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# Characterization Of Oxidized Plantain (*Musa paradisiaca)* Starch As Influenced By Reaction Temperature

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**ABSTRACT**
In Nigeria, unconventional sources of a natural bio-polymer such as a native starch have not been extensively studied. In order to proffer solutions to the major problem associated with plantain including banana which is high perishability that prevents the fruit from surviving extended period of drought, extraction of starch from the fruit followed by chemical modification of the starch was carried out.

Starch (a polysaccharide) isolated from unripe plantains (*Musa paradisiaca* L.) was modified chemically by oxidation with sodium hypochlorite (NaOCl) at three different reaction temperatures (40°C, 50°C and 60°C). The oxidized starches were characterized chemically, physically and thermally: quantification of carbonyl and carboxyl contents of the representative oxidized plantain starch ($OPS_{60}$) corroborated the effectiveness of the chemical treatments which was verified by Infrared spectroscopy (The band at 1730$cm^{-1}$ was attributed to the formation of carbonyl functional group). Proximal chemical analysis (Moisture content), Swelling power, Solubility, oil and water absorption capacities and Gelation studies were also investigated and results were within acceptable limit.

Modification of the native starch by oxidation increased the moisture content. The values increased with increased reaction temperature in a parallel manner. Studies conducted on swelling power and solubility of oxidized starches increased with increased temperature in a parallel manner. The swelling power of the oxidized starch samples decreased when compared to their native counterpart unlike solubility studies which increased indicating structural alterations of the oxidized starches. Hydrophilic and hydrophobic tendencies of starch improved after oxidation with increase in the reaction temperature(s). Among all starch samples, native plantain starch had better gelating property.

Keywords: Natural Polymer, Plantain starch, Oxidized plantain starch, Chemical Modification.

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## I. INTRODUCTION

High demand for starch at both industrial and domestic levels has placed this natural polymer significant and relevant to economic revitalization and prosperity in all countries across the globe. Starch has found wide applications in industrial settings of medicine, pharmaceuticals, petroleum, paper, food, confectionary, synthetic polymer industries as well as in technological developments due to its desirable functional properties.Sources of starch vary worldwide depending on local traditions and the condition of the climate. However, it is apparent that most of the starches used for industrial purpose are isolated from common

crops like cassava, potato and maize (Swinkels, 1985). This indeed has necessitated the need for research on other means of sourcing for starch.Many researches have been conducted in the extraction of starch from different unconventional sources such as Jack bean (Lawal and Adebowale, 2005), Lentil (Matina et al., 2017), Cocoyam (Ishiwu et al., 2017; Jacob and Adeleke, 2019), Bambara groundnut (Sirivongpaisal, 2008; Kaptso et al., 2014; Oyeyinka et al., 2015; Oyeyinka et al., 2016a; Oyeyinka et al., 2016d; Oyeyinka et al., 2018a;Samson et al., 2019).

Unconventional sources of starch that can be utilized are perishable staple foods such as Plantain, which is believed to have originated from the hot tropical of southern Asia into the humid tropics of western and central Africa (Sweinen, 1990).The plantain, which is a huge shrub's green to yellow boat-shaped fruit that is frequently fried before consumption, is a member of the genus Musa and family Musacae. With a working reproduction of 33 million metric tonnes, it is cultivated in 52 nations (FAO, 2005). It is the fourth most important food crop in the world after rice, wheat and maize with Uganda being the largest producer in Sub-Saharan Africa followed by Rwanda, Ghana, Nigeria and Cameroun. The plantation of the fruit in Nigeria often involves planting alongside cocoa where it serves as a nurse crop during its early stage of development. Bush plantation is another way by which the fruit is planted whereby it intercrops with many field crops such as cassava, cocoyam and yam.The fruit is widely used as food, beverages, fermentable sugars, medicines, flavouring cooked foods, fragrance and numerous ceremonial and religious uses. The fruit is extremely low in fat, high in dietary fibre, low in cholesterol and salt too. It is a good source of vitamin A (in the treatment of visual cycle), vitamin $B_6$ (in the treatment of anaemia), vitamin C (helps body develop resistance against infections), iron, magnesium (for bone strengthening), phosphorus and potassium. The water content in the green plant is about 61% and this increases on ripening to about 68%. The breakdown of carbohydrates during breathing is blamed for the rise in water. In the green fruit, the sugar concentration ranges from 0.9% to 2%, but it is more noticeable when the fruit is mature, when it reaches around 17%. (Marriot et al., 1981; Marriot and Lancaster, 1983; Ogazi, 1996). Unripe fruit has a protein level that ranges from 0.5% to 1.6%, with no discernible variation as the fruit ripens. Beta-alanine, amino-butyric acid, glutamine, aspagirine, serine, and leucine are some of the amino acid components. The fruit also contains a lot of ascorbic acid.

The fruit is a source of food security and income for small scale farmers who represent the majority of the producers and in Nigeria, it is processed and consumed as flour, snacks in form of chips and "dodo ikire" (Ukhum and Ukpebor, 1991). However, a major impediment of plantain is its high perishability which often renders the fruit not to survive extended period of drought unlike most other crops (Fagbohu et al., 2010). This is the reason why most of its production is consumed domestically and only about 15% of its global production is involved in international trade and during its bumper harvest, it is always in abundance and it is often sold at low price.In order to increase its economic value, addressing its high perishability and extending its storage life through extraction of starch (21-25%) from the fruit becomes imperative for both industrial and domestic uses (zaakpa et al., 2010).

Commercial utilization of starch isolated from both conventional and non-conventional sources in its pure state is however limited due to some undesirable properties exhibited by the native starch such as its insoluble nature, low mechanical properties and its unstable nature at high temperature, pH and shear during processing. To expand the application and flexibility of this native starch, some of its inimical properties need to be solvedeither through physical, enzymatic or chemical form of modifications (Richardson and Gorton, 2003). The essence of any form of starch modification is to modify its cooking characteristics, increase its freeze-thaw and process stability, decrease its retrogradation, gelling properties and improve its film forming properties (Kaur et al., 2012).The focus of this research work was channeled to the chemical form of starch modification. In recent times, numerous starch derivatives have been synthesized by acid hydrolysis, carboxymethylation, hydroxypropylation, esterification, oxidation, cross-linking and many more(Adebowale et al., 2002; Adebowale and Lawal, 2002; Santacruz et al., 2002; Afolabi, 2012; Oyeyinka et al., 2016b; Oyeyinka et al., 2016c; Oyeyinka et al., 2017b; Oyeyinka et al., 2018a).Starch modification via oxidation involves the introduction of carboxyl and carbonyl functional groups into the structure of the native starch, with subsequent depolymerization of the native starch through the interaction of an oxidizing agent introduced with the free hydroxyl groups in the glucose monomer of the native starch. Such starches have been established to be whiter in color and have restricted retrogradation (Kuakpetoon and Wang, 2001).

This research work was aimed at providing insights on the effect of varying the reaction temperature on samples of synthesized oxidized plantain starch granule in terms of determining its unique characteristics that are exploited for different industrial purposes when compared to other sources of starch and this may become useful information to other scientific studies in future. However, the objectives of the study include isolation of starch from plantain, preparation of oxidized samples of plantain starch at three different reaction temperatures using sodium-hypochlorite as the modification reagent and investigation on the effects of oxidationon the physicochemical properties of all samples of oxidized plantain starch prepared at the different working temperatures.

## II. EXPERIMENTAL

### 2.1 MATERIALS AND CHEMICALS

Unripe plantain fruits whose cultivar is known as "Agbagba" were obtained from Ilaro, Ogun State, Nigeria. Household bleach containing 3.85% active sodium hypochlorite (w/v) was obtained from Ago Iwoye, Ogun State, Nigeria. All other chemicals used in thisresearch include Hydrochloric acid (BDH), Sodium hydroxide (Merck), Sodium chloride (Purex chemicals), Silver nitrate (BDH), Hydroxylamine (BDH) and Phenolphthalein (BDH), distilled waterwhich were of analytical grade and used as received.

### 2.2 EXTRACTION OF STARCH FROM UNRIPE PLANTAIN FRUITS.

The method employed for starch isolation is outlined in Fig. 1. Peeled unripe plantain fruits were diced into pieces by the use of a conventional knife, and these were collected into a calibrated bucket filled with water to prevent browning of the fruit. Thereafter, the sliced plantain was wet milled in order to reduce the particle size to a mechanically possible minimum level that will facilitate the recovery of starch. The slurry was dispersed in distilled water before sieving through screen cloth. The retentate on the first screen was re-slurried in water and screened again. Impurities were removed by washing the permeate counter-currently with distilled water until the overflow had the same clarity as the inlet water. The supernatant was then decanted off after sedimentation and the required starch was obtained and air dried at 30 ± 2°C for 48 h.

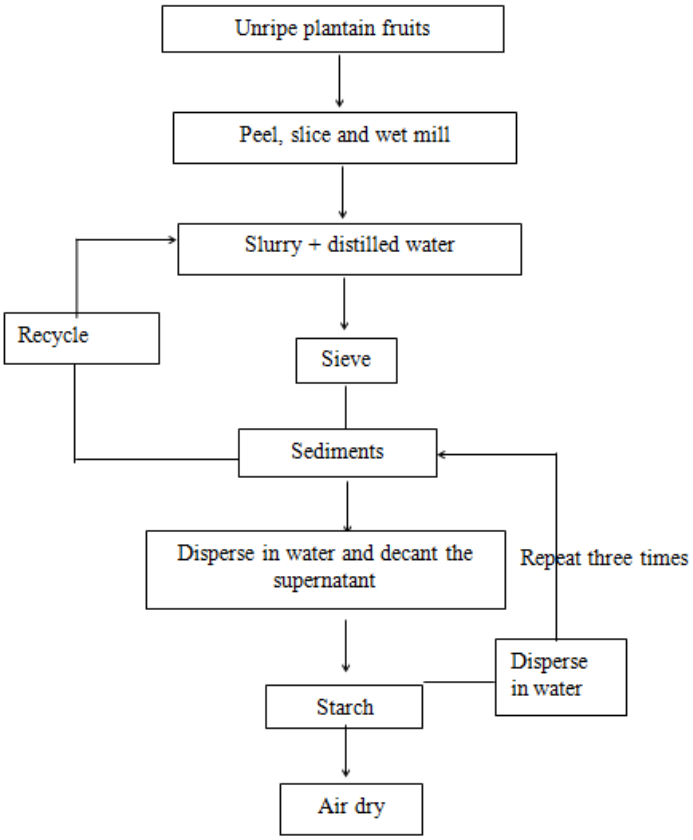



Fig 1: Flow diagram for extraction of plantain starch.

### 2.3 PREPARATION OF OXIDISED STARCH

Threei samplesi ofi oxidizedi plantaini starchi werei preparedi usingi thei methodi describedi byi Adebowalei *eti al*.,i (2005).i Nativei plantaini starchi (20g)i wasi mixedi withi 100i mLi ofi distilledi wateri andi thei pHi ofi thei mixturei wasi broughti toi 9.5i withi 0.3Mi sodiumi hydroxidei (NaOH).i Ai pHi ofi 9.0-9.5i wasi maintainedi whilei twoi gramsi ofi sodiumi hypochloritei (NaOCl)i werei addedi dropwisei overi thei coursei ofi thirtyi minutesi withi continuousi stirring.i Crushedi icei andi sodiumi chloridei werei usedi toi chilli thei reactioni vesseli (ai 400i mLi Beaker)i (NaCl).i Afteri alli thei NaOCli hadi beeni added,i thei slurryi wasi placedi ini ai 250i mLi Erlenmeyeri flaski andi thei reactioni wasi allowedi toi runi fori 1i houri ini ai thermo-regulatedi wateri bathi ati variedi temperaturesi ofi 40°C,i 50°C,i andi 60°C,i respectively.i Thei 0.3Mi hydrochlorici acidi (HCl)i wasi usedi toi adjusti thei pHi toi 7.0,i andi Whatmani filteri paperi No.i 4i wasi usedi toi filteri thei slurry.i Thei obtainedi starchi wasi cleanedi fouri timesi withi distilledi wateri andi driedi fori 48i

hoursi ati 30i 2°C..i Thei oxidizedi starchi samplesi werei designatedi asi $OPS_{40}$,i $OPS_{50}$i andi $OPS_{60}$i accordingi toi thei workingi temperaturesi duringi thei modificationi processi withini thei rangei 40i –i 60°Ci respectively.

### 2.4 DETERMINATIONi OFi CARBONYLi CONTENT

Thei modifiedi Lawali techniquei wasi usedi toi determinei thei carbonyli contenti (2004a).i Ai 2gi samplei ofi oxidizedi starchi wasi slurriedi ini 100i mLi ofi distilledi water.i Thei mixturei wasi cooledi toi 40°C,i gelatinizedi ini ai boilingi wateri bathi fori 20i minutes,i andi theni hadi itsi pHi raisedi toi 3.2i usingi 0.1Mi HCl.i Thei hydroxylaminei reagenti wasi theni addedi ini ai volumei ofi 15i mL.i Aluminumi foili wasi usedi toi protecti thei samplei ini thei flaski beforei iti wasi submergedi ini ai wateri bathi heatedi toi 40°C.i Ini orderi toi identifyi thei excessi hydroxylamine,i thei samplei wasi quicklyi titratedi withi 0.1Mi HCli toi pHi 3.2i afteri 4i hours.i Ini thei samei way,i ai blanki determinationi usingi simplyi hydroxylaminei reagenti wasi carriedi out.i Toi makei thei hydroxylaminei reagent,i 25gi ofi hydroxylaminei hydrochloridei werei dissolvedi ini 100i mLi ofi ai 0.5Mi NaOHi solution.i Thei finali volumei wasi theni adjustedi toi 500i mLi withi distilledi water.i Thei %i carbonyli contenti wasi determinedi asi follows:

$$\%\ \text{Carbonyl} = \frac{(\text{Blank titre} - \text{sample titre})\ \text{mL} \times \text{Acid molarity} \times 0.028 \times 100}{\text{Dry sample weight (g)}}$$

### 2.5 DETERMINATIONi OFi CARBOXYLi CONTENT

Thei carboxylcontenti wasi ascertainedi usingi thei modifiedi Lawali approachi (2004a).i Slurriedi ini 100i mLi ofi distilledi wateri wasi ai 2gi samplei ofi oxidizedi starch.i Afteri beingi cooledi toi 40°C,i thei mixturei wasi gelatinizedi ini ai boilingi wateri bathi fori 20i minutes,i andi itsi pHi wasi theni increasedi toi 3.2i usingi 0.1Mi HCl.i Then,i 15i mLi ofi thei hydroxylaminei reagenti wasi added.i Beforei beingi immersedi ini ai wateri bathi thati wasi heatedi toi 40°C,i aluminumi foili wasi utilizedi toi shieldi thei samplei withini thei flask.i Thei samplei wasi promptlyi titratedi withi 0.1Mi HCli toi pHi 3.2i afteri 4i hoursi toi detecti thei extrai hydroxylamine.i Thei samei procedurei wasi followedi toi performi ai blanki determinationi utilizingi onlyi thei hydroxylaminei reagent.i 25gi ofi hydroxylaminei hydrochloridei werei dissolvedi ini 100i mLi ofi ai 0.5Mi NaOHi solutioni toi createi thei hydroxylaminei reagent.i Thei originali samplei wasi subjectedi toi thei identicali procedurei fori ai blanki determination,i excepti thati distilledi water,i noti 0.1Mi HCl,i wasi usedi toi agitatei thei sample.i Followingi isi howi thei percentagei carboxyli contenti wasi determined:

$$\%\ \text{carboxyl} = \frac{(\text{Sample titre} - \text{blank titre})\ \text{mL} \times \text{Alkali molarity} \times 0.045 \times 100}{\text{Sample weight (g)}}$$

### 2.6 PHYSICOCHEMICALi PROPERTIES

#### 2.6.1 GELATIONi STUDIES

Gelationi investigationsi werei conductedi utilizingi thei Lawal-describedi techniquei (2004a).i 5i mLi ofi distilledi wateri wasi usedi toi createi samplesi ofi starchi rangingi fromi 2-14%i (w/v)i ini testi tubes.i Thei starchi suspensionsi werei blendedi fori fivei minutesi ini ai mixer.i Thei testi tubesi werei heatedi fori 30i minutesi ati 80°Ci ini ai controlledi wateri bath,i theni quicklyi cooledi downi underi coldi runningi water.i Fori ani additionali 2i hours,i thei testi tubesi werei chilledi ati 4°C.i Thei samplei fromi thei invertedi testi tubei didi noti dropi ori slip,i hencei thati concentrationi wasi identifiedi asi thei leasti gelationi concentration.

#### 2.6.2 OILi ANDi WATERi ABSSORPTIONi CAPACITY

Thei methodi describedi byi Lawal*i eti al*.i (2005)i wasi usedi toi determinei oili andi wateri absorptioni capacityi ofi thei starch.i 10mli ofi distilledi wateri ori oili (Goyai oili ofi densityi 0.9g/$cm^3$)i wasi addedi toi 1i gi ofi sample.i Thei mixturei wasi mixedi withi ai stiri rodi fori 30i si andi allowedi toi standi fori 30min.i Then,i thei volumei ofi thei supernatanti wasi recorded.i Thei massi ofi oili ori wateri absorbedi wasi expressedi asi g/gi starchi oni ai dryi weighti basis.

#### 2.6.3 FREEi SWELLINGi CAPACITYi STUDIES

Effecti ofi temperaturei oni freei swellingi capacityi wasi carriedi outi ini thei temperaturei rangei ofi 50i –i 80°C,i usingi thei teabagi methodi asi describedi byi Lawali (2011).i Starchi (1i g)i wasi weighedi andi packedi insidei teabagi (TeaGschwendner,i meckenheim).i Iti wasi placedi oni ai testi tubei racki andi theni connectedi toi ai thermostati ini whichi thei wateri wasi circulatedi ati specifici temperature.i Thei samplei wasi takeni outi ofi thei wateri afteri onei houri andi allowedi toi draini fori fivei minutesi toi ensurei homogeneity.i Thei teabag'si swelledi starchi wasi weighed.i Thei weighti ofi thei emptyi teabagi andi thei volumei ofi wateri iti absorbedi werei specified.

Thei freei swellingi capacityi (FSC)i wasi determinedi ini g/gi asi follows:

$$FSC = \frac{Wss}{Wcs}$$

$$Wss = W_4 - W_2 - W_1$$

$$Wcs = W_3 - \left(\frac{W_3 \times M_t}{100}\right)$$

$Wcs$ = Corrected weight of starch

$Wss$ = Weight of swollen starch

$Mt$ = g / 100mL moisture content of the starch

$W_1$ = Weight of dry tea bag

$W_2$ = Weight of water absorbed by the empty bag

$W_3$ = Weight of starch taken

$W_4$ = Total weight after swelling.

### 2.6.4 SOLUBILITYi STUDIES

Solubilityi determinationsi werei carriedi outi usingi thei methodi ofi Leach,i McCoweni andi Scochi (1959).i 0.1gi ofi samplei wasi accuratelyi weighedi andi quantitativelyi transferredi intoi ai cleari driedi testi tubei andi weighed.i 10$cm^{3i}$ ofi distilledi wateri wasi addedi toi thei testi tubei andi thei mixturei wasi mixedi thoroughlyi fori 30s.i Thei resultanti slurriesi werei heatedi ini thei temperaturei rangei 50-80°Ci fori 30i mini ini ai temperaturei regulatedi wateri bath.i Thei mixturei wasi cooledi toi roomi temperaturei andi centrifugedi (5000i xi g)i fori 15i min.i Aliquotsi (5i mL)i ofi thei supernatanti obtainedi afteri centrifugationi werei driedi toi ai constanti weighti ati 110°C.i Thei residuei obtainedi afteri dryingi thei supernatanti representsi thei amounti ofi starchi solubilizedi ini water.i Solubilityi wasi calculatedi ini percentagei (%)i

### 2.6.5 MOISTURE CONTENT OF STARCH

The moisture content was determined using the method described by Ashveen, Randhir, Rohindra and Khurma, (2008).

Petri dishes with lids were washed and dried in an oven at 105°C for 2h, cooled to ambient temperature in a desiccator. Approximately 5g of starch samples were weighed accurately in petri dishes. The samples were dried for 2 h at 105°C, cooled in a desiccator and weighed. Moisture content (MC) was calculated as shown below:

$$\% \text{ moisture content} = \frac{\text{Loss in weight}}{\text{Weight of sample}} \times 100\%$$

### 2.6.6 FOURIER TRANSFORM INFRARED SPECTRA

The FT-IR spectroscopy technique was used to analyse chemical structural changes in both native and oxidized plantain starch prepared at 60°C working temperature. The spectra were run as KBr pellets on FTIR spectrometer in the frequency range 4000-500$cm^{-1}$.

## III. RESULTS AND DISCUSSION

### 3.1 CARBONYL AND CARBOXYL CONTENT

Thei resultsi obtainedi fori carbonyli andi carboxyli contenti ofi alli oxidizedi plantaini starchi samplesi arei presentedi ini Tablei 1.i i Plantaini starchi oxidizedi ati 40°C,i 50°Ci andi 60°Ci reactioni temperaturesi showedi higheri valuesi ofi carboxyli groupi thani carbonyli functionali groups.i Thei formeri (carboxyli content)i increasedi ini ai paralleli manneri withi increasedi reactiontemperatureandthismaybeduetolimitationofthecontactbetweenthestarchmoleculesandtheoxidizingagent.Thelatter(carbonylcontent)ontheotherhandalsofollowedsimilartrendbyincreasingwithincreasedreactiontemperature.Theresultsobtainedareinagreementwiththeresultsobtainedforcassavastarch(Sangseethong*etal*.,2010),Jackbeansstarch(LawalandAdebowale,2005)andhybridmaizestarch(Lawal*etal*.,2005).Itwashoweverreportedthathypochloriteoxidationofstarchwhenbeingperformedunderalkalineconditionsfavourstheformationofcarboxylgroups(Wurzburg, 1986;WangandWang,2003;Lawal*etal*.,2005andSangseethong*etal*.,2010).Thepatternofcarbonylandcarboxylgroup

formationasi ai functioni ofi reactioni temperaturei supportsi thati reactioni pathi ofi hypochloritei oxidationi isi consecutivei withi carbonyli groupi asi intermediates,i whichi arei rapidlyi oxidizedi furtheri toi carboxyli groupsi asi thei primaryi finali product.i Thei resultsi suggesti thati furtheri oxidationi ofi thei carbonyli toi carboxyli groupsi wasi veryi fast.i

Table1:i Carboxyli andi carbonyli contentsi ofi oxidizedi plantaini starchesi preparedi ati differenti Temperatures.

| Samples | Carboxyli contenti (%) | Carbonyli contenti (%) |
|---|---|---|
| $OPS_{40}$ | 0.22 | 0.41 |
| $OPS_{50}$ | 0.24 | 0.43 |
| $OPS_{60}$ | 0.25 | 0.44 |

### 3.2 GELATIONi STUDIES

Gelationi propertiesi ofi thei nativei andi oxidizedi starchesi preparedi underi variousi reactioni temperaturesi arei presentedi ini Tablei 2.

Thei leasti gelationi concentrationi (LGC)i wasi usedi asi thei indexi ofi gelation.i Ati ai constanti reactioni time,i LGCi increasedi followingi oxidationi asi thei reactioni temperaturei increased.i Oxidizedi starchi preparedi ati 50°Ci andi 60°Ci respectivelyi hadi LGCi valuei ofi eighti (8).i Afteri thisi concentration,i $OPS_{40}$,i $OPS_{50}$i andi $OPS_{60i}$ appearedi asi geli andi firmi geli ati 12%i andi 14%i (w/v)i respectivelyi whilei NPSi appearedi asi ai firmi geli andi veryi firmi geli ati thei samei respectivei concentrations.i Thesei observationsi indicatei thati nativei starchi couldi havei betteri gelatingi propertiesi thani thei oxidizedi starches.i Iti shouldi howeveri bei notedi thati amongi thei starches,i nativei starchi hadi thei leasti LGCi andi thisi isi ini accordancei withi thei resultsi obtainedi fori Jacki beani starchi (LawalandAdebowale,2005),i breadfruiti starchi (Adebowale*et al*.,2005)i andi hybridi maizei starchi (Lawal*etal*.,2005).i i Starchi gelationi isi ai complexi processi thati involvesi gelatinization,i swellingi andi absorptioni ofi wateri toi buildi ai three-dimensionali networki thati offersi structurali rigidityi ini variousi foodi applications.i Thei buildingi ofi thei structurali networki involvesi asi well,i thei bridgingi ofi thei inter-granulari bindingi forcesi amongi thei starchi molecules,i whichi largelyi involvesi hydrogeni bonding.i Introductioni ofi carbonyli andi carboxyli groupsi followingi oxidationi probablyi limitedi thisi interactioni andi causedi electrostatici repulsioni amongi thei starchi molecules,i thus,i increasingi LGC.

Table 2: Gel strength of plantain starch samples (native and oxidized).

| Sample concentration (%w/v) | | NPS | $OPS_{40}$ | $OPS_{50}$ | $OPS_{60}$ |
|---|---|---|---|---|---|
| 2% | observation | liquid | liquid | liquid | liquid |
| | Inference | - | - | - | - |
| 4% | observation | viscous | liquid | viscous | viscous |
| | Inference | - | - | - | - |
| 6% | observation | Gel | viscous | viscous | viscous |
| | Inference | + | - | - | - |
| 8% | observation | Gel | viscous | Gel | Gel |
| | Inference | + | - | + | + |
| 10% | observation | Gel | Gel | Gel | Gel |
| | Inference | + | + | + | + |
| 12% | Observation | Firm gel | Gel | Gel | Gel |
| | Inference | + | + | + | + |
| 14% | observation | very firm gel | firm gel | firm gel | firm gel |
| | Inference | + | + | + | + |
| LGC | | 6% | 10% | 8% | 8% |

NPSi i i i =i Nativei plantaini starch;i $OPS_{40}$i =i Oxidizedi plantaini starchi preparedi ati 40$^{o}$C;i $OPS_{50i}$ i =i Oxidizedi plantaini starchi preparedi ati 50$^{o}$C;i $OPS_{60}^{i}$ =i Oxidizedi plantaini starchi preparedi ati 60$^{o}$C;LGCi =i Leasti Gelationi Concentration.

### 3.3 OILi ANDi WATERi ABSORPTIONi CAPACITY

Wateri andi oili absorptioni capacitiesi ofi nativei andi oxidizedi starchi samplesi arei presentedi ini Table3.i Thei resultsi showedi thati oxidationi improvedi ori increasedi thei tendencyi ofi thei starchi toi absorbi water.i Hydrophobici tendencyi ofi thei starchesi oni thei otheri handi improvedi afteri oxidation.i i Improvementi ini oili absorptioni ofi thei starchi samplesi isi attributedi toi thei introductioni ofi carboxyli andi carbonyli groupsi whichi ini turni causedi electrostatici repulsioni amongi starchi molecules,i therebyi facilitatingi thei absorbancei ofi oili byi thei starchi matrices.i Thisi resulti agreesi withi thei resultsi (92.3%i andi 94.6%i respectively)i reportedi oni oili capacitiesi ofi nativei andi oxidizedi cocoyami starchesi (Lawal,2004a).i $OPS_{40}$,i $OPS_{50}$i andi $OPS_{60i}$ havei higheri valuesi ofi oili absorptioni thani unmodifiedi plantaini starch.i Followingi ai similari trend,i alli preparedi samplesi ofi OPSi havei higheri valuesi ofi wateri absorptioni capacityi thani unmodifiedi plantaini starch.i Iti wasi howeveri observedi thati hydrophobici tendencyi wasi greateri thani hydrophilici propertiesi ini alli starchi samplesi andi thisi isi ini accordancei withi thei studiesi reportedi oni oili andi wateri absorptioni capacitiesi ofi nativei andi oxidizedi Jacki beani starches(LawalandAdebowale,2005).

Table3:i Valuesi ofi Oili andi wateri absorptioni ofi plantaini starchi samplesi (nativei andi oxidized)

| starchi samples | wateri absorbedi (g/g) | oili absorbedi (g/g) |
|---|---|---|
| $OPSi_{40}$ | 1.74 | 2.50 |
| $OPS_{50}$ | 1.76 | 2.52 |
| $OPS_{60}$ | 1.80 | 2.58 |
| NPS | 1.62 | 1.40 |

### 3.4 SWELLINGi POWERi STUDIES

Swellingi poweri ini bothi nativei plantaini starchi andi oxidizedi starchesi preparedi ati variousi reactioni temperaturesi werei temperaturei dependent.i Thei resultsi arei presentedi ini Table4.i Additionally,i accordingi toi Zakpaai eti al.,(2010),i findingsi fromi thei differenti analysesi ofi plantaini starchi showedi bigi granulei sizei andi lowi amylosei concentration.i Becausei thei starchi granulesi ini plantainsi arei soi big,i theiri tendencyi toi expandi isi increased.i Greateri swellingi resultsi fromi thei weakeri hydrogeni bondi connectioni thati largeri granulesi havei duei toi theiri loweri surfacei areai toi volumei ratio.i Ini orderi toi preventi poori producti recoveryi andi toi restricti thei interactioni betweeni thei starchi moleculesi andi thei oxidizingi agent,i iti isi therefore i instructivei toi operatei ati temperaturesi loweri thani thei starch'si gelatinizationi temperature.i Swellingi poweri increasedi withi temperaturei withini thei studiedi temperaturei rangei (50-80$^{o}$C).i Thisi isi ini linei withi studiesi thati werei publishedi oni otheri starchesi thati underwenti thei samei alterationi procedurei(Lawal,i 2005;i Adebowalei eti al.,i 2002).i Thisi increasei isi madei easieri byi thei starchi granules'i abilityi toi absorbi water,i especiallyi ini theiri amorphousi areas.i Samplesi ofi oxidizedi plantaini starchi hadi loweri swellingi capabilitiesi thani nativei plantaini starchi ati alli temperatures.i Thisi mayi bei explainedi byi structurali breakdowni occurringi withini thei starchi granulesi duringi thei alterationi process.

Hypochloritei oxidation,i accordingi toi Adebowalei eti al.,i (2002)i andi Ogungbenlei (2009),i isi ai veryi efficienti wayi toi depolymerizei andi degradei thei internali structurei ofi starchi granules,i whichi enhancesi solubilityi andi reducesi swellingi power.i Mosti starches'i swellingi patternsi havei ai bigi impacti oni howi they'rei used.i Processingi parametersi includingi temperature,i duration,i andi stirringi affecti swellingi poweri (Wooteni andi Bamnuaruchi,i 1978).i Therefore,i thei lowi valuesi seeni ini plantaini starchesi thati werei bothi nativei andi oxidizedi mayi bei thei resulti ofi thei creationi ofi ani amylose-lipidi complexi thati constrainedi granulei expansion.i Thus,i wheni thei temperaturei rose,i therei wasi ai correspondingi risei ini starchi granulei swelling.

Tablei 4:Valuesi ofi swellingi poweri ofi plantaini starchi (nativei andi oxidized)i ati variousi temperatures.

| Swellingi poweri i i i $OPS_{40i}$ (g/g)$_i$ i i $OPS_{50i}$ (g/g)$_i$ i i i i i i i i $OPS_{60i}$ (g/g)i i i i NPS(g/g) | | | | |
|---|---|---|---|---|
| temperaturei rangei | | | | |
| 50$^{o}$C | i i i i 4.50 | i i i i i i i | 4.56 | i 4.60 | i i i i i i 7.60 |
| 60$^{o}$C | i i i i 6.82 | i i i i i i i | 6.84 | i 6.88 | i i i i i i 8.40 |
| 70$^{o}$C | i i i i 7.34 | i i i i i i i i 7.40 | i 7.42 | i i i i 10.20 | |
| 80$^{o}$C | i i i i 9.78 | i i i i i i i i 9.84 | i 9.84 | i i i i 11.i 20 | |

### 3.5 SOLUBILITYi STUDIES

Thei temperaturei hasi ani impacti oni thei solubilitiesi ofi bothi nativei andi oxidizedi starches.i Tablei 5i andi Fig.i 2i exhibiti thei findings.i Thei solubilityi ofi thei oxidizedi starchesi alsoi increasedi asi thei reactioni temperaturei rose.i Increasedi wateri absorptioni uponi oxidationi acceleratedi amylosei leachingi

fromi thei starches'i amorphousi areas.i Additionally,i wheni thei temperaturei rose,i thei granulari structurei ofi thei starchesi wasi degraded,i thei inter-i andi intramoleculari hydrogeni bondsi ini thei starchi chainsi werei broken,i andi thei motionali freedomi ofi thei starchi chainsi increased.i However,i iti shouldi bei emphasizedi thati alli ofi thei plantaini starchi samples'i solubilitiesi hadi lowi values.i Thisi wasi explainedi byi thei developmenti ofi ani amylose-lipidi combinationi thati limitedi solubility.i Thisi isi consistenti withi thei outcomesi thati Zakpaai *eti al*.i (2010)found.i Sincei thei granulesi swelledi asi thei temperaturei rose,i therei isi ai comparablei risei ini starchi solubilityi asi ai result.i Thei maximumi solubilityi wasi demonstratedi byi oxidizedi starchi producedi ati 50°Ci andi 60°C,i respectively.

Table 5: Values of solubility of plantain starch at various temperatures (native and oxidized)

| Solubility temperature range | $OPS_{40}$ (%) | $OPS_{50}$ (%) | $OPS_{60}$ (%) | NPS (%) |
|---|---|---|---|---|
| 50°C | 2.32 | 2.36 | 2.40 | 1.18 |
| 60°C | 3.02 | 3.08 | 3.12 | 2.20 |
| 70°C | 4.10 | 4.16 | 4.18 | 3.38 |
| 80°C | 6.70 | 6.72 | 6.72 | 5.28 |

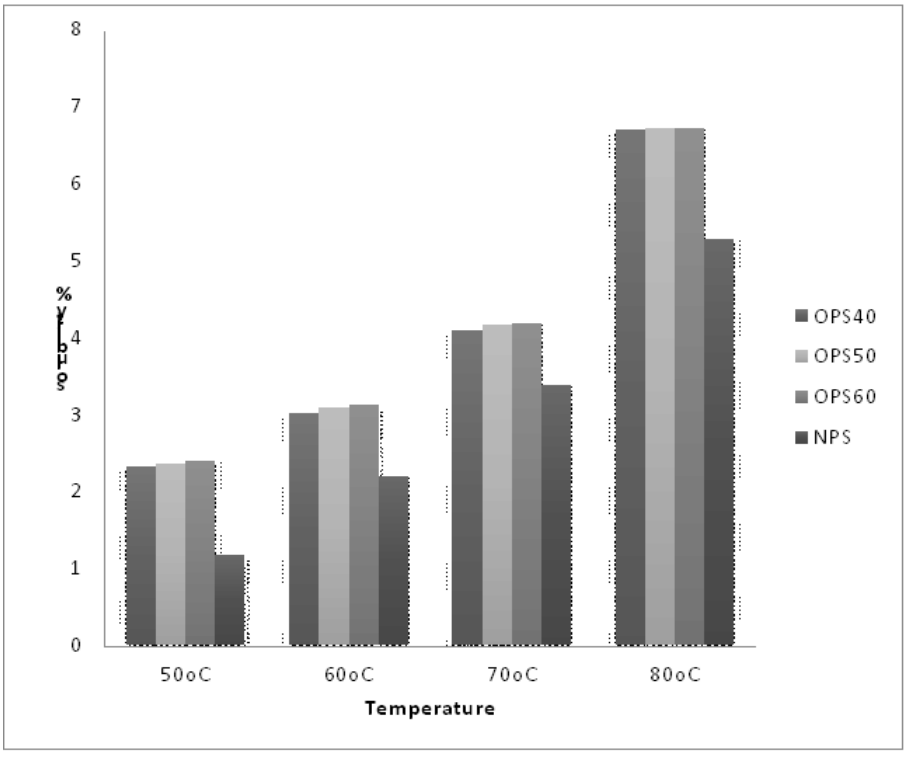



Fig.i 2: Ai charti ofi thei effecti ofi temperaturei oni solubilityi ofi plantaini starchi samplesi (nativei andi oxidized).

### 3.6 MOISTUREi CONTENT

Tablei 6i displaysi thei resultsi ofi thei moisturei content.i Thei starches'i moisturei contenti variedi fromi 12.80%i toi 20.50%.i Alli oxidizedi starchi samplesi hadi morei moisturei ini themi thani theiri nativei counterpartaftermodification.i Thisi couldi bei becausei ofi thei hydrophilicityi broughti oni byi oxidation.i Thei oxidizedi starchesi madei ati 60°Ci preservedi thei highesti moisturei content,i whereasi thei oxidizedi samplei preparedi ati 40°Ci hadi thei lowesti moisturei level.i Thesei findingsi arei ini linei withi resultsi oni mucunai beani starchi andi bambarai groundnuti starchi byi thei samei authorsi (Adebowalei eti al.,i 2002 and Adebowalei andi Lawal,i 2003).

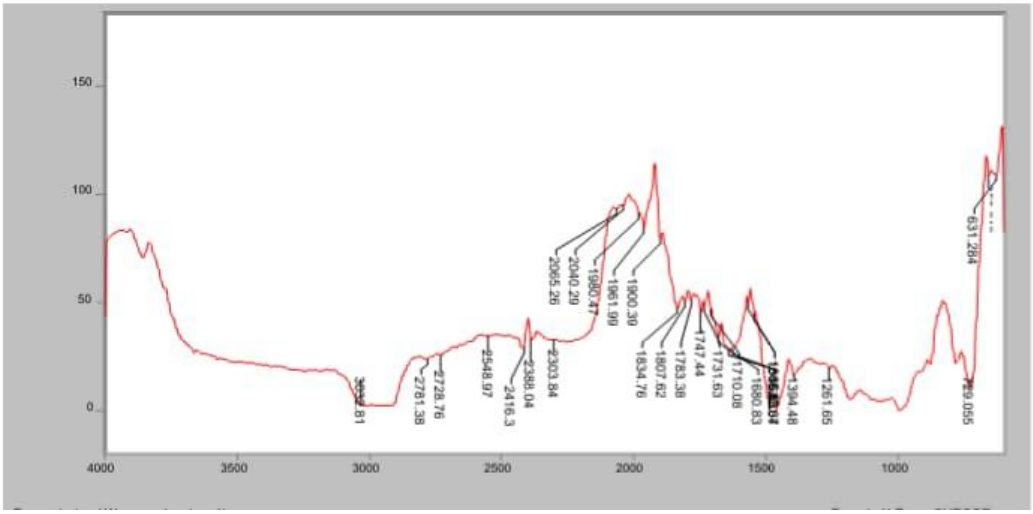
Table 6: Moisture content values of both native and oxidized samples

| Starch samples | moisture content (%) |
|---|---|
| Native Plantain | 12.80 |
| $OPS_{40}$ | 20.40 |
| $OPS_{50}$ | 20.00 |
| $OPS_{60}$ | 20.50 |

$OPS_{40}$ = Oxidized plantain starch prepared at 40°C; $OPS_{50}$ = Oxidized plantain starch prepared at 50°C; $OPS_{60}$ = Oxidized plantain starch prepared at 60°C.

### 3.7 FT-IRi ANALYSIS

Thei Infraredi Spectrai ofi nativei plantaini starchi andi ai representativei oxidizedi starchi ($OPS_{60}$,i carbonyli andi carboxyli contentsi respectivelyi =i 0.25i andi 0.44)i arei presentedi ini fig.i 3i andi fig.i 4i respectively.i Thei bandi aroundi 2782cm$^{-1i}$ wasi attributedi toi $CH_{2}$i symmetricali stretchingi vibrationi ini bothi starches.i Thei broadi bandi fromi 3000i toi 3500cm$^{-1i}$ wasi duei toi thei hydrogeni bondi ofi thei hydroxyli groupsi thati contributedi toi thei stretchingi vibrationsi associatedi withi thei freei inter-andi intra-i moleculari bondi ofi thei hydroxyli group,i ai characteristici thati isi particulari toi starchi structure.i Ini thei nativei plantaini starch,i thei bandi aroundi 762i andi 631cm$^{-1i}$ werei duei toi skeletali stretchingi vibrations.i Thei bandi aroundi 1731cm$^{-1i}$ wasi assignedi toi thei absorptioni ofi wateri moleculesi byi thei nativei starch.i Ini thei representativei oxidizedi starch,i thei peaksi locatedi ati 1961i andi aroundi 1730cm$^{-1i}$ werei assignedi toi thei flexioni ofi thei $CH_{2i}$ groupi andi formationi ofi thei carbonyli functionali groupi respectivelyi withi thei latteri confirmingi thati oxidationi reallyi tooki placei ini thei modifiedi starchi molecules.




Wavenumber [cm$^{-1}$]

Fig. 3: FTIR spectrum of native plantain starch.

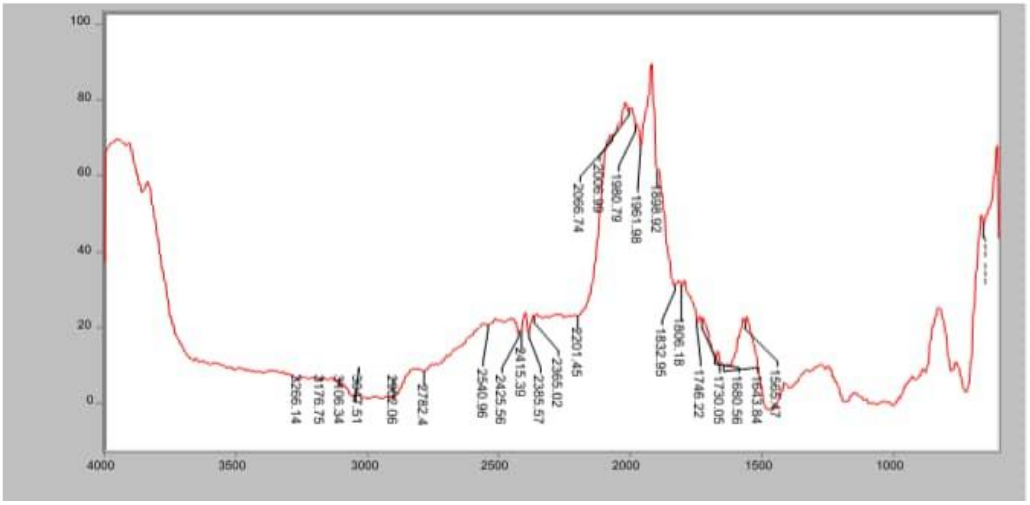



Fig. 4: FTIR spectrum of oxidized plantain starch prepared at 60°C [$OPS_{60}$

Wavenumber [$cm^{-1}$]

## IV. CONCLUSION

Oxidized starch samples were prepared from unripe plantain fruits at different reaction temperatures with sodium hypochlorite as the oxidizing agent and their physicochemical properties studied. The results obtained from this research which are in agreement with the results obtained from other sources of starch by previous researchers have indeed proven that, plantain starch has potentials of global acceptance with good functional parameters that could be exploited for commercial applications especially as a potential additive or compression excipient in pharmaceutical industries thereby positioning the starch for good market globally. The oxidized samples of plantain starch particularly the $OPS_{60}$could also be a potential replacement of cereal and root crop starches in foods and drinks where neutral tasting and low viscosity are required such as in beverage industries since oxidation contributes to the viscosity and gel strength of native starch.

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