bending length of starched fabric (mm)	Increase in the bending length of starched fabrics (mm)
Cassava	54	24
Potato	39.9	9.9
Rice	55.7	25.7
Maize	58.2	28.2
Guinea-corn	49.12	19.12

Table 3.18 Air Permeability of fabric

Air permeability of unstarched fabric at 10mm H_2O = 330a/sec

Starch used	Air permeability of starched fabrics (cc/sec)	Increase in air permeability of starched fabrics (cc/sec)
Cassava	255	45
Potato	200	130
Maize	170	160
Rice	226	104
Guinea-corn	205	95

4.2 Results shown in Tables 3.19 to 3.24 are for modified starches.

Table 3.19 Viscosity of 5% Wt/Vol Modified starch Pastes

(a) 1.5% cassava starch paste

Temp of paste (°C)	Time (second)	Kinematic viscosity: τ/ρ (stokes)	density of paste ρ (g/cm^3)	dynamic viscosity μ (poise)
50	57.9	0.1215	0.96	0.11664
65	52.3	0.1031	0.96	0.9897
75	51.2	0.0995	0.96	0.09555
85	50.6	0.09757	0.96	0.0937
95	51.5	0.1005	0.96	0.09648

(b) 1.5% Maize starch paste

50	48.5	0.091	0.92	0.08372
65	47.0	0.086	0.92	0.07912
75	47.0	0.086	0.92	0.07912
85	44.4	0.0767	0.92	0.07056
95	42.3	0.0638	0.92	0.0638

(c) 3.0% cassava starch paste

50	: 47.5	: 0.0873	: 0.082	: 0.94
65	: 48.0	: 0.08897	: 0.08363	: 0.94
75	: 46.5	: 0.08392	: 0.07889	: 0.94
85	: 45.0	: 0.07878	: 0.0741	: 0.94
95	: 40.7	: 0.06356	: 0.05975	: 0.94

(d) 3.0% Maize starch paste

50	: 50.5	: 0.0972	: 0.08456	: 6.87
65	: 49.8	: 0.09498	: 0.08263	: 0.87
75	: 45.8	: 0.08153	: 0.0709	: 0.87
85	: 44.0	: 0.0753	: 0.0655	: 0.87
95	: 42.5	: 0.0700	: 0.0609	: 0.87

Key

a = Cassava starch modified with 1.5% HCl at 45-50°C for 5hrs

b = Maize starch modified with 1.5% HCl at 45-50°C for 5hrs

c = Cassava starch modified with 3.0% HCl at 45-50°C for 8hrs

d = Maize starch modified with 3.0% HCl at 45-50°C for 8hrs

Table 3.20 pH of modified starch pastes

Starch source	Unneutralised strach paste	neutralised starch paste
1.5% Maize	2.1	6.9
3.0% Maize	2.15	6.69
1.5% Cassava	1.5	7.1
3.0% Cassava	1.6	6.9

Table 3.21 Air permeability of fabrics starched with unmodified starch

Air permeability of unstarched fabrics = 330 cc/sec

Starch used	Air permeability of starched fabrics (cc/sec) at 10mm H_2O	Increase in air permeability of starched fabrics
1.5% Maize	254	86
3.0% Maize	228	72
1.5% Cassava	256	44
3.0% Cassava	253	47

Table 3.22 Bending Length Measurements of fabrics starched with modified starches

Bending length of unstarched fabric = 30mm

	Bending length (mm)	Increase in bending length (mm)
1.5% Maize	42.5	12.5
3.0% Maize	48.8	18.8
1.5% Cassava	62.9	32.9
3.0% Cassava	57.3	27.3

Table 3.23 Yarn strength of yarns sized with modified starches

Yarn count = 52 tex

Breaking load for unsized yarns = 400g

Yarn strength of unsized yarns = 7.69g/tex

Starch used	Breaking load (g)	Yarn strength (g/tex)	Increase in yarn strength (%)
1.5% Maize	530	10.192	32.5
3.0% Maize	620	11.92	55.0
1.5% Cassava	750	14.42	87.6
3.0% Cassava	630	12.12	57.6

Table 3.24 Thickness of fabrics starched with modified starches

Unstarched fabric thickness = 0.36 x 0.01mm

Starch used	Thickness of starched fabric (x 0.01mm)	Increase in thickness of fabric (x 0.01mm)
1.5% maize	0.410	0.050
3.0% maize	0.455	0.095
1.5% Cassava	0.419	0.059
3.0% Cassava	0.374	0.014

3.3 Subjective Assessment of Starched fabric

(i) Stiffness

The stiffness of the fabric was found to follow this descending order;

Rice starch > Guinea corn > Cassava > Maize > Potato > 1.5% Cassava > 1.5% Maize > 3.0% Cassava > 3.0% Maize.

(ii) Odour

It was difficult to arrange the starched fabrics' odour in any order therefore different categories emphasizing the strength of perceived odour was used.

Strong odour - None

Moderate odour - Rice and potato starches

Very faint odour - Cassava, Maize and Guinea corn starches

No odour - All the modified starches

Change in Colour

Drastic change in colour - None

Moderate change in colour - None

Light change in colour - Guinea corn starch

Very faint change in colour - Rice starch

No change in colour - Potato, maize, cassava and all the modified starches.

3.4 DISCUSSION

3.4.1 Description of Starch Powder

(i) Colour

The colour of starch powder is expected to be white if that starch is to be of commercial importance. This is important because starch is often used in certain applications where it is not required to change the colour of the substrate or other additives present. Another important fact is that colour is an indication of the presence of non-starch residues particularly pigments associated with protein (13).

Table 3.1 shows that the corn and cassava starches produced are white in colour and will not require any bleaching. The light brown colouration of guinea corn starch is due to the presence of certain pigments. These pigments cannot be removed by normal processing, hence, the starch should be bleached if it is to be used for commercial purposes.

The light brown discolouration of potato and rice starch can be attributed to the processing method used. Since commercial drying and separation methods were not available, the probability of total removal of the protein component and very fast drying may be remote. The consequence of these shortcomings, especially the latter, is that, some of the starch granules may be acted

upon by micro-organisms. Another probable cause of the light brown colouration of rice and potato starch may be atmospheric oxygen oxidation or u.v/visible light degradation. It was noticed that only the top layer of the starch was light brown while the remaining [illegible]ch was white. This could be as a result of atmospheric oxygen oxidation, since the inner layers of starch were not exposed to the atmosphere. Likewise, certain chemicals may exist in trace quantities in the starches which are reactive to uv or visible radiation. These may be inorganic compounds or molecules which are broken down on exposure to the radiation, thereby resulting in the formation of different compounds or elements which have different radiation absorbing characteristics. These postulations have not been proved but the probability of the existence of such reactions is high, since potato and rice starches which were not exposed to atmospheric oxygen are with white in colour.

However, since this discolouration is very minimal, very light bleaching will take care of the colour problem.

(ii) Odour

It is of ultmost importance that starch should not possess any odour, since in most cases starch is utilized as a filler, hence it should not interfere with the properties of other additives. Another important fact is that, odour in starch is an

indication of microbial activity in the starch; odour is therefore a parameter for measuring the quality of the starch (13).

From Table 3.1, all the starches are relatively odourless. The very faint odour perceived in the case of cassava, guinea-maize and rice starch is of no real consequnce. Infact the odour will dissappear if the starch is left to dry for three days. The issue of odour would not even have arisen at all if the starches were dried using dryers since the drying time will be reduced to hours instead of days.

(iii) Appearance of powder

All starches are expected to be very fine when ground into powder. This would enhance the dispersion of the starch in water. All the starches were acceptably fine, hence the use of these starches for a variety of purposes will not be hindered.

3.4.2 Description of starch paste

(i) Colour

As earlier stated, most applications of starch require that the starch does not add any colour to the substrate. Infact, it is ideal if the starch paste is transparent as this is completely neutral in colour.

From Table 3.2, potato starch paste possessed the best colouration since it was almost transparent. Infact when the viscosity of the starch paste was reduced, it became completely transparent. The starch paste of maize, cassava and rice on the other hand were translucent and tended to be white. However on dilution with water, they tended to be transparent though the pastes were not completely transparent. In the case of guinea-corn, the paste was light-brown and this is undesirable since it will change the colour of any white substrate (e.g white cotton fabric). The rest of the starches can be used for most applications while the guinea-corn starch must be bleached before it can be used for most applications.

(ii) Odour

As in the case of 3.4.1 (iii), the starch pastes were found to be odourless or faintly scented. These starches will be acceptable for most commercial purposes if odour was used as a criteria for judging the quality of the starch paste.

3.4.3 Starch Recovery

Recovery of the starch is of importance as it indicates the viability of the process used for starch production. From Table 3.3 it is shown that maize and guinea-corn starch recovery were 61% and 57% respectively. These figures are very high since the

total amount of starch in corn grains is around 71% (see Table 1.6). The starch recovered in the case of potato is also appreciably high since the percentage of total solids in potato is around 22% (13). Hence the 21% recovery value for potato gives a recovery value of about 94% if the recovery value was based on the dry weight of the potato.

Though, the recovery value for cassava is very high (i.e 69%), it may be difficult to assess the efficiency of this method. This is because, fresh cassava was not used in this extraction and some starch must have been lost during the production of the cassava flour. Also, there are no available literature on the total amount of starch present in cassava flour.

The case of rice is quite different from the rest. From Table 1.7 it is shown that rice has an average starch content of 90%, most of which are chemically bound to proteins. To separate this rice, chemical treatment is necessary.

Commercial production of rice starch gives recovery values of 60 to 94% (13), a vast improvement over the 38% recorded during this project. This great variation can be attributed to one major factor; the absence of a very good grinding machine. The one used for this project was not able to grind the grains into fine powder, hence some of the starch particles were trapped in the broken grains.

3.4.4 Relationship between Steeping Time and Starch Recovered (%)

Steeping as already mentioned in chapter two, softens the grains and enables the starch to be easily freed from the grains. Inadequate steeping time will result in the grains not being soft enough and hence not all the starch will be liberated while excessive steeping will lead to a reduction in the amount of starch recovered due to the activity of yeast, bacteria and fungi.

Steeping time was found to be optimal between 24-48hrs in the case of maize and 48-72hrs in the case of guinea-corn. Starches produced after 72 hrs possessed an unacceptable odour which completely rules out the usability of such starches.

3.4.5 Moisture Content

The moisture content of starches is of great importance since this affects the period for which the starch can be stored. Moisture content of commercial cereal starches falls around 12% while commercial tuber starches are often higher (13).

Table 3.6 indicates that starches had moisture content between 9-13%. The moisture content of rice starch is 25% less than that of maize starch, while the moisture content of potato starch is less than that of corn by 1.11%. That of guinea corn and cassava are essentially the same.

The low moisture content of rice starch may be essentially due to the swelling restriction of the small granules hence it is less hygroscopic than potato and cassava starches. The tuber starches have large granule sizes and hence readily swell. The result of this is that such starches are more hygroscopic hence they have higher moisture content than rice starches. However, the moisture content level of potato and cassava starches is much lower than what is often obtained in most commercial starches. The low moisture content level may be attributed to thorough air drying of the starch during production and storage in an air tight polythene bag. The moisture content of the starches is quite moderate (even low in certain cases) and can therefore be used in industries where starch is often stored for long periods before use.

It is also important to state that, air drying is an effective technique for drying starch, though this might be a bit time-consuming.

3.4.6 Ash Content Determination

Apart from cassava starch, which had a high ash content, the remaining starches had relatively low level of ash content. The values obtained for the ash content of the starches with the exception of cassava starch falls within the acceptable range for commercial starches. Expected ash content for commercial

starches is around 0.2% (13). The high ash content may be due to the presence of dust and sand in the cassava flour prior to processing.

3.4.7 Fatty matter and Protein content

The presence of high values of fatty matter and protein content in starches is a direct indication of the efficiency of the processing method used. Most commercial starches often have protein contents lower than 0.7% (13). The high protein content of corn and guinea-corn starches may be linked to the fact that a simple decantation of the protein layer was performed during starch production, instead of using centrifuges as is the case in most starch industries. In the case of rice, it is likely that lack of sufficient agitation of the slurry while resuspending the slurry in aqueous alkali for 20hrs is responsible for the high protein content value. However, with the exception of pharmaceutical industries, these starches will effectively perform well when used for commercial purposes.

In the case of fatty matter, the fatty matter content recorded is not far from those found in commercial starches; commercial starches ought to have a fatty matter content not exceeding 0.6% (13) and this is correct for most of the starches produced in this study.

3.4.8 Gelatinization temperature range

The absence of a Kofler hot stage greatly reduced the possiblity of obtaining an accurate result. However, the gelatinization temperature ranges obtained were not very much different from those obtained using the Kofler hot stage. A good illustration of the method used can be seen in Table 4.1.

Table 4.1 Gelatinization temperature ranges of starch

Starch source	Gelatinization temp. range (°C)	
	Kofler hot stage	Project values
Maize	62-67-72	64-69.5-75
Cassava	58-64-70	53-56.5-60
Potato	59-63-68	65-69.5-74
G/corn	-	69-73-77
Rice	68-74.5-78	70-73-77

From the above table it can be seen that the gelatinization temperature ranges obtained in this project are quite similar to that obtained using the Kofler hot stage.

No correlation seem to exist between the linear fraction of the starches and their gelatinization temperature ranges. However it is noticed that the root starches possess lower gelatinization temperature ranges than the cereal starches. This may be related to the large size of their granules and high swelling power.

3.4.9 pH of starch paste

Most commercial starches possess pH values of between 5-7 with the exception of rice starch which has a pH value of 8 (13). The pH vaules in Table 3.9 show that the pH of rice falls within the expected range while that of maize, cassava and guinea-corn starches were lower than expected. The low pH of cassava, maize and guinea corn starches may be attributed to the long drying time, thus resulting in bacteria and yeast liberating large amount of lactic acid. In addition to this, the extremely low value of corn starch is primarily due to the use of old corn grains to produce the starch. This low pH value would have been avoided if fresh grains were used.

In most commercial starch-producing industries, the pH of the starch pastes are often adjusted to fall between pH 5-7, even if they originally were lower or higher than that value.

The pH values recorded for the unneutralised modified starch pastes were much lower than that of their unmodified counterparts. This is attributed to the use of HCl during modification. It was observed that the pH of the starch paste was dependent on the type of starch rather than the concentration of the HCl used during modification. From Table 3.20 it can be seen that the pH values for modified maize starches were virtually the same for both 1.5% maize and 3.0% maize starches while the pH value of 1.5% cassava and 3.0% cassava starches were relatively lower. Likewise, the pH values of 1.5% cassava and 3.0% cassava starches were very similar. This phenomena indicates

that the pH of starch pastes is dependent on certain parameters which are dependent on the starch source.

On neutralisation all starch pastes had pH values near 7.0.

3.4.10 Viscosity of starch pastes

Viscosity, is an important property of starch pastes because it affects the usability of the starch. For instance, highly viscous starch pastes make good adhesives.

Viscosities of starch pastes at different temperatures were measured and a graph of viscosity against temperature was drawn (see Fig 3.1).

Potato starch had the highest viscosity, while rice starch had the lowest at 50°C. The viscosity of the starch pastes decreased in all the starches with temperature. The bevaviour of the paste viscosity at different temperatures may be explained as follows; on pasting, the starch granules would have imbibed as much water as possible thereby resulting in the granules swelling. On cooking the paste, some retrogradation takes place, whereby some granules disintegrate due to mechanical shear.

The paste will now consist of swollen granules, granule fragment and starch leached from granules. Changes in the granule dimensions during the heating process is the[illegible]e cause of the changes in the rheological properties of the starch paste. Another important point to note is that, starch pastes

which have high viscosities possess a high rate of decrease in viscosity while those having low viscosities have a low rate of decrease in viscosity. This may be explained on the basis of swelling power and retrogradation. Starches that are capable of swelling to a high degree are more prone to break down on cooking and stirring (11). This explains why potato starch paste viscosity decreases with an average of 250cps while guinea corn starch paste viscosity decreases with an average of 100cps.

The high viscosity of potato and cassava starch pastes is also attributed to the large granule size of the starches and their high swelling power, while the small granule size and low swelling power of rice starch is responsible for its relatively low viscosity.

Visual assessment of the viscosities of the starch pastes was found to be of this order; Potato >> cassava > maize > rice > guinea corn. This agrees with the values obtained while using the brookfield viscometer.

The viscosity of the modified starch pastes are many times lower than the viscosity of the unmodified starch pastes. Fig 4.1 and Fig 4.2 shows that the viscosity of the modified starches is about 100 times lower than the viscosity of the unmodified starches. This explains why it was not possible to measure the viscosity of the starch pastes using the brookfield viscometer. This great reduction in the viscosity of the starch pastes may be attributed to the cleavage of the polymer chains of the starch particularly amylopectin chains. The result is a net linearity

in the polymer structure. The reduction in the viscosity of the modified starches is as a result of the cleavage of amylopectin bonds in the amorphous intermicellar regions of the granules. The net result will be the disintegration of this amorphous region and consequent weakening of the granule structure, permitting granule fragmentation with little swelling.

From Table 3.19, it is observed that 1.5% cassava starch paste had the highest viscosity, ranging from 0.09-0.11 poises, while the rest of the starch pastes had fairly similar viscosities ranging from 0.05-0.08 poises.

From Table 3.19 it can be observed that 1.5% cassava starch paste had a much higher viscosity than 3.0% cassava starch paste while 1.5% maize starch paste and 3.0% maize starch paste had a fairly similar viscosity, though 3% maize starch paste was slightly lower in viscosity than 1.5% maize starch. The great difference in the viscosities of 1.5% cassava starch and 3.0% cassava starch may be due to differences in the extent of cleavage of amylopectin bonds. It is likely that more disintegration of the amylopectin bonds present in the amorphous intermicellar region of the starch granules takes place in 3.0% cassava starch than in 1.5%. This will ultimately result in the lowering of the viscosity of the starch paste.

In the case of maize starch, the viscosities of 1.5% maize and 3.0% corn starch pastes are essentially similar, though 3.0% corn starch seem to possess a higher viscosity at low temperatures. However, at higher temperature 1.5% maize starch

paste has a higher viscosity than 3.0% maize starch paste.

The reason for this similarity in viscosity may be traced back also to the extent of cleavage of the bonds of amylopectin polymer. It is likely that the extent of hydrolysis of the bonds of amylopectin polymers of 1.5% maize starch is similar to that of 3.0% maize starch. This will result in the fragmentation of the starch granules in such a manner whereby the starch granules will possess similar swelling power. For such an event to occur, the amylopectin polymer in 1.5% maize starch must have disintegrated to such an extent that the starch will contain mostly linear chains. Thus, subsequent addition of acid during modification and increasing the temperature and duration of modification will not have much effect on the viscosity of the starch paste.

Another important observation that can be made is that the viscosity of all the modified starches decreased with increase in temperature, though certain deviations to this is present. The increase in viscosity with temperature in few instances may be due to experimental error. This may be as a result of error arising from the stop watch used to determine the time taken for 50mls of the starch paste to pass through a small hole. However, this increases are very small and occur in very few instances.

3.4.11 Yarn Strength Measurement

The percentage increase in the yarn strength of all the sized yarns was higher in all cases than the required 10-30%

increase in yarn strength for warp yarns. This means that, lower concentration of starch paste will be required for industral purposes.

However, since the major focus of interest is the comparison of the efficiencies of the various starches as sizing agents, the result shown in Tables 3.16 and 3.23 are still useful.

Tables 3.16 and 3.23 show that the percentage increase in yarn strength follows this order;

Rice > Guinea-corn > 1.5% Cassava > Maize > 3.0% Cassava > 3% Maize > Cassava > Potato > 1.5% Maize.

From the above order, it is apparent that cereal starches increased the yarn strength of the yarns more than starches obtained from tubers. Also, Table 3.23 shows that the increase in yarn strength observed when modified starches are used is quite different from that observed when unmodified starches are used.

The results of Tables 3.16 and 3.23 can be explained using the following postulations.

(a) The smaller the size of the granules the greater the probability of the starch granules penetrating the interstices of the yarn, hence, the more the amount of starch in the yarn the higher the yarn strength.

(b) The less viscous the starch paste, the higher the probability of the starch granules penetrating the interstices of the yarn.

(c) The greater the ability of the starch within the yarn to form a film, the higher the yarn strength of the yarns.

From Table 1.2, granular size of the starches follow this order; Potato > Cassava > Maize and Guinea Corn > Rice.

Likewise the size of the starch granules of the modified and unmodified starches is likely to follow this order: Unmodified maize starch > 1.5% maize > 3.0% maize and unmodified cassava starch > 1.5% cassava > 3.0 cassava.

This is because acid modification breaks down the starch chains by hydrolysis at different points thereby reducing the size of the starch granules. This reduction in size generally relates to the cleavage of amylopectin chains (13).

Though no established criteria for determining the film forming capability of starch exists, it is well known that viscous starch pastes often have strong films.

The first two postulations can therefore explain the lower sizing efficiencies of potato and cassava starches. Likewise the extremely high yarn strength of yarns sized with rice, guinea corn and corn starches can be attributed to these postulations.

The high strength of yarns sized with 1.5% cassava starch can be attributed to the smaller size of the granules compared to unmodified cassava starch. However, the very low value for yarns sized with 3.0% cassava can be attributed to the loss of strength in the film formed by this starch. This is as a result of loss in viscosity due to severe hydrolysis. In the case of maize

starch, the lower yarn strength of modified corn starch compared to the modified cassava starch may be attributed to the loss of film forming ability of the modified starches in the yarn. This seems to indicate that acid modification has a more profound effect in maize starch than in cassava starch. This is supported by the low viscosity of the modified corn starch compared to the viscosity of the modified cassava starch. The higher strength of yarns sized with 3.0% maize starch may be explained as follows. Since the viscosity of 3.0% maize starch paste is similar to that of 1.5% maize starch paste it is likely that both starch pastes form films that have similar strength. However, it is likely that the size of starch granules of 3% maize starch is smaller, hence it possesses a greater ability to penetrate the yarns. This will lead to a higher strength of yarns sized with 3% maize starch.

3.4.12 Bending Length Measurement

As earlier stated in section 2.13, the bending length of a fabric is a measure of the stiffness of the fabric. Thus a fabric with a very high bending length may be regarded as a stiff fabric.

From Table 3.17 fabrics starched with maize, rice and cassava starches have high bending lengths while fabrics starched with guinea corn and potato starches have low bending lengths. In the case of modified starches Table 3.22 indicates that fabrics

starched with 1.5% cassava and 3.0% cassava starches had high bending lengths while those ... ...ed with 1.5% maize, and 3.% maize starches had low bending lengths.

It is apparent that the result is similar to that found in section 3.4.11. The postulations outlined in sectin 3.4.11 is thereby applicable here with certain modifications. The major modification being that the stiffness of the fabric is not only dependent on how much the starch penetrates the interstices of the yarn but also, how much starch penetrates the pores of the fabric. The bending lengths of fabrics starched with modified starches exhibits a similar pattern to that of the yarn strength measurements in section 3.4.11. The explanation for this is similar to that given in section 3.4.11.

3.4.13 Air Permeability of Fabric

Air permeability of textile fabbrics is a means of assessing the serviceability of the farics for different end uses. The manner in which the yarn are interlaced in a woven fabric allows the presence of air spaces. The air permeability of fabrics influence properties such as warmth, protection against wind, and some industrial uses. The addition of starch onto a fabric will result in some pores or air spaces in the fabric being clogged by starch granules. The net result will be a decrease in air permeability value of the fabric. It is most likely that the reduction in the air permeability of the fabric will be dependent on the amount of starch granules that actually

block the air spaces. It is important to also note that starches manufactured by industries for application to textile fabrics shuld as much as possible not clog the interstices offabrics, therefore a decrease in air permeability of fabric is an indication of clogging in the fabric.

From Tables 3.18 and 3.21 it is noted that fabrics starched with the modified starches had relatively high air permeability values while those starched with unmodified starches had very low air permeability values. This is as a result of the small size of the granules which are not capable of properly clogging the fabric. Table 3.18 and 3.21 also shows that the air permeability values of fabrics starched with unmodified and modified forms of cassava starch fell within 256-253cc/sec while the air permeability vaules of fabrics starched with the unmodified and modified form of maize starch ranged from 170-254cc/sec. The implication of these is that, there is no apparent change in the air permeability values of fabrics starched with modified and unmodified forms of cassava starch while there exist a great variation in the case of fabrics starched with modified and unmodified forms of starch. Another important conclusion is that fabrics treated with cassava starch have minimal clogging while those treated with maize starch are often clogged to a large extent.

The low air permeability value obtained for fabrics treated with potato starch is primarily due to the large granule size of the starch. Air permeability value of rice and guinea corn

starches are relatively high because the size of the granules are quite small, though these granules are likely to be larger than granules of the modified starches.

3.4.14 Fabric Thickness Measurement

Fabric thickness is important in the study of other fabric characteristics, such as thermal insulation, resistance, dimensional stability, fabric stiffness, abrasion resistance etc.

The measurement of fabric thickness of starched fabrics enables a better understanding of the effect of different starches on the thickness of fabrics.

Tables 3.15 and 3.24 shows that the use of rice starch to stiffen cotton fabric resulted in cotton fabrics having the highest thickness. This is followed by potato, 3% maize, Guinea-corn, maize, cassava, 1.5% cassava, 1.5% maize, and 3% cassava.

The high value for fabrics treated with rice starch is probably due to the small size of the starch while the high value for fabrics treated with potato starch is probably due to the presence of some starch grains on the fabric surface which did not penetrate into the fabric. The values obtained for fabrics treated with guinea corn and corn starches are moderate due to their averagely small size starch granules.

In the case of the modified starches, the thickness of fabrics treated with maize starch decreased with increase in the concentration of the acid used. This is in contrast to fabrics

treated with cassava starch where the fabric thickness increased greatly for 1.5% cassava and decreased sharply for 3.0% cassava. The reason for this behaviour may be difficult to deduce but it is likely that some of the fine starch granules must have penetrated into the yarns of the fabric, thereby resulting in an increase in fabric thickness.

3.4.15 Subjective Assessment Of Fabrics

(i) Stiffness

The order of decreasing stiffness recorded during the subjective assessment is very different from that obtained using the bending length measurement. Variations, such as the position of 1.5% cassava starch are very prominent. Many reasons may be responsible for these variations. These may range from human error during subjective assessment to irregularities in the fabric structure, thereby resulting in some fabrics possessing a higher bending length than others. However, discrepancies between results obtained by objective means and subjective means are common. It is often safer to stick to that obtained by objective techniques since these are often reprducible.

(ii Odour

The odour perceived in the fabric was as a result of ironing. The heat given off by the iron degrades the excess starch on the fabric. The odour perceived in fabrics starched

with potato and rice starch was therefore due to the degradation of the large deposits of starch on the surface of the fabric. Likewise, the faint odour perceived in the case of fabrics treated with maize, guinea corn and cassava starches was due to small depsits of starch on the surface of the cloth.

(iii Change in Colour

The change in colour noticed for fabrics treated with guinea-corn was due to the light brown colouration of the guinea-corn starch paste while that of rice starch may be attributed to either the dirty white colouration of the rice starch powder or to degradation as a result of ironing. These discolourations are extremely faint and cannot be noticed for most fabrics.

CHAPTER FOUR

4.0 CONCLUSIONS

4.1 Starch Production Methods

Starch recovery is relatively high using the extraction methods employed in the project. The low recovery for rice starch can be improved upon by the use of a very good dry milling machine. These methods are relatively easy and required the use of very few complicated machines. However, it is recommended that for commercial purposes cassava starch should be made from fresh cassava tubers instead of cassava flour, because the starch produced in this instance is cheaper and better in quality.

4.2 Quality of starch produced

The starches produced in this study are of relatively high grade. The physical appearance of the starches produced meets the minimum standards required for usage in most industries with the exception of food and pharmaceutical industries.

The chemical properties of the starches produced are also within the permissible range for commercial use in most industries. It is important to note that most industries particularly the pharmaceutical and food industries often require starches that are of extremely high grade. The result of this is that the price of such starches are quite high since it is the

purpose of this project to produce starches that are relatively cheap and which will be used mainly by textile industries and textile consumers, much emphasis was not made on the production of pharmaceutical grade starches.

4.3 Rheological properties of the Starch pastes

The viscosity of the starch pastes was found to be of this order; Potato starch > Cassava starch > Maize starch > Rice starch > Guinea Corn starch.

Generally, the starches extracted from tubers were found to be more viscous than starches from cereals.

Starches that had larger granule sizes possessed higher paste viscosities than those with smaller granule sizes. However, the viscosity of starches with smaller granule sizes tended to be more stable to heat than the viscosity of starches with bigger granule sizes.

The viscosity of acid modified starches appears to be dependent on the conditions of modification. It is likely that the viscosity of acid-modified starches will cease to reduce after most of the branched amylopectin chain have been broken. This is likely to be the reason for the similarity in the viscosity of 1.5% maize, 1.5% cassava and 3.0% maize starch pastes. The viscosity of a modified starch paste decreases with temperature, this may be as a result of mechanical shear.

Though no experiment was conducted to find out the size of the granules of the modified starches, it is likely that modified starches that underwent the more severe modifying conditions will possess smaller granule size and hence lower viscosity. This may explain why 3.0% cassava and 3.0% maize starch pastes were relatively lower in viscosity than 1.5% cassava and 1.5% maize pastes.

4.4 Efficiency of Starches in Textile Applications

(i) Sizing of yarns

The starch pastes of cereal starches are more efficient than the starch pastes of tubers when used to size warp yarns. However it might be possible that cassava and potato starches could possess a higher efficiency if the sizing was performed using a much less concentrated starch paste. However, since this was not verifed during the course of this work no concrete assertions can be made.

Likewise, modified starches of cassava are very efficent when used to size warp yarns, infact cassava starches modified with 1.5% HCl for 4hrs at 45-50°C produced better results than the unmodified starch. In the case of maize starch, the unmodified starch performed better than the modified starches. It is important to note that while modified starches are reasonably efficient for the sizing of warp yarns; since they increase the

yarns strength to a reasonable level and they do not make the yarns brittle as the unmodified starches, the cost of production of modified starches is much higher and their production is time-consuming.

(ii) As a stiffening agent

The cereal starches (except Guinea corn) produce more stiffness in fabrics than the tuber corn starches. However it may be possible that the stiffness of fabrics may also depend on characteristics of the woven febric, since more open fabrics will have larger pores, thereby enabling large starch granules to penetrate into the fabric.

Modified starches of cassava produced fabrics with more stiffness than the unmodified starches while the reverse is true for maize starches.

From the results of this project, rice starch produces the best result when using starch as a stiffening agent. This is because both the subjective and objective assessment results showed that rice starch had a very high stiffening effect on fabrics while maize starch gave a high bending length but gave poor stiffening effect on the fabric when a subjective assessment was carried on the fabric.

From the results of this project, potato starch has the lowest stiffening effect on the fabric, while in the case of the modified starches, the modified corn starch are less efficient than the modified cassava starch.

It is important to note however, that the viscosity of the starch paste affects the effeciency of the starch when used as a stiffening agent. Hence, change in the concentration of starch paste may alter the order of efficiency of the starches when used as a stiffening agent.

4.5 Effect of Starch on the Air permeability and thickness of starch

The air permeability of the fabric, was greatly reduced when starches of maize, guinea corn, rice and potato were used. The modified starches reduced the air permeability of the fabric to a minimal extent. However it can be seen that the air permeability of the modified corn starches are relatively higher than those of cassava. The implication of this results is that for applications that require that the fabric remain reasonably open, modified starches are recommended.

The thickness of the fabric was increased by 0.014-0.170mm by the starched fabrics. The modified starches with the exception of 3% maize starch produced small increases in the fabric thickness while starches of potato, rice and guinea corn produced large increases. Maize and cassava starch gave moderate increases in the fabric thickness.

4.6 Recommendations

Modification of starches using oxidising agents produce starches with lower tendency to retrograde. This aspect is of

particular interest to the textile and allied industries. Further work in this area may produce interesting and useful results.

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Fig 3·1: Viscosity of unmodified starches at different temperatures

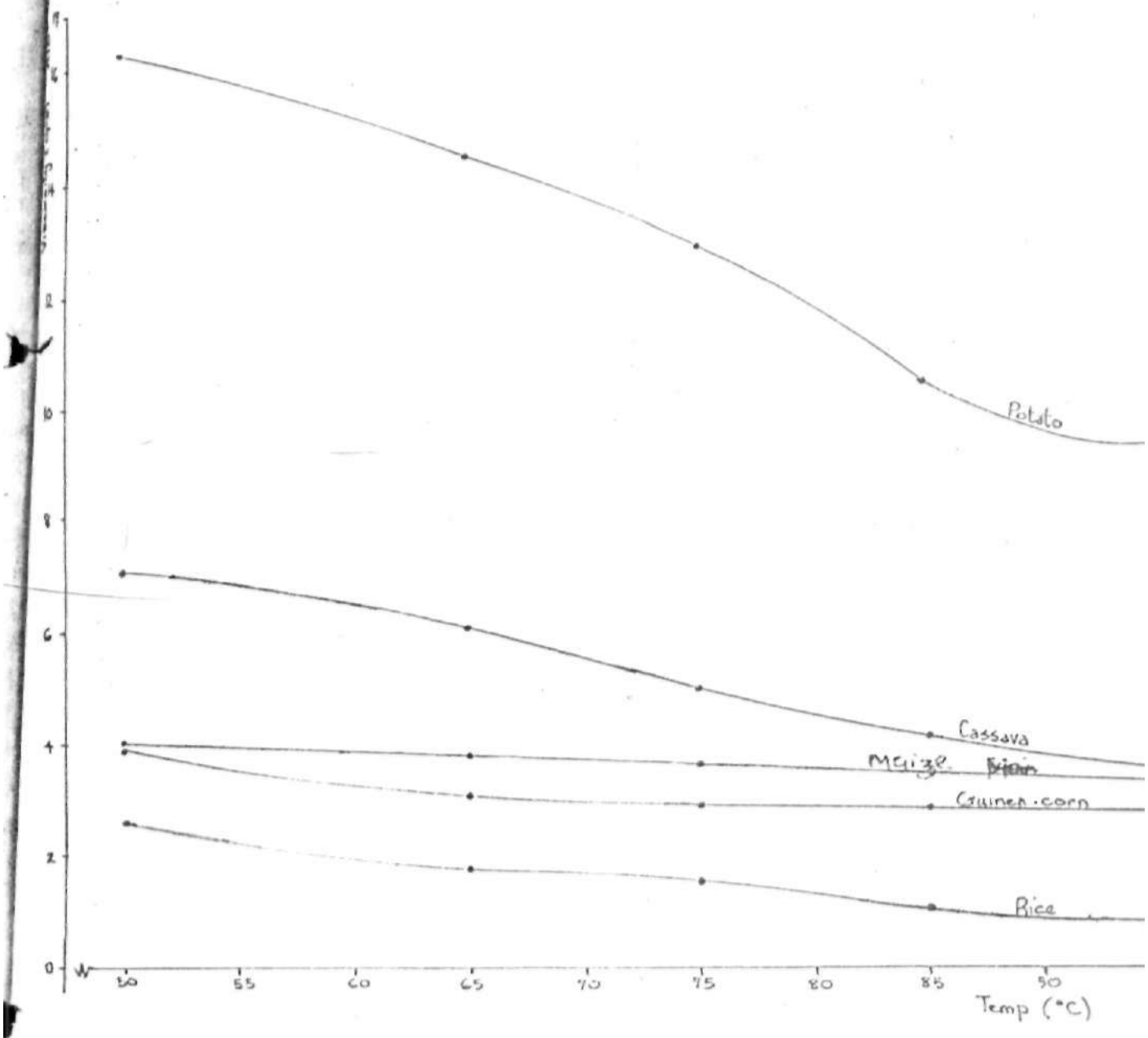


Fig. 3.2: Viscosity of modified starches at different temperatures

