Effects of tempering (annealing), acid hydrolysis, low-citric acid substitution on chemical and physicochemical properties of starches of four yam (*Dioscorea* spp.) cultivars

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Abstract The effects of tempering (annealing), acid hydrolysis and low-citric acid substitution on chemical and physicochemical properties of starches of four Nigerian yam cultivars were investigated. Crude fat and protein contents of the native starches decreased significantly after the modifications, while nitrogen-free extract increased significantly with acid hydrolysis and citric acid substitution. Acid hydrolysis and low-citric acid substitution reduced the least concentration for gel formation of the starches from 4 to 2% w/v, but tempering had no effect. Swelling power of the starches reduced significantly, and water solubility increased significantly at 75 and 85 °C, especially with acid hydrolysis and low-citric acid substitution. However, tempering significantly reduced starch solubility in the four cultivars. Paste clarity of starches of white (29.17%), water (18.90%), yellow (30.90%) and bitter (10.57%) yams reduced significantly with tempering to 14.43, 11.83, 16.93 and 7.27%, but increased significantly with acid hydrolysis to 41.40, 35.37, 28.77 and 32.33%, and low-citric acid substitution to 36.60, 44.17, 50.67 and 14.33%, respectively. Pasting properties such as peak, trough, breakdown, final, and setback viscosities and peak time of native starches reduced significantly with acid hydrolysis and low-citric acid substitution, however, tempering significantly increased their pasting temperature, peak time, setback and final viscosities.

Keywords Yam starches · Starch modifications · Physicochemical properties · Tempering · Citric acid substitution · Acid hydrolysis

Introduction

Yam ranks next to cassava in order of economic importance in West Africa (Opara 2003). Yam tubers are an important source of carbohydrates, which comprises 17–38% of its fresh weight (Orkwor 1998). It is an economic staple crop as well as a good source of calories for many people of the sub-Sahara region, especially in West Africa. It is also an important source of food in several other tropical regions including East Asia, South America, and South-East Asia, including India (Iwueke et al. 2003). Traditionally, yam tubers were kept underground in the soil or underground structures or stored in barns because of the susceptibility to spoilage. Postharvest losses of yams not treated with fungicides during storage could vary between 10–15% and 30–50% after three and six months, respectively (Opara 2003). Hence, production of starches from yams would help reduce wastage, enhance value addition, and revenue generation (Odeku and Picker-Freyer 2007). Due to the relatively high carbohydrate content, yam is considered as a source from which starch could be extracted for both household and industrial applications.

Native starches have many uses in food systems, but their use is limited by properties, such as low shear and thermal resistance (Singh and Kaur 2009). Thus, native starches could be modified as appropriate for industrial food applications. Modified starches have numerous food and non-food applications because their physicochemical properties and other attributes (viscosity, stability to acid, heat, and shear, heat penetration, and workability) which

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have been altered chemically or physically for specific uses (Ortega-Ojeda et al. 2005). There are numerous methods for starch modification.

Tempering (annealing) is physical modification method involving heating slurry of starch at temperatures below gelatinization point for prolonged periods of time, while acid hydrolysis, to generate low molecular weight modified starch, is usually carried out by treatment of starch with hydrochloric acid, O-phosphoric acid or sulphuric acid (Collado and Corke 1999; Singh and Kaur 2009). Derivatization by chemical substitution of hydroxyl group on starch molecules by esters under alkaline conditions is another common modification technique (Singh and Kaur 2009; Singh et al. 2007; Agboola et al. 1991).

Potato and cassava have been explored as sources of commercial starches, however, yams have been comparatively underutilized (Brunnschweiler et al. 2005; Odeku and Picker-Freyer 2007). Moreover, literature is sparse on the effect of tempering, acid hydrolysis and, particularly, low-citric acid-substitution on chemical and physico-chemical properties of Nigerian yam cultivars in a comparative manner. Few available literature had focused on properties of native yam starches for tablet formation (Odeku and Picker-Freyer 2007; Zuluaga et al. 2007), citric and ascorbic acid treatment of white yam flour (Akubor 2013), acid hydrolysis of water and white yam starches in disintegrant or excipient development (Odeku and Akinwande 2012), and γ -irradiation of white and water yams (Falade et al. 2011). Also, Falade and Ayetigbo (2015) had studied the effects of annealing, acid hydrolysis and low-citric acid modifications on physical and functional properties of yam starches. The objective of the research was to investigate the effects of tempering, acid hydrolysis and low-citric acid-substitution on the chemical and physico-chemical properties of starches of four Nigerian yam cultivars.

Materials and methods

White (*Dioscorea rotundata* Poir), water (*Dioscorea alata* L.), yellow (*Dioscorea cayenensis* Lam), and white bitter (*Dioscorea dumetorum* Pax) yam cultivars were obtained from the yam section of Bodija market in Ibadan, South-west Nigeria, and were identified at the International Institute of Tropical Agriculture (IITA), Ibadan, Nigeria.

Starch extraction and determination of yield

Starch was extracted from the yam cultivars using the modified method of Walter et al. (2000). The modified method was well described by Falade and Ayetigbo (2015).

The yield of extracted starch was calculated as weight of dry starch per weight of yam flesh.

Tempering modification of yam starches

Tempering modification of yam starches were carried out using the method described by Jacobs and Delcour (1998). Starch was suspended in distilled water (1:2 w/v) and heated without stirring in water bath at 50 °C for 24 h in beakers sealed with aluminum foil. The suspension was decanted, air-dried at 30 °C for 72 h, milled and kept in Ziploc® bags at 27 ± 2 °C.

Acid hydrolysis of yam starches

Native starch (300 g) was suspended in 600 mL of HCl (6% w/v) at 27 ± 2 °C for 192 h without stirring (Atichokudomchai and Varavinit 2003). The slurry was neutralized with alkali (10% w/v NaOH) solution to terminate the hydrolysis. The starch slurry was washed five times with distilled water and oven-dried at 45 °C for 24 h. Dried starch cake was subsequently dry milled using a laboratory mill. The milled starch was filtered through a 250 μ m mesh sieve and the resulting powder was stored in Ziploc® double zipper (26.8 cm $\times$ 27.3 cm $\times$ 100 μ m) bags (Ziploc Brand Products, WI, USA). The yield of acid hydrolysed starch was expressed as percentage of the weight of recovered starch to the initial weight of starch.

Preparation of low-citric acid-substituted starches

Low-citric acid substituted starches were produced using the method described by Agboola et al. (1991). Starch (300 g) was suspended in 400 mL of distilled water, and about 10 mL of 1 M NaOH was used to adjust the pH of the slurry to about 9. The alkaline slurry was held for 30 min with occasional stirring. A 15% citric acid monohydrate and 1% H₂SO₄ (both based on starch dry weight) mixture was made up to 100 mL with distilled water. The mixture was added to the alkalized slurry and held for 5 h at room temperature (28 ± 2 °C) and then washed with 600 mL of distilled water, filtered, dried at 50 °C for 24 h, milled, packaged and stored.

Determination of chemical composition of native and modified starches

Moisture content was determined gravimetrically by drying in a cross flow Gallenkamp Size two BS model OV-160 hot air oven (Gallenkamp, England, UK) at 102 °C until constant weight (AOAC 2011). Protein and fat contents were determined using standard Kjeldahl distillation (AOAC 2011) and solvent extraction method, respectively. Ash

content (%) was calculated after 5 g of starch sample was incinerated in a furnace (Carbolite AAF 11/18, England) at 600 °C for 5 h. Total carbohydrate (Nitrogen-free extract, NFE) was determined by difference. i.e. % NFE = 100% – (Ash + Crude fat + Moisture + Crude protein) %. To determine pH, 1 g of starch was dispersed in 25 mL of distilled water at 27 ± 2 °C and agitated for 5–10 min (Sokhey and Chinnaswamy 1993). The pH was measured using pH meter (JENWAY Model 3520, Bibby Cci. Ltd., Essex, UK) which was previously calibrated with buffer 4.0 and 7.0.

Apparent amylose, swelling power and solubility of native and modified yam starches

Apparent amylose content (hereafter referred to simply as amylose) was determined using the method of Hoover and Ratnayake (2002). Amylose content (%) was determined using a spectrophotometer (Spec UNICO® 1100 RS, United products and Instruments Inc.) at 620 nm using the iodine binding method. Swelling power and solubility were determined using the method of Collado and Corke (1997). Sample (0.5 g) was mixed with 15 mL distilled water in centrifugal tubes, and the suspension was heated at 55, 65, 75 and 85 for 30 min. The gelatinized sample was cooled to 27 ± 2 °C and centrifuged (HIMAC CR 21GII, Hitachi Koki Co. Ltd., Japan) at $200 \times g$ for 15 min. The supernatant was decanted in a petri dish and dried at 100 °C to constant weight. Solubility was expressed in percentage weight of dried solid based on weight of dry sample. Swelling power was presented as the ratio of the weight of the wet sediment to the weight of initial dry sample.

Least gelation concentration (LGC) of native and modified yam starches

Least concentration required for gelation of the starches was determined using the method described by Desphande (1982). The LGC (% w/v) was determined as the least concentration when sample from the upturned tube did not fall or slip.

Starch paste clarity

Starch paste clarity was determined by the method of Bello-Pérez et al. (1999) by measuring percentage transmittance of a 1% (w/w) starch suspension at 650 nm, using a spectrophotometer (Spec UNICO® 1100 RS, United products and Instruments Inc.). Starch suspension in tubes was placed in a water bath at 100 °C for 30 min, shaken every 5 min and left to cool to 27 ± 2 °C. Percentage transmittance (%T) of these suspensions was determined.

Pasting properties of native and modified yam starches

Pasting characteristics of native and modified starches (3 g) were determined using a Rapid Visco Analyzer (RVA-super 4, Newport Scientific® Pty Ltd., Warriewood, Australia). The procedure involved a 13 min cycle program with temperature regime as follows: an idle temperature of 50 °C for 1 min, heating from 50 to 95 °C in 3 min 45 s, holding at 95 °C for 2 min 30 s, cooling to 50 °C for 3 min 45 s, and holding at 50 °C for 2 min. Pasting parameters were analyzed by *Thermocline*® for Windows® RVA software. In order to further characterize the pasting behaviors, the viscosity breakdown ratio (BDR) was defined as the ratio of trough to peak viscosity (Waramboi et al. 2011), and used to classify the cultivars as highly (<0.5), moderately (0.5–0.8) or slightly (0.8–1.0) shear-thinning, or shear-thickening (≥ 1.0).

Statistical analysis

Data of replicated experiments were analyzed by analysis of variance (ANOVA) statistical method using SPSS16 (Chicago, IL.) software for Windows®. Means were separated using Duncan's multiple range test. Significant differences were established at $p \leq 0.05$. Pearson's correlation of the parameters was determined at statistical significance level of 5% ($p < 0.05$).

Results and discussion

Chemical composition of native and modified starches

Moisture content of yam starches (10.02–19.70%) varied significantly with the cultivars and modification methods (Table 1). The tempered starches showed higher moisture content than the native, acid hydrolysed and low-citric acid substituted starches. Tempered African yam bean and mucuna bean starches showed higher moisture content than their native counterparts as reported by Adebawale et al. (2009) and Adebawale and Lawal (2003) respectively. Both native and modified starches had low ash content (0.1–0.7%). Crude fibre was not detected, while the crude fat of the starches ranged from 0.11 to 0.69%. Comparable values of crude fat for native yam starches (0.28–0.69%) have been reported (Rugchati and Thanacharoenchanaphas 2010; Odeku and Picker-Freyer 2007; Zuluaga et al. 2007). Modification of the starches significantly reduced the crude fat contents. In other studies, reduction in crude fat of tempered African yam bean starch and acid-hydrolysed hybrid maize starch has been reported (Lawal et al. 2005;

Table 1 Chemical composition of native and modified yam starches

Cultivar	Modification	Moisture (%)	Ash (%)	Crude fat (%)	Crude protein (%)	NFE (%)	Amylose (%)	pH
White yam	Native	15.70 ^d ± 0.19	0.30 ^{bcd} ± 0.14	0.31 ^c ± 0.06	1.88 ^{bc} ± 0.31	81.82 ^{ef} ± 0.09	33.54 ^{cd} ± 1.13	6.69 ^c ± 0.06
	Tempered	18.31 ^c ± 0.04	0.20 ^{cd} ± 0.00	0.25 ^{cdef} ± 0.03	1.26 ^{bc} ± 0.86	79.98 ^{fg} ± 0.79	31.43 ^{de} ± 0.74	4.72 ^g ± 0.11
	Acid hydrolysed	10.87 ^k ± 0.08	0.20 ^{cd} ± 0.00	0.20 ^{efgh} ± 0.01	0.7 ^{6c} ± 0.75	87.98 ^a ± 0.67	31.18 ^e ± 0.99	8.73 ^a ± 0.13
	Citric substituted	11.45 ^j ± 0.18	0.30 ^{bcd} ± 0.14	0.11 ⁱ ± 0.01	0.54 ^c ± 0.48	87.60 ^{ab} ± 0.44	8.42 ^j ± 1.86	3.31 ^{jk} ± 0.04
Water yam	Native	14.87 ^{ef} ± 0.12	0.30 ^{bcd} ± 0.14	0.28 ^{cde} ± 0.04	2.93 ^b ± 1.05	81.62 ^f ± 1.07	36.51 ^b ± 1.17	6.69 ^c ± 0.05
	Tempered	18.94 ^b ± 0.04	0.40 ^{bc} ± 0.00	0.22 ^{defg} ± 0.02	2.21 ^{bc} ± 0.33	78.23 ^g ± 0.31	36.29 ^b ± 0.35	4.42 ^h ± 0.11
	Acid hydrolysed	10.51 ^l ± 0.12	0.30 ^{bcd} ± 0.14	0.14 ^{ghi} ± 0.02	0.39 ^c ± 0.29	88.68 ^a ± 0.58	31.82 ^{de} ± 1.38	6.37 ^d ± 0.11
	Citric substituted	10.76 ^{kl} ± 0.42	0.20 ^{cd} ± 0.00	0.15 ^{ghi} ± 0.01	0.52 ^c ± 0.33	88.37 ^a ± 0.09	15.02 ^h ± 1.60	4.24 ⁱ ± 0.17
Yellow yam	Native	14.64 ^f ± 0.22	0.20 ^{cd} ± 0.00	0.29 ^{cd} ± 0.05	1.38 ^{bc} ± 0.94	83.50 ^{de} ± 1.11	38.92 ^a ± 0.17	6.68 ^c ± 0.13
	Tempered	19.70 ^a ± 0.05	0.20 ^{cd} ± 0.00	0.20 ^{efgh} ± 0.03	1.41 ^{bc} ± 1.30	78.49 ^g ± 1.38	36.45 ^b ± 0.22	5.65 ^f ± 0.12
	Acid hydrolysed	11.06 ^k ± 0.04	0.50 ^{ab} ± 0.14	0.19 ^{fghi} ± 0.03	0.58 ^c ± 0.17	87.67 ^{ab} ± 0.39	34.06 ^c ± 0.13	7.27 ^b ± 0.09
	Citric substituted	13.01 ^h ± 0.04	0.10 ^d ± 0.14	0.18 ^{fghi} ± 0.03	0.68 ^c ± 0.06	86.03 ^{bc} ± 0.13	12.55 ⁱ ± 0.35	3.47 ^j ± 0.04
Bitter yam	Native	14.12 ^g ± 0.04	0.50 ^a ± 0.14	0.69 ^a ± 0.04	5.49 ^a ± 1.56	79.21 ^g ± 1.43	21.02 ^f ± 0.13	6.20 ^e ± 0.09
	Tempered	15.17 ^e ± 0.11	0.40 ^{bc} ± 0.00	0.49 ^b ± 0.05	2.78 ^b ± 1.54	81.16 ^f ± 1.38	20.72 ^f ± 0.13	5.58 ^f ± 0.04
	Acid hydrolysed	12.63 ⁱ ± 0.11	0.20 ^{cd} ± 0.00	0.23 ^{cdef} ± 0.04	1.76 ^{bc} ± 0.07	85.18 ^{cd} ± 0.00	18.21 ^g ± 0.13	5.64 ^f ± 0.06
	Citric substituted	10.02 ^m ± 0.04	0.70 ^{ab} ± 0.14	0.12 ^{hi} ± 0.01	1.63 ^{bc} ± 0.82	87.53 ^{ab} ± 0.73	12.97 ⁱ ± 1.56	3.30 ^k ± 0.02

Means in columns not followed by same alphabet(s) are significantly different at 5% level ($p < 0.05$)

Adebowale et al. 2009). Fat-starch interaction may influence functional, thermal and retrogradation properties of starches (Singh and Kaur 2009). The crude protein of the starches ranged from 0.385 to 5.488% (Table 1). Native bitter yam starch had higher crude protein than other native yam starches, indicating that bitter yam starch granules may be more associated to protein bodies (Otto et al. 1997) than the other yam starches. Comparable protein contents of 0.28–2.89% (Odeku and Picker-Freyer 2007), 3.23% (Aprianita et al. 2009) and 1.22–2.27% (Olayinka et al. 2011) has been reported for yams, Australian *D. alata*, and red and white sorghum starches, respectively. All modifications significantly reduced crude protein of the native starches. For instance, tempering reduced crude protein of the yam starches significantly. Adebowale et al. (2009) likewise reported reduction in crude protein of African yam bean starch after tempering. Acid hydrolysis and low-citric acid substitution may have caused significant reduction in protein content by hydrolysis and denaturation due to pH adjustment. Most proteins precipitate out of solutions when adjusted to their iso-electric point. Nitrogen-free extract (NFE) of the starches ranged between 78.23 and 88.68%. In all, low crude protein (except for native bitter yam

starch), crude fat and other non-starch compositions indicated low presence of undesirable constituents, and high purity of the starches.

Amylose content of native and modified starches

Apparent amylose content of the native (21.02–38.92%) yam starches varied significantly (Table 1). Literature showed comparable amylose contents reported for native starches of water yam (27.47–41.90%) (Oke et al. 2013), water (28.99–33.11%) and white (27.45–32.80%) yams (Otegbayo et al. 2011), and *D. alata* (21.69–31.56%) and *D. rotundata* (27.36%) (Wireko-Manu et al. 2011). Native bitter and yellow yam starches had lowest (21.02%) and highest (38.92%) apparent amylose contents, respectively. Differences in amylose/amylopectin ratio of native starches could be due to enzyme activity involved in starch biosynthesis, genetic variation, climatic condition, cultivation practice, and botanical source (Odeku and Picker-Freyer 2007).

Apparent amylose content of tempered, acid hydrolysed and citric acid substituted yam starches significantly varied from 20.72 to 36.45%, 8.21 to 34.06% and 8.42 to 15.02%,

respectively (Table 1). Tempering significantly reduced apparent amylose content of native white and yellow yam starches from 33.54 to 31.43% and 38.92 to 36.45%, respectively, probably due to some amylose leaching as well as partial hydrolytic effect of the heat treatment. Similar findings have been reported for African yam bean starch where tempering resulted in reduction in the apparent amylose content from 35.2 to 29.8% (Adebawale et al. 2009). However, tempering increased apparent amylose content of black and pinto bean starches (Simsek et al. 2012). Tempering may have caused physical reorganization and partial hydrolysis of starch without disrupting granular structure.

Acid hydrolysis resulted in a significant decrease in the apparent amylose content of the native yam starches, particularly, water and yellow yam starches from 36.51 to 31.82% and 38.92 to 34.06%, respectively. The decrease could be due to the degrading or hydrolytic effect of hydrochloric acid on amylose chains in the starch granules. Amylose content of hybrid maize starch derivative reduced significantly from 20.42% to 17.65% after acid hydrolysis (Lawal et al. 2005). Low-citric acid substitution resulted in significant reduction in the apparent amylose content of the starches. For instance, native white yam and bitter yam starches decreased from 33.54 to 8.42%, and from 21.02 to 12.97%, respectively. Citric acid modification may have resulted in acidification, a condition which may have caused the hydrolysis of amylose.

Swelling power (SP) and solubility of native and modified starches

The SP and solubility of the starches varied with temperature, yam cultivar and modification method. The SP of the yam starches increased with increasing temperature (Table 2). The SP at 55, 65, 75 and 85 °C ranged from 1.91 to 2.55, 1.98 to 2.69, 2.07 to 9.64 and 0.62 to 13.82 g/g, respectively. At 55 and 65 °C, tempering significantly reduced the SP in starches of all yam cultivars studied except for white yam starch. At these temperatures, acid hydrolysis and citric acid substitution either significantly reduced SP or had no significant effect. As temperature increased to 75 and 85 °C, the SP consistently and significantly decreased in all the modified starches. Physical reorganization and reinforcement within starch granules during tempering, and weakening of crystalline and amorphous regions during acid modifications could be adduced for the reduced SP. In addition, increased leaching of amylose and its diluent effect (Tester and Morrison 1990) could have limited the swelling. Based on cultivar differences, native bitter yam starch had significantly higher swelling power (13.82 g/g) than native yellow (10.69 g/g), water (8.77 g/g), and white (8.63 g/g) yam

starches at 85 °C. This may be due to its small granule, relatively low amylose content, and higher specific surface area (Singh and Kaur 2009). Smaller granules of potato and sweet potato cultivars' starches have been discovered to have higher hydration and swelling power than larger granules (Singh and Kaur 2009).

Solubility of the native and modified starches subjected to elevated temperatures of 55, 65, 75 and 85 °C varied from 2.0 to 10.0%, 2.0 to 20.0%, 2.0 to 64.67% and 2.0 to 84.67%, respectively. At 55 and 65 °C, low solubility could have resulted from possible leaching of amylose from the starch granules. Low-citric acid substitution significantly increased solubility at 55 and 65 °C consistently; tempering had no effect, while acid hydrolysis gave no definite trend. However, at 75 and 85 °C, solubility significantly reduced for tempered starches, but increased significantly for acid hydrolysed and citric acid substituted starches. Tempering may have induced rearrangement and reinforced packing within starch, thus limiting access to water penetration. Reduced solubility has been reported for tempered red sorghum starch at 80 °C (Adebawale et al. 2005), and tempered cassava starch at 85 °C (Chatakanda et al. 2011). Acid hydrolysis may have resulted in increased solubility by de-polymerization and alteration of starch crystalline and amorphous region as reported for maize starch by Lawal et al. (2005). Increased solubility of the citric acid substituted starches could be due to substitution of selected –OH sites by hydrophilic citrate group on starch molecules. Based on cultivar differences, native bitter yam starch had significantly higher solubility (14.67%) at 85 °C than native yellow (7.33%), water (8.67%), and white (6.67%) yam starches. This may be due to the relative higher protein bodies; and low, but readily leachable amylose content of the starch.

Least gel formation concentration (LGC) of native and modifies starches

The LGC of the native starches varied between 2 and 4% w/v (Table 2). Modification of the starches enhanced their gelling capacity. Tempering had no effect on LGC of native yellow, bitter and water yam starches, but reduced the concentration required for gel formation by the native white yam starch. Adebawale et al. (2009) and Adebawale and Lawal (2003) reported that the LGC of African yam bean starch and *mucuna* bean starch were improved by tempering, respectively. Tempering may have rearranged parts of amylose and amylopectin arrays, forming crystalline micelles connected together by molecular filaments which improved the strength and rigidity of the starch gel (Adebawale et al. 2009). The LGC of the acid hydrolysed and low-citric acid substituted starches improved from 4% (native) to 2% (Table 2) across cultivars, except for bitter

Table 2 Physicochemical properties of native and modified yam starches

Cultivar	Modification	Swelling power (g/g)				Solubility (%)				LGC (%) w/v	Paste clarity (%)
		55 °C	65 °C	75 °C	85 °C	55 °C	65 °C	75 °C	85 °C		
White yam	Native	2.11 ^{efg} ± 0.08	2.00 ^g ± 0.04	5.29 ^c ± 0.23	8.63 ^{de} ± 0.24	2.00 ^c ± 0.00	2.00 ^e ± 0.00	2.67 ^h ± 1.15	6.67 ^{ghi} ± 2.31	4	29.17 ^e ± 2.02
	Tempered	2.13 ^{efg} ± 0.01	1.98 ^g ± 0.09	5.03 ^{cd} ± 0.15	9.27 ^c ± 0.14	2.00 ^c ± 0.00	2.00 ^e ± 0.00	2.00 ^h ± 0.00	2.67 ^{hi} ± 1.15	2	14.43 ^{fg} ± 1.63
	Acid hydrolysed	2.05 ^{gh} ± 0.20	2.00 ^g ± 0.02	2.57 ^{gh} ± 0.06	4.37 ^h ± 0.22	2.00 ^c ± 0.00	3.33 ^c ± 1.151	12.00 ^e ± 2.00	42.00 ^e ± 3.46	2	41.40 ^{bc} ± 1.22
	Citric modified	2.09 ^{fg} ± 0.04	2.08 ^{defg} ± 0.02	2.07 ⁱ ± 0.03	1.14 ⁱ ± 0.17	4.67 ^b ± 1.15	9.33 ^c ± 1.15	33.33 ^c ± 2.31	82.00 ^a ± 0.00	2	36.60 ^{cd} ± 2.69
Water yam	Native	2.33 ^{cd} ± 0.08	2.15 ^{cde} ± 0.06	5.15 ^{cd} ± 0.21	8.77 ^{cde} ± 0.30	3.33 ^{bc} ± 1.15	2.67 ^e ± 1.15	13.33 ^e ± 1.15	8.67 ^g ± 1.15	4	18.90 ^f ± 3.66
	Tempered	1.94 ^{hi} ± 0.04	1.99 ^g ± 0.07	4.73 ^{de} ± 0.03	8.44 ^{ef} ± 0.27	3.33 ^{bc} ± 2.31	3.33 ^c ± 1.15	7.33 ^{fg} ± 1.15	2.00 ⁱ ± 0.00	4	11.83 ^{gh} ± 2.00
	Acid hydrolysed	2.09 ^{fg} ± 0.03	2.09 ^{cdefg} ± 0.05	2.36 ^{hi} ± 0.04	3.77 ⁱ ± 0.47	2.00 ^c ± 0.00	2.67 ^e ± 1.15	10.67 ^{ef} ± 1.15	42.00 ^e ± 5.29	2	35.37 ^{cde} ± 4.41
	Citric modified	2.16 ^{efg} ± 0.02	2.02 ^{fg} ± 0.00	2.23 ^{hi} ± 0.05	1.63 ^k ± 0.05	4.00 ^{bc} ± 0.00	7.33 ^d ± 1.15	28.00 ^d ± 2.00	72.67 ^b ± 5.77	2	44.17 ^b ± 0.59
Yellow yam	Native	2.24 ^{cde} ± 0.07	2.61 ^a ± 0.06	9.64 ^a ± 0.42	10.69 ^b ± 0.38	2.00 ^c ± 0.00	2.67 ^e ± 1.15	7.33 ^{fg} ± 3.06	7.33 ^{gh} ± 1.15	4	30.90 ^{de} ± 1.56
	Tempered	1.91 ⁱ ± 0.05	2.13 ^{cdef} ± 0.06	8.67 ^b ± 0.20	7.99 ^f ± 0.29	2.67 ^c ± 1.15	3.33 ^c ± 1.15	5.33 ^{gh} ± 1.15	4.67 ^{ghi} ± 1.15	4	16.93 ^{fg} ± 4.05
	Acid hydrolysed	2.17 ^{efg} ± 0.09	2.21 ^c ± 0.05	4.47 ^e ± 0.13	3.25 ^j ± 0.25	2.00 ^c ± 0.00	2.00 ^e ± 0.00	34.00 ^c ± 2.00	64.00 ^c ± 2.00	2	28.77 ^e ± 4.24
	Citric modified	2.21 ^{def} ± 0.08	2.06 ^{efg} ± 0.05	2.09 ^j ± 0.09	0.62 ^m ± 0.09	10.00 ^a ± 2.00	20.00 ^a ± 0.00	64.67 ^a ± 1.15	84.67 ^a ± 4.16	2	50.67 ^a ± 8.22
Bitter yam	Native	2.47 ^{ab} ± 0.05	2.43 ^b ± 0.08	4.73 ^{de} ± 0.77	13.82 ^a ± 0.64	2.67 ^c ± 1.15	2.67 ^e ± 1.15	5.33 ^{gh} ± 1.15	14.67 ^f ± 3.06	2	10.57 ^{gh} ± 1.17
	Tempered	2.35 ^{bc} ± 0.05	2.19 ^{cd} ± 0.11	3.72 ^f ± 0.17	9.01 ^{cd} ± 0.32	2.67 ^c ± 1.15	2.67 ^e ± 1.15	2.67 ^h ± 1.15	4.00 ^{ghi} ± 0.00	2	7.27 ^h ± 2.50
	Acid hydrolysed	2.35 ^{bc} ± 0.03	2.41 ^b ± 0.05	2.93 ^g ± 0.29	3.14 ^j ± 0.03	4.00 ^{bc} ± 0.00	8.00 ^{cd} ± 0.00	44.00 ^b ± 5.29	62.67 ^c ± 2.31	2	32.33 ^{de} ± 5.83
	Citric modified	2.55 ^a ± 0.01	2.69 ^a ± 0.12	4.28 ^e ± 0.06	5.01 ^g ± 0.13	8.67 ^a ± 1.15	12.00 ^b ± 2.00	42.00 ^b ± 2.00	50.67 ^d ± 1.15	2	14.33 ^{fg} ± 4.22

LGC Least gelation concentration

Means in columns not followed by same alphabet(s) are significantly different at 5% level ($p < 0.05$)

Table 3 Pasting properties of native and modified yam starches

Cultivar/modification	Peak viscosity (cP)	Trough viscosity (cP)	Breakdown viscosity (cP)	Final viscosity (cP)	Setback viscosity (cP)	Peak time (min.)	Pasting temp. (°C)	Breakdown ratio (BDR)
White yam								
Native	8415.0 ^a ± 100.41	6464.5 ^a ± 71.42	1950.5 ^e ± 28.99	9231.0 ^b ± 100.41	2766.5 ^{cd} ± 28.99	5.10 ^b ± 0.04	82.88 ^{cd} ± 0.60	0.77 (MST)
Tempered	7590.5 ^b ± 177.48	6321.5 ^a ± 153.44	1269.0 ^f ± 24.04	10176.0 ^a ± 138.59	3854.5 ^b ± 14.85	5.13 ^b ± 0.00	82.90 ^{cd} ± 0.57	0.83 (SST)
Acid hydrolysed	141.0 ^e ± 1.41	75.0 ^g ± 1.41	66.0 ^g ± 0.00	118.0 ^f ± 0.00	43.0 ^f ± 1.41	4.70 ^c ± 0.05	89.05 ^a ± 0.28	0.53 (MST)
Citric substituted	20.5 ^e ± 0.71	10.5 ^g ± 0.71	10.0 ^g ± 0.00	16.0 ^f ± 0.00	5.5 ^f ± 0.71	4.13 ^{ef} ± 0.00	78.50 ^g ± 0.42	0.51 (MST)
Water yam								
Native	5071.5 ^d ± 149.20	2776.5 ^{de} ± 14.85	2295.0 ^d ± 164.05	5003.0 ^d ± 130.11	2226.5 ^d ± 144.96	4.67 ^c ± 0.00	81.55 ^{def} ± 0.07	0.55 (MST)
Tempered	6213.5 ^e ± 16.26	5100.0 ^b ± 132.94	1113.5 ^f ± 116.67	9178.5 ^b ± 144.96	4078.5 ^b ± 12.02	5.04 ^b ± 0.05	83.13 ^c ± 0.04	0.82 (SST)
Acid hydrolysed	100.5 ^e ± 2.12	59.0 ^g ± 1.41	41.5 ^g ± 0.71	105.0 ^f ± 2.83	46.0 ^f ± 1.41	4.64 ^c ± 0.05	81.03 ^{ef} ± 1.38	0.59 (MST)
Citric substituted	36.0 ^e ± 0.00	16.5 ^g ± 0.71	19.5 ^g ± 0.71	24.5 ^f ± 0.71	8.0 ^f ± 0.00	4.30 ^{de} ± 0.05	78.80 ^g ± 0.99	0.46 (HST)
Yellow yam								
Native	6141.5 ^e ± 68.59	2525.0 ^e ± 12.73	3616.5 ^a ± 55.86	5719.0 ^c ± 1081.87	3194.0 ^c ± 1069.15	4.50 ^{cd} ± 0.14	77.15 ^h ± 0.57	0.41 (HST)
Tempered	5846.5 ^e ± 1245.22	3219.5 ^d ± 1016.11	2627.0 ^c ± 229.10	8807.0 ^b ± 520.43	5587.5 ^a ± 495.68	4.67 ^c ± 0.28	78.68 ^g ± 0.60	0.55 (MST)
Acid hydrolysed	167.0 ^e ± 1.41	54.5 ^g ± 0.71	112.5 ^g ± 0.71	96.5 ^f ± 2.12	42.0 ^f ± 1.41	4.00 ^f ± 0.00	80.68 ^{ef} ± 0.04	0.33 (HST)
Citric substituted	18.0 ^e ± 0.00	9.0 ^g ± 0.00	9.0 ^g ± 0.00	14.0 ^f ± 0.00	5.0 ^f ± 0.00	3.73 ^g ± 0.00	76.55 ^h ± 0.92	0.50 (MST)
Bitter yam								
Native	4796.0 ^d ± 190.92	1364.5 ^f ± 48.79	3431.5 ^b ± 142.13	2488.0 ^e ± 19.80	1123.5 ^e ± 28.99	4.50 ^{cd} ± 0.05	82.03 ^{cde} ± 0.53	0.28 (HST)
Tempered	5066.5 ^d ± 0.71	3870.0 ^e ± 12.73	1196.5 ^f ± 13.44	5202.5 ^{cd} ± 4.95	1332.5 ^e ± 17.68	6.13 ^a ± 0.00	85.68 ^b ± 0.04	0.76 (MST)
Acid hydrolysed	34.0 ^e ± 1.41	22.5 ^g ± 0.71	11.5 ^g ± 0.71	48.5 ^f ± 0.71	26.0 ^f ± 0.00	4.14 ^{ef} ± 0.09	80.55 ^f ± 0.49	0.66 (MST)
Citric substituted	78.5 ^e ± 2.12	35.5 ^g ± 2.12	43.0 ^g ± 0.00	71.0 ^f ± 2.83	35.5 ^f ± 0.71	4.30 ^{de} ± 0.05	80.75 ^{ef} ± 0.35	0.45 (HST)

HST highly shear thinning (BDR < 0.5); *MST* moderately shear thinning (BDR of 0.5–0.8); *SST* slightly shear thinning (BDR of 0.8–1.0)

Means in columns not followed by same superscript(s) are significantly different ($p < 0.05$)

In literature, 1cP = 0.0833RVU units

Fig. 1 Pasting profiles of **a** native, **b** tempered, **c** acid hydrolysed, and **d** low-citric-acid substituted starches of four yam cultivars

yam starch. Acid hydrolysed hybrid maize starch, corn starch, potato, rice, red bean and cassava starches showed improved gelling capacity over their unmodified counterparts (Lawal et al. 2005; Wang and Wang 2001; Osunsami et al. 1989; Kim and Ahn 1996). In our study, native and tempered starches developed soft viscous gels at lower concentrations (2–4% w/v), which became progressively hard or very hard as concentration increased (10–20% w/v), while acid hydrolysis and low-citric acid-substitution enhanced formation of hard gels at the different concentrations (2–20% w/v) in all cultivars. However, the bitter yam starches showed a different trend.

Starch paste clarity (SPC) of native and modified starches

Paste clarity of the starches varied significantly (7.27–50.67%) between native and modified starches. The SPC was significantly increased by acid hydrolysis and low-citric acid-substitution of the native starches. Increased clarity of acid-hydrolysed starches could be due to reduction of starch molecular weight and concentration (Miles et al. 1985). The introduction of citrate groups in the starch structure of citric acid substituted yam starches may be responsible for increased gel clarity. Improved paste clarity

could have resulted from addition of substituent groups such as acetyl- and hydroxypropyl- in potato starch (Kaur et al. 2004) by steric repulsion among the substituent groups. Reorganization of molecular components of starch granules and its closely-packed structure during tempering may have influenced the reduction in paste clarity. Among the native starches, differences in SPC could be attributed to genetic differences of the cultivars and peculiar properties of their starch granules as well as the amylose and amylopectin compositions. The small granule sizes of native and modified bitter yam starches (3.5–4.45 μm), as reported by Falade and Ayetigbo (2015), and their low amylose content, may have contributed to the lower clarity of their pastes compared to those of the other yam cultivars. Clarity of a starch paste would directly affected the sheen and appearance of products that contain starch as thickener.

Pasting properties of native and modified starches

Peak viscosity (PV) of the native starches varied significantly from 4796.0 to 8415.0 cP (Table 3). White yam starch showed the highest PV, and bitter yam starch the least. Similar effect of varietal differences on PV was

Table 4 Pearson's correlation between chemical and pasting properties of native and modified yam starches

	Ash	Moisture	Crude protein	NFE	Crude fat	pH	Amylose (%)
Peak viscosity	−0.060	0.858**	0.476	−0.873**	0.490	0.170	0.600*
Trough viscosity	−0.077	0.840**	0.297	−0.791**	0.313	0.065	0.535*
Breakdown viscosity	−0.009	0.605*	0.647**	−0.734**	0.651**	0.306	0.521*
Final viscosity	−0.130	0.930**	0.278	−0.854**	0.261	0.067	0.620*
Setback viscosity	−0.184	0.941**	0.223	−0.838**	0.165	0.063	0.661**
Peak time	0.096	0.512*	0.340	−0.551*	0.448	0.210	0.344
Pasting temperature	0.198	0.054	0.245	−0.146	0.302	0.520*	0.248

NFE nitrogen-free extract

* Significant correlation at 5% level

** Significant correlation at 1% level

Table 5 Pearson correlation between physicochemical and pasting properties of native and modified yam starches

	Starch paste clarity	Swelling power at 75 °C	Swelling power at 85 °C	Solubility at 75 °C	Solubility at 85 °C	Least gelation concentration
Peak viscosity	−0.612*	0.687**	0.834**	−0.754**	−0.0906**	0.691**
Trough viscosity	−0.564*	0.491	0.657**	−0.684**	−0.822**	0.590*
Breakdown viscosity	−0.497	0.820**	0.883**	−0.632**	−0.760**	0.647**
Final viscosity	−0.579*	0.653**	0.686**	−0.699**	−0.857**	0.703**
Setback viscosity	−0.534*	0.788**	0.645**	−0.640**	−0.805**	0.770**
Peak time	−0.623**	0.204	0.581*	−0.768**	−0.759**	0.232
Pasting temperature	−0.327	−0.211	0.280	−0.495	−0.388	−0.125

* Significant correlation at 5% level

** Significant correlation at 1% level

reported by Singh et al. (2006) for potatoes. Pasting patterns of the starches from the yam cultivars (Fig. 1) showed that tempered starches revealed significant higher PV than other modified starches. Tempering had variable effect on PV depending on cultivar, as tempering reduced PV of white yam starch, increased PV of water yam starch, and had no significant effect on PV of yellow and bitter yam starches. The low PV of acid hydrolysed (34.0–167.0 cP) and citric acid substituted starches (18.0–78.5 cP) could be due to weak pastes resulting from hydrolytic effects of the acid modifications on amorphous regions of starch. Abdorreza et al. (2012) and Lawal et al. (2005) reported similar trends for sago and maize starches respectively.

Cultivar differences may also be responsible for the significantly different breakdown viscosity (BV) between the native yam starches, with the starches from white yam showing the least susceptibility to breakdown. The applied modification methods significantly reduced the breakdown viscosity of the native starches. Reorientation of starch granules during tempering, and alteration of amorphous and crystalline regions during the acid modifications, may

have contributed to the reduction in BV. These agreed with findings for tempered African yam bean starch (Adebawale et al. 2009) and acid hydrolysed maize starch (Lawal et al. 2005). Generally, the tempered yam starches showed moderately- to slightly-shear thinning characteristics while the acid hydrolysed and citric acid modified starches indicated moderately- to highly-shear thinning behaviours as highlighted by the breakdown ratio (Table 3).

Final viscosity (FV) is viscosity attained after cooling cooked starch, and is an important quality attribute of starch-based foods. Native white yam starch had the highest FV (9231cP) compared to native starches of the other cultivars. Tempering significantly increased FV of the native starches, and this could be attributed to the amount of leached-out amylose that remained unchanged during pasting, leading to formation of a continuous gel matrix (Chung et al. 2000). However, acid hydrolysis and citric acid substitution significantly reduced FV, probably due to the considerable loss of amylose during pasting.

Setback viscosity (SV), a proxy for measuring susceptibility of the starches to retrogradation, differed

significantly with cultivar type and modification method. Tempering of the native yam starches significantly increased SV, and may thus make them more susceptible to retrogradation as the swollen solubilized starch, which were earlier leached out tend to re-associate, re-establishing a highly ordered network of increased intermolecular bonding between the molecules. Earlier work revealed that tempering increased SV of African yam bean starch (Adebowale et al. 2009). Acid hydrolysis and low-citric acid substitution significantly reduced SV of native yam starches. Acid hydrolysis could weaken intermolecular interactions between amylose molecules thus hindering realignment of the molecules. Introduction of functional groups substituting –OH on starches could cause electrostatic hindrance which restricted setback (Lawal et al. 2005) as shown in citric acid substituted starches.

Pasting temperature (PT) of native yam starches differed significantly (77.15–82.88 °C) with cultivars. Tempering increased the PT of the native starches probably due to increased crystallinity, as a result of starch granules reorientation. The strengthening of intra-granular bonded forces of tempered starch required more energy before structural disintegration and paste formation occurred (Eliasson 1980). Low-citric acid substitution significantly reduced PT, but acid hydrolysis had varied effect on PT depending on cultivar. Peak time of the starches significantly varied from 3.73 to 6.13 min. Peak time increased significantly with tempering, but reduced significantly with acid-hydrolysis and low-citric acid substitution of the starches.

Correlations between chemical and pasting properties of native and modified yam starches

Pasting properties of the starches depend considerably on their chemical composition among other factors. Moisture content of the starches had significant correlation with pasting properties. Ash of the starches did not have any significant correlation with all pasting (Table 4). This was expected as it is a non-starch component. Breakdown viscosity had a correlation with crude protein ($r = 0.647$, $p < 0.01$) and crude fat ($r = 0.651$, $p < 0.01$). There was high correlation between nitrogen free extract (which is deemed predominantly starch), and pasting viscosities. Amylose which plays an important role in pasting of starch positively correlated to all pasting viscosities ($p < 0.05$). This agrees with reports of studies from others. Nuwamanya et al. (2010) and Waramboi et al. (2011) found significant positive correlation between amylose and hot paste viscosity of cassava starches; and amylose and trough and final viscosities of sweetpotato flours respectively.

Correlations between physicochemical and pasting properties of native and modified yam starches

Pasting viscosities correlated significantly ($p < 0.01$) with least concentration for gelation, paste clarity, swelling power, and solubility at 75 and 85 °C (Table 5). This could be due to the fact that gelling and viscosity changes may require solubilization, swelling and gelatinization of starch components as influenced by factors such as pH, concentration and temperature. Paste clarity may depend on how completely melted, and dispersed starch crystallites are. In earlier studies, significant correlations were found between swelling power and peak viscosity; peak time, and swelling power and solubility (Nuwamanya et al. 2010), and swelling power and breakdown viscosity (Srichuwong et al. 2005) of tuber and root starches. High negative correlation was also reported between pasting viscosities and solubility of sweetpotato starches (Waramboi et al. 2011).

Conclusion

Native yam starches from yam cultivars showed different properties due to genetic variations. The chemical and physicochemical properties of the modified starches were generally different from the native starches. The modifications also reduced the starch constituents (amylose) and non-starch constituents (protein, fat) of the starches. Acid hydrolysis and low-citric acid-substitution resulted in appreciable improvement in pasting properties of the starches by reducing peak, trough and setback viscosities, peak time, and pasting temperature. The chemical and physicochemical properties of the starches correlated significantly with pasting properties. Tempered, acid hydrolysed and low-citric acid substituted yam starches showed promising properties that may effectively improve their industrial applications. In conclusion, relevant and suitable application of the tempered, acid hydrolysed and particularly, low-citric acid substituted yam starches may be developed with a view of using them in industrial production, or as additives for specific purposes in foods.

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