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PHYSICOCHEMICAL AND RETROGRADATION CHARACTERISTICS OF MODIFIED SWEET POTATO (*Ipomoea batatas L.* (Lam)) STARCH

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ABSTRACT

Sweet potato starch (*Ipomoea batatas L.* (Lam)) was isolated and subjected to chemical modifications to generate oxidized (OSPS), acetylated (ASPS) and acid-thinned (ATSPS) sweet potato starches. The native sweet potato starch (NSPS) and various modified starches were characterized in terms of proximate, physicochemical, pasting properties, gelation and paste clarity. Results obtained showed that moisture, ash and protein were significantly ($P \leq 0.05$) reduced following modification. Also, there were significant variation ($P \leq 0.05$) in all the physicochemical parameters evaluated. Solubility of the native starch was greatly improved after modification with OSPS (25.25%) having the highest value. Acetylation and oxidation enhanced the swelling power of the native starch while acid-thinning reduced it. Water and oil absorption capacities (WAC & OAC) of the starch improved after oxidation and acetylation whereas acid-thinning reduced them. Gel forming capacity of the native starch improved after acetylation and acid-thinning though ATSPS (7.0% w/v) had a better gelating property. The pasting temperature (86.25^{o}C), peak viscosity (220.42BU), final viscosity (256.08BU) and setback viscosity (208.41BU) of NSPS were reduced after modification. The paste clarity revealed that percentage transmittance (650nm) increased after modification with OSPS and ASPS showing improved paste clarity.

KEY WORDS: acetylation, acid-thinning, oxidation, retrogradation, sweet potato starch.

INTRODUCTION

Sweet potato (*Ipomoea batatas L.* (Lam)) is an important source of carbohydrate in tropical Africa. In Nigeria, it is consumed as fresh boiled roots and traditionally processed into low quality dried chips and flour primarily for domestic use and household food security. The high carbohydrate content of sweet potato and its wide availability makes it a very good source of starch for both domestic and industrial uses in Nigeria and tropical Africa. In the development of any food products from starchy crops, the knowledge of their physicochemical properties in particularly those of the starch which is the major component is needed to predict behaviour under given processing conditions [1]. The sweet potato starch is similar to other starch in being a biopolymer composed of anhydroglucose units and is the major storage energy in various plants in nature. The starch of sweet potato is composed of a mixture of amylose and amylopectin and is reported to posses A-type (high swelling) pattern and like those from many other roots and tubers, its starch granules are medium sized with a smooth round oval shape[2]. Sweet potato starch is used in many products including noodles, cakes, bread, biscuits, desserts, alcoholic and non-alcoholic drinks, puddings and confectionery products. Applications of starch in food systems are primarily governed by gelation, gelatinization, pasting, solubility, swelling, colour and digestibility[3]. Depending on the end use, any of these aformentioned properties can be altered by suitably modifying the starch to provide starch products with suitable properties to meet the needs for specific uses. Starch modification is often used to circumvent the limitations inherent in using biopolymers in its native state. Usually, starch modification can be accomplished through physical alteration, chemical degradation, enzymatic modifications or genetic transformation[4]. Oxidation as a form of chemical modification introduces carboxyl and carbonyl functional groups with subsequent depolymerisation of the starch. Such starches have been established to be whiter in colour and have restricted retrogradation or "setting up" on standing[5]. Acetylation of biopolymers is obtained by esterification of native starch with acetic anhydride and the modified starches generally show better paste clarity, stability, increased resistance to retrogradation and increased freeze-thaw stability[6]. Acid-modified or acid-thinned starch is a granular modification of the native starch achieved through treatment of starch below its gel point in aqueous acid suspension[7]. The present study was undertaken to isolate starch from the white cultivar of sweet potato tuber, subject it to chemical modifications (oxidation,acetylation and acid-thinning), and investigate the

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physicochemical and retrogradation characteristics of the native and modified starches with a view to providing relevant information towards their effective utilization particularly in the food industries.

MATERIALS AND METHODS

Materials

The white cultivar of sweet potato(*Ipomoea batatas L.* (Lam)) tubers were obtained from the National Root Crop Research Institute, Umudike, Abia State, Nigeria. All chemicals used were of analytical grade.

Starch Isolation

The sweet potato starch was obtained following the method of Lawal [8] with some modifications. The cleaned sweet potato tubers were peeled, weighed, sliced and ground for 2 min at high speed in a Waring blender with small volumes of distilled water. The homogenate was passed through an 80-mesh sieve. This grinding and screening operation was repeated four more times. The resulting starch slurry was filtered through a 200-micron screen, passed again through a 100-micron screen and centrifuged (Eltek centrifuge, MP 400R, Electrocraft, Mumbai, India) at 1500 rpm for 20 min. After removing the mucilagenous layer, the sediment was washed several times by suspension in distilled water and centrifuging until it appeared to be free of non-starch material. The sediment was then air dried at 30±2^{o}C for 48h. The dried starch cake was ground, passed through a 75-micron screen and stored in LDPE bags at ambient temperature till further use.

Starch Modification

Acetylation

The method of Sathe and Salunkhe [9] was adopted. Native sweet potato starch (100g) was dispersed in 500ml of distilled water and stirred magnetically for 20min. The pH of the slurry obtained was adjusted to 8.0 using 1.0M NaOH. Acetic anhydride (10.2g) was added slowy to the mixture while maintaining a pH range of 8.0–8.5. The reaction proceeded for 5min after the addition of acetic anhydride. The pH of the slurry was finally adjusted to 4.5 using 0.5M HCl. It was filtered, washed four times with distilled water and air dried at 30±2^{0}C for 48h.

Oxidation

This was performed according to the method described by Forssel *et al*[10]. The native sweet potato starch (100g) was mixed with 500ml of distilled water and the pH of the mixture adjusted to 9.5 with 2.0M NaOH. Ten grams (10g) of NaOCl was added to the slurry over a period of 30min with constant stirring while maintaining a pH range of 9.0 – 9.5. The reaction was allowed for 10min after all the NaOCl had been added. Subsequently, the pH was adjusted to 7.0 with 1M H_2SO_4 and the oxidized starch was filtered, washed four times with distilled water and air dried at 30±2^{0}C for 48h.

Acid-thinning

The method as described by Lawal [8] was used. The native sweet potato starch (100g) was slurried in 500ml of 0.15M HCl. The mixture was stirred magnetically for 8h while maintaining a temperature of 50^{0}C. The acid modified starch was filtered and the residue obtained was washed four times with distilled water and air dried at 30±2^{0}C for 48h.

Proximate analysis

The proximate analysis of the starch samples were estimated using the standard methods of AOAC [11] and Nielsen[12].

Physicochemical properties

These were determined by using aliquots from 10%m/v,db aqueous suspension of each starch sample at 30±2^{0}C in triplicates. The pH was determined by the method of AOAC[13] . The swelling index, solubility and iodine affinity were assayed according to the method described by Iwuoha[14]. Water and oil absorption capacities were determined using the method of Mbofung *et al.*[15].

Pasting Characteristics

The pasting characteristics of the starch samples were measured in a standard Brabender viscoamylograph (Brabender Instrument Inc., Duisburg, West Germany) according to the procedure described by Lawal *et al.* [16]. The

starch suspension (8%) was heated from 30^0C to 95^0C and kept at this temperature for 30mins before it was cooled to 50^0C. A constant rotational velocity of 75rpm was maintained and the heating as well as cooling rate was 1.5^0C/min throughout the process.

Light transmittance

The paste clarity was studied using the method of Bhandari and Singhal[17]. Fifty milligrams (on dry weight basis) of the starch samples were suspended in 5ml of distilled water using 10ml cotton-plugged test tubes. The test tubes were then heated in a boiling water bath (with occasional shaking) for 30min. After cooling to ambient temperature the percentage transmittance (%) was determined at 650nm against a water blank using a spectrophotometer (Hewlett-Packard spectrophotometer). Also, to monitor tendency for retrogradation, samples were stored for 24h at 4^0C to effect nucleation after which they were stored at 30 ± 2^0C for 1 – 10 days before determining the absorbance.

Gelation properties

The gelation studies were conducted according to the method described by Lawal[8] . Samples of starch,1-10%(w/v) were prepared in test tubes with 5ml of distilled water. The starch suspensions were mixed with a Vari-whirl mixer (Model A901, Salver Chem. Chicago, IL, USA) for 5min. The test tubes were heated for 30min at 80^0C in a water bath, followed by rapid cooling under running cold water. The test tubes were further cooled at 4^0C for 2h. Least gelation concentration was determined as that concentration when the samples from the inverted test tube did not fall down or slip.

Statistical analysis

All the analysis were carried out in triplicates and data obtained were analysed by analysis of variance(ANOVA) method using the General Linear Models procedure(GLM) and calculating the least significant difference(LSD) on an SAS (User's Guide Version 6) package[18].

RESULTS AND DISCUSSION

Proximate composition

The results of the native and modified sweet potato starches are presented in Table 1. The moisture content of the starches ranged from 10.07% - 10.23% with the acetylated starch having the least value and the native the highest. This compares favourably with the study carried out by Aiyeleye *et al* [19] on cassava starch and Raja and Sindhu [20] on arrowroot starch but is higher than the 8.02%-8.39% moisture level of similar work carried out on cocoyam starch by Lawal [8]. The ash content of the modified starches were lower compared to the native sweet potato starch(2.33%) with the OSPS (0.67%) having the least value This reduction can be attributed to the possible chemical degradation of the starch during modification and is in agreement with reports by Lawal [8] and Adebowale *et al*.[21]. In all the starch samples, the percentage protein and crude fibre were below 1% though some values were beyond detection limit. Also, the crude fat was entirely not detected in all the starch samples.

Physicochemical properties

The result of the physicochemical properties of the sweet potato starches as shown in table 2 indicates that the water absorption capacity (WAC) and oil absorption capacity (OAC) of the starches ranged from 0.80ml/g – 4.80ml/g and 0.35ml/g – 4.14ml/g respectively. The modification effect on the WAC and OAC of the starch samples were significantly different ($P \leq 0.05$). With regards to WAC, the acetylated starch have the highest value (4.80ml/g) followed by oxidation (0.87ml/g) and acid thinning the least value (0.73ml/g). This shows that hydrophilic tendency of the starches improved after oxidation and acetylation whereas acid thinning reduced the tendency of the starch to absorb water. Following similar trend, ASPS has the highest value of oil absorption (4.14ml/g) against 0.35ml/g and 0.15ml/g observed in OSPS and NSPS respectively, with ATSPS still maintaining the least value of 0.12ml/g. Hence, both hydrophilic and hydrophobic properties of the native starch were impaired after acid-thinning [8]. Also, the result revealed that acid-thinning and acetylation reduced the swelling power of the starches. Reduction in swelling power due to acid-thinning was more pronounced than the effect of acetylation. Similar reduction in swelling power had been reported after acid thinning and acetylation [22]. The solubility of the native sweet potato starch increased considerably after chemical modification. Oxidation, acetylation and acid thinning markedly improved solubility of the native starch with the oxidized starch having the highest value of 25.25%. This result confirmed the findings that acid modification also improved solubility of cassava starch [23]. The blue value index (BVI) increased after oxidation and acetylation but decreased after acid-thinning. The highest value (122.00ppm)

was obtained for OSPS followed by 83.67ppm, 50.11ppm and 21.33ppm for ASPS, NSPS and ATSPS respectively. This result suggest that oxidation caused a high degree of rupture or damage of the starch granules and reflects the amount of amylose content in the starches[24]. The pH of the starches varies between 6.30 -7.70 though oxidation had the most pronounced improvement on pH value.

Pasting characteristics

The pasting profile of the native and modified sweet potato starches are presented in table 3. The pasting temperature of the NSPS (86.25^0C) was reduced after acetylation, oxidation and acid-thinning. The reduction in pasting temperature of NSPS following modification indicates granular fragility [25] and is not only economical in terms of cooking energy, but also may have greater utilization in products that are susceptible to high temperature[26]. Compared with other starches the pasting temperature of NSPS is above 76^oC reported for cocoyam starch [8] and 80.4^0C reported for *D. dumetorum* [26]. Also the modification processes reduced the peak viscosity of the NSPS. The values obtained for peak viscosity ranged from 8.58-220.42BU with NSPS having the highest value (220.42BU). This is in agreement with the findings of Lawal [8] for cocoyam starches and reports by Adebowale *et al*. [21] and Atichokudomchai *et al*. [27]. The trough (holding viscosity) reduced considerably after modification of the native sweet potato starch. This indicates that modification greatly affect shear thinning during the holding period and NSPS which has the highest value (47.67BU) will have more ability to withstand breakdown during cooling than the modified starches. The breakdown viscosity which is a measure of fragility of starch[28] decreased following oxidation, acetylation and acid thinning. The NSPS has the highest value (172.75BU) and as such has the ability to withstand heating and shear stress which is an important factor for many processes especially those requiring stable paste and low retrogradation/syneresis[29]. The final viscosity of the sweet potato starches also reduced after modification with OSPS having the least value of 22.54BU. The considerable decrease in viscosity after oxidation was caused by partial cleavage of the glycosidic linkages upon oxidation thereby resulting in a decrease in molecular weight of starch molecules. This partially degraded network, was not resistant to shear and could not maintain the integrity of the starch granule thereby producing a lower viscosity[8].The setback value, an index of retrogradation tendency in the starch paste [30] was reduced after modification. This reduction indicates that new substituent groups have been introduced into the modified derivatives and this restricted the tendency of the starch molecules to realign after cooling, thereby encouraging a lower setback value for the modified starches [16]. In respect of peak time observed to attain peak viscosity, OSPS had the highest value (5.88min) and ASPS the least (5.22min). This suggests that deformation of the starch granules occurs easily in ASPS than in other starches.

Light transmittance (Paste clarity)

The effect of storage days on paste clarity of the sweet potato starches is shown in table 4. There was an increase in percentage transmittance at 650mm after modification. ASPS produced the most remarkable increase in % transmittance. The increase in % transmittance after modification can be attributed to chemical substitution of the hydroxyl groups in the starch molecules with acetyl, carbonyl and carboxyl functional groups. This causes repulsion between adjacent starch molecules and apparently reduces interchain association which facilitates improved % transmittance[8]. In all the starches the percentage transmittance was reduced as the storage days increase. However, pronounced reduction in percentage (%) transmittance was observed in NSPS and ATSPS. This is in agreement with report by Bello-perez *et al*.[31] for banana starch and Lawal [8] for cocoyam starch.

Gelation properties

The gelation properties of the native and modified sweet potato starches are presented in table 5. In line with the least gelation concentration (LGC) which is considered an index of gelation capacity, the result obtained indicates that lowest LGC was observed in ATSPS and the highest in NSPS and OSPS. The LGC of the NSPS is 9% (w/v) and also was the same after oxidation but reduced after acetylation (8%w/v) and acid-thinning (7%w/v). This indicates that oxidation had no effect on the gel strength of the native starch but suggests that ASPS and ATSPS are better gelating food additives than NSPS though ATSPS has the best and enhanced gelating property This is in agreement with report by Lawal [8] on cocoyam starch and Wang and Wang [7] on improvement of gelation capacity of corn starch, potato starch and rice starch following acid thinning. Similarly, it confirms the report by Kim and Ahn [23] that acid hydrolysis improved the gel strength of acid modified red bean starch over the unmodified starch.

Table 1: Proximate composition* of modified sweet potato starches

STARCHES	COMPOSITION (%)				
	Moisture	Ash	Crude fibre	Fat	protein
NSPS	10.23±0.01[a]	2.33±0.01[a]	0.11±0.02	ND	0.15±0.04[a]
OSPS	10.11±0.02[b]	0.67±0.01[d]	ND	ND	ND
ATSPS	10.21±0.02[a]	1.67±0.01[b]	ND	ND	0.12±0.05[a]
ASPS	10.07±0.03[b]	1.40±0.02[c]	ND	ND	ND
LSD	0.055	0.021	NIL	NIL	0.098

* Triplicate determinations
ND – Not detected
NSPS – Native sweet potato starch, OSPS – oxidized sweet potato starch, ATSPS – Acid-thinned sweet potato starch and ASPS – Acetylated sweet potato starch.
LSD – Least significant difference.
Means within columns with different superscripts are significantly different (P≤0.05).

Table 2: Physicochemical properties of sweet potato starches.

	STARCHES			
Property	NSPS	OSPS	ATSPS	ASPS
Water absorption capacity (ml/g)	0.80±0.08[b]	0.87±0.05[b]	0.73±0.05[b]	4.80±0.19[a]
Oil absorption capacity (ml/g)	0.15±0.04[c]	0.35±0.014[b]	0.12±0.04[c]	4.14±0.27[a]
Swelling index (cm^3/cm^3)	1.14±0.01[b]	1.15±0.01[b]	1.07±0.02[c]	1.21±0.05[a]
Solubility (%)	8.03±0.91[d]	25.25±0.80[a]	16.03±0.96[c]	17.48±0.31[b]
Blue value index (ppm)	50.11±0.88[c]	122.00±0.68[a]	21.33±0.41[d]	83.67±0.60[b]
pH	6.70±0.12[c]	7.70±0.21[a]	6.30±0.23[d]	7.05±0.13[b]

Values are means of triplicate determinations.
Means within rows with different superscripts are significantly different (P≤0.05).

NSPS, OSPS, ATSPS and ASPS are as defined in table 1.

Table 3: Pasting characteristics of sweet potato starches.

Pasting Property	Starches			
	NSPS	OSPS	ATSPS	ASPS
Peak viscosity (BU)	220.42	8.55	79.17	58.88
Trough (BU)	47.67	0.66	0.59	2.30
Breakdown (BU)	172.75	7.92	78.58	56.58
Final viscosity (BU)	256.08	22.25	129.67	104.08
Setback (BU)	208.41	21.59	129.08	101.78
Peak Time (min)	5.66	5.88	5.48	5.22
Pasting temp. (^0C)	86.25	85.33	85.26	84.75

BU – Brabender unit

NSPS, OSPS, ATSPS and ASPS are as defined in table 1.

Table 4: Effect of storage days on paste clarity of sweet potato starch.

Starch	Percentage transmittance (650nm)					
Samples	1st day	2nd day	4th day	6th day	8th day	10th day
NSPS	27.08±0.09[a]	17.78±0.06[b]	12.81±0.21[b]	11.58±0.14[b]	10.46±0.35[b]	7.22±0.96[b]
OSPS	33.83±0.33[a]	33.00±0.43[a]	32.89±0.1[a]	32.84±0.87[a]	32.51±0.65[a]	32.14±0.87[a]
ATSPS	34.86±0.43[a]	17.04±0.74[b]	12.36±0.53[b]	10.65±0.89[b]	9.06±0.52[b]	5.12±0.62[b]
ASPS	38.91±0.31[a]	37.78±0.61[a]	36.65±0.08[a]	36.38±0.23[a]	35.84±0.22[a]	31.20±0.77[a]

Values are means of triplicate determinations.
Means within columns with different superscripts are significantly different (P≤0.05).

NSPS, OSPS, ATPS and ASPS are as defined in table 1.

Table 5: Gelation properties of sweet potato starches.

Concentration (% w/v)	Starches			
	NSPS	OSPS	ATSPS	ASPS
1	NG	NG	NG	NG
2	NG	NG	NG	NG
3	NG	NG	NG	NG
4	NG	NG	NG	NG
5	NG	NG	NG	NG
6	NG	NG	NG	NG
7	NG	NG	GEL	NG
8	NG	NG	GEL	GEL
9	GEL	GEL	GEL	GEL
10	GEL	GEL	GEL	GEL
LGC (%)	9.0	9.0	7.0	8.0

NG – Not gel
LGC – least gelation concentration

NSPS, OSPS, ATSPS and ASPS are as defined in table 1.

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