

FOOD SCIENCE & TECHNOLOGY | RESEARCH ARTICLE

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Cogent Food & Agriculture (2017), 3: 1306934







Received: 25 November 2016 Accepted: 12 March 2017 First Published: 20 March 2017

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Reviewing editor: Fatih Yildiz, Middle East Technical University, Turkey

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Comparative analyses of functional, pasting and morphological characteristics of native and modified tigernut starches with their blends

Olugbenga Olufemi Awolu^{1*}, Modupe E. Ojewumi², John Isa³, Deborah O. Ojo¹, Hellen I. Olofin¹ and Stella O. Jegede¹

Abstract: The effect of some physical and chemical treatments on the functional and pasting characteristics of native tigernut starch (TNNS), native sweet potato starch (SPNS) and blends of tigernut-sweet potato starch were studied. Native tigernut and sweet potato starches were subjected to physical (annealing and heat-moisture) and chemical (acetylation) modifications and compared to tigernut (T)-sweet potato (S) starches blends (T75:S25, T50:S50, T25:S75). Only heat-moisture treatment (THMT) significantly ($p \le 0.05$) increased water absorption capacity of the TNNS while only acetylation significantly ($p \le 0.05$) increased the oil absorption capacity of the native tigernut starch. The bulk density was significantly ($p \le 0.05$) reduced by annealing and acetylation. In addition, TNAS, mixture of blends and SPNS had higher swelling capacity than TNNS. The final and peak viscosities of TNNS, SPNS and all the starch blends were between (217-280 RVU) and (214-395.3 RVU) respectively with SPNS having the highest values, followed by T75:S25 (75% tigernut starch: 25% sweet potato starch) and TNNS in that order. TNNS also had the highest setback viscosity. Samples THMT, TANN and TNAS significantly ($p \le 0.05$) reduced the breakdown viscosity and the pasting temperature. The scanning electron microaraph showed that the native and modified starches of tigernut were similar to those of other starches. Overall, the results showed that many of the pasting characteristics of TNNS were comparable to SPNS, while SPNS had with better functional characteristics.

Subjects: Food Science & Technology; Food Analysis; Processing

Keywords: functional; modification; pasting characteristics; scanning electron micrograph; starch; sweet potato; tigernut

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PUBLIC INTEREST STATEMENT

Native starches have been found to have limited use industrially due to its inability to perform well when subjected to high temperature and stress. Modification has been suggested as appropriate mechanism to enhance utilization of native starches. Mixture of starches has been found to behave like either a chemically or physically modified starch. In this study, the functional, pasting and morphological properties were evaluated for native tigernut starch, physically and chemically modified tigernut starches and, mixture of tigernut and sweet potato starches.

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1. Introduction

Starch is a glucose polymer comprising amylose and amylopectin macromolecules (Obanni & Bemiller, 1997). It is widely available in nature and produced in all green plants. It has been extracted from several raw sources including tubers, cereals and legumes and utilized to influence food characteristics such as aesthetics, moisture, consistency, and shelf life (Adebowale, Sanni, & Fadahunsi, 2011).

When native starch granule is heated in the presence of water, it gelatinizes but later retrogrades during storage. This behaviour, in addition to its low shear stress resistance and thermal decomposition, high retrogradation and syneresis, makes native starches to have limited industrial application. Modification (chemical, physical and enzymatic) treatments and mixture of blends of starches of different sources have evolved to reduce, if not totally remove the negative attributes of native starches (Adebowale et al., 2011; Awolu & Olofinlae, 2016; Ortega-Ojeda & Eliasson, 2001).

Starch modifications have been used to alter the physicochemical properties of starches in order to meet various industrial demands (Eliasson & Gudmundson, 1996) and specifications. Modifications have been used to improve the water holding capacity and heat resistant behavior; reinforce starches binding ability, minimize syneresis of starch and improve thickening (Miyazaki, Maeda, & Morita, 2005). Blending different starches is gaining widespread acceptability and resulted in improved and desirable functional properties which help to mitigate some end-use limitations of native starches (Adebowale et al., 2011).

Sweet potato tubers (*Ipomoea batatas*) have high starch content. It is a dependable source of native and modified starches (Eke-Ejiofor, 2015). Tigernut (*Cyperus esculentus*) is an underutilized crop with a nutritious starch and rich carbohydrate contents (Bamigbola, Awolu, & Oluwalana, 2016; Temple, Ojobe, & Kapu, 1990). The high carbohydrate content of tigernut has promoted its used in composite flour formulation (Awolu, Omoba, Olawoye, & Dairo, 2017; Bamigbola et al., 2016).

This study is therefore aimed at investigating and comparing the effect of different modification treatments (heat-moisture treatment, annealing and acetylation) and mixture of tigernut and sweet potato native starches at different ratios on the functional and pasting characteristics of native tigernut starch, native sweet potato starch (SPNS), modified tigernut starches and tigernut/sweet potato starch blends.

2. Materials and method

2.1. Material

Sweet potatoes and tigernut tubers were purchased from the local Oba market in Akure, Ondo State, Nigeria. All chemicals were of analytical grades.

2.2. Extraction of starches

Tigernut and sweet potato native starches were extracted by the modified method of Umerie, Obi, and Okafor (1997). Tigernut and sweet potato tubers were washed and steeped in sodium metabisulphite solution (0.8133 g/L) at 30°C for 48 h. The steep water was changed thereafter and the tubers milled to slurry. The slurry was stirred and passed through a 100 mesh muslin sieve cloth to remove the shaft and the suspension obtained was allowed to stand for 24 h after which the supernatant was decanted and the starch sediment was collected and washed in distilled water. For tigernut starch, the starch milk was then centrifuged to obtain the pure starch.

2.3. Heat-moisture modification treatment (THMT)

The method of Franco, Ciacco, and Tavares (1995) was used. The moisture level of the starch samples (100 g) was raised to 30% by dispersing in distilled water. The slurry obtained was heated at 100°C for 16 h in an air oven, cooled immediately and air dried at 30°C for 72 h. The samples were then stored in sealed polyethylene bag at 4°C prior to use.

2.4. Modification by annealing (TANN)

Annealing of the starch was carried out according to the modified method of Jacobs, Eerlingen, Clauwert, and Delcour (1995). Starch suspension in distilled water (1:2 w/v) was incubated at 50°C for 24 h in a sealed container in a water bath after which the suspension was filtered through a Whatman number 1 filter paper and air-dried at 30°C for 72 h. It was then stored in sealed polyethylene bag at 4°C prior to use.

2.5. Modification by acetylation (TNAS)

The process was carried out according to the method of Sathe and Salunke (1981). Starch was dispersed in distilled water (1:5 w/v); it was stirred magnetically for 20 min. The pH of the slurry obtained was adjusted to 8.0 using 1 M NaOH. About 10.2 g acetic anhydride was added, while maintaining a pH range of 8.0–8.5 for 1 h. The pH of the slurry was again adjusted to 4.5 using 0.5 M HCl. It was filtered, washed four times with distilled water and air-dried at 28°C for 48 h.

2.6. Blending of starches

Native starches of tigernut and sweet potato were mixed together in the ratios of 25:75 (T25:S75), 50:50 (T50: S50) and 75:25 (T75:S75) respectively as an alternative to modification.

2.7. Determination of water absorption capacity (WAC)

This was determined by the method outlined by Diniz and Martin (1997) with some modifications. About 0.5 g of the sample was dissolved with 10 mL of distilled water in centrifuge tubes and vortexed for 30 s. The dispersions were allowed to stand at room temperature for 30 min, centrifuged at 3,000 rpm for 25 min. The supernatant was filtered with whatman Number 1 filter paper and the volume retrieved was accurately measured. The difference between initial volumes of distilled water added to the sample and the volume obtained after filtration was determined. The results were reported as mL of water absorbed per gram of sample.

Water absorption capacity (%) = $\frac{\text{Volume of water absorbed}}{\text{Weight of sample}} \times 100$

2.8. Determination of oil absorption capacity (OAC)

OAC was determined using the method of Adebowale, Adeyei, and Oshodi (2005). About 10 ml of oil of known specific gravity was added to 1 g of sample in a beaker. The suspension was stirred using a magnetic stirrer for 3 min. The suspension obtained was thereafter centrifuged at 3,500 rpm for 30 min and the supernatant was measured into a 10 ml graduated cylinder. The density of oil was 0.931 g/ml. The oil absorbed by the starch was calculated as the difference between the initial volume of the oil and the volume of the supernatant.

 $OAC = \frac{Volume of oil absorbed}{Weight of sample} \times D \times 100$

where D = Density of oil (0.931 g/ml).

2.9. Determination of bulk density (BD)

The method described by Oladele and Aina (2007) was used for the determination of bulk density. Fifty gram of starch sample was put into 100 ml measuring cylinder. The measuring cylinder was then tapped continuously until a constant volume was obtained. Bulk density (g/cm³) was calculated using the formula:

Bulk density $(g/ml) = \frac{\text{Weight of sample}}{\text{Volume of sample after tapping}}$

2.10. Determination of pH

This was determined by the method of Benesi (2005). Starch samples (10 g) were weighed in triplicate into a beaker, mixed with 100 ml of distilled water. The resulting suspension was stirred for 5 min and left to settle for 10 min. The pH of the water phase was measured using a calibrated pH meter.

2.11. Determination of swelling capacity (SC)

This was determined by the method described by Kulkarni, Kulkarni, and Ingle (1991) as modified by Akanbi, Nazamid, and Adebowale (2009). The starch (10 g) was suspended in 100 ml measuring cylinder and distilled water was added to reach a volume of 100 ml. The set up was stirred vigorously and allowed to settle for 3 h. The volume of settled particles was recorded and subtracted from 100.

Swelling capacity = 100 - V

where V = Volume of particle recorded.

2.12. Determination of pasting characteristics

Pasting properties of starch were evaluated using a Rapid Visco Analyzer (Newport Scientific, RVA Super 3, Switzerland). The starch suspension (6%, w/w) was held at 50°C for 1 min, and then heated to 95°C at 6°C/min. It was held at 95°C for 2.7 min before cooling from 95 to 50°C at the rate of 6°C/min and then held at 50°C for 2 min. A programmed heating and cooling cycle was used. The pasting curve obtained were analysed using a RVA Starch Master Software setup Tool (SMST) to obtain the characteristic parameters.

2.13. Morphology of starch granules

Scanning electron microscopy (SEM) was used for granule morphology studies. A thin layer of starch granules was mounted on an aluminum specimen holder by double-sided tape. The specimen holder was loaded in a polaron SC 7610 sputter coater (Fison Instrument, UK). It was coated with gold palladium, to a thickness of about 30 nm. The specimen holder was then transferred to a XL-20 series (Philips) scanning electron microscope and starch samples were examined at 10 kV.

2.14. Statistical analyses

Data obtained were analysed by single factor analyses of variance (ANOVA) using SPSS for Windows version 17.0. Confidence interval of sample means was reported at the 95% confidence probability. Comparisons of means were made using Duncan's test at 5% significance level (p < 0.05).

3. Results and discussion

3.1. Functional properties of native and modified starches

The results of the WAC, OAC and SC are shown in Figure 1a. The native tigernut starch (TNNS) had a high WAC (200%). Heat moisture treatment (THMT) increased the WAC of the TNNS significantly ($p \le 0.05$), while annealing decreased the WAC of TNNS. Chemical modification (TNAS) also reduced the WAC. The WAC of the mixture of tigernut and sweet potato starches does not differ significantly ($p \le 0.05$) from TNNS. The WAC of native sweet potato starch did not exhibit any significant ($p \le 0.05$) difference from TNNS. Acetylation had been shown to significantly ($p \le 0.05$) reduce WAC of water yam from 1.17 to 1.09 g/g (Awolu & Olofinlae, 2016). Oxidation and acetylation of jack bean starch had also been shown to significantly reduce the WAC (Yusuf, Ayedun, & Logunleko, 2007). In addition, mixture of cassava starch and sweet potato starch in the blends had been found to reduce water absorption index significantly ($p \le 0.05$) (Adebowale et al., 2011). A WAC of 164.7 and 93.0% had been recorded for 100% cassava starch and 100% durum wheat semolima (Oladunmoye, Aworh, Maziya-Dixon, Erukainure, & Elemo, 2014). Water absorption is important in products bulking and constituency; and helps in regulating dough characteristics (Eke-Ejiofor, 2015). Thus all the native and modified starched have acceptable WAC with heat-moisture modification enhancing the WAC.

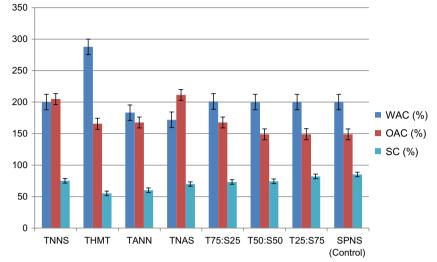
The OAC of the TNNS was significantly ($p \le 0.05$) reduced by physical modification (heat-moisture treatment and annealing), and significantly ($p \le 0.05$) increased by chemical modification (acetylation). Blends of starches had also been discovered to significantly ($p \le 0.05$) reduced the OAC. OAC values ranging from 1.0 to 2.5 g/g (100 to 250%) has been confirmed as a vital food product development property because of its ability to impart flavor and mouthfeel to foods (Omodamiro, Iwe, & Ukpabi, 2007). Acetylation significantly ($p \le 0.05$) increased OAC of TNNS, meaning that acetylation would be the best modification techniques where high OAC is the target of the starch product.

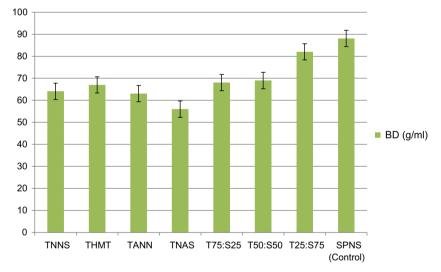
Figure 1a. WAC, OAC and SC of native and modified tigernut starches, tigernut/sweet potato blend starches and native sweet potato starch.

Key: TNNS = native tigernut starch, THMT = heat moisture treated tigernut starch, TANN = annealed tigernut starch, TNAS = acetylated tigernut starch, T75:S25 = 75% tigernut starch and 25% sweet potatoes, T50:S50 = 50% tigernut and 50% sweet potatoes native starch, T25:S75 = 25% tigernut starch and 25% sweet potatoes, SPNS = native sweet potatoes starch, WAC = Water absorption capacity; OAC = oil absorption capacity, and SC = swelling capacity.

Figure 1b. Bulk density of native and modified tigernut starches, tigernut/sweet potato blend starches and native sweet potato starch.

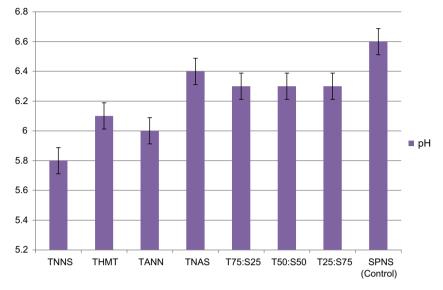
Key: TNNS = native tigernut starch, THMT = heat moisture treated tigernut starch, TANN = annealed tigernut starch, TNAS = acetylated tigernut starch, T75:S25 = 75% tigernut starch and 25% sweet potatoes, T50:S50 = 50% tigernut and 50% sweet potatoes native starch, T25:S75 = 25% tigernut starch and 25% sweet potatoes, SPNS = native sweet potatoes starch, and BD = bulk density.





The bulk density of the native, modified and mixture starches is shown in Figure 1b. Annealing and acetylation reduced the bulk density of the TNNS significantly ($p \le 0.05$) while heat moisture treatment and mixture of blends significantly ($p \le 0.05$) increased the bulk density. Bulk density values ranging from 0.64 to 0.97 g/ml were reported for cassava, sweet potato and cassava: sweet potato blend starches by Adebowale et al. (2011). The bulk density of starch is a reflection of the particle size (the degree of coarseness of the starch particles). Tigernut native starch and the acetylated starch with relatively low bulk density indicate that the particles were less coarse and could find application in pharmaceutical industries for tablets production. In addition, low bulk density starches could provide smooth texture that indicates fat absorption properties (Otegbayo, Oguniyan, & Akinwumi, 2013). Bulk density affects sensory acceptability of starch noodles Figure 1c. The pH of native and modified tigernut starches, tigernut/sweet potato blend starches and native sweet potato starch.

Key: TNNS = native tigernut starch, THMT = heat moisture treated tigernut starch, TANN = annealed tigernut starch, TNAS = acetylated tigernut starch, T75:S25 = 75% tigernut starch and 25% sweet potatoes, T50:S50 = 50% tigernut and 50% sweet potatoes native starch, T25:S75 = 25% tigernut starch and 25% sweet potatoes, and SPNS = Native sweet potatoes starch.



(Nwabueze, Glory, & Anoruoh, 2009), ease of packaging and transportation of powdery or particulate foods (Adebowale et al., 2011).

The pH of the native, modified and mixture starches (Figure 1c) ranged from 5.8 to 6.3. There were no significant ($p \le 0.05$) differences between the pH of the mixture components. In addition, modification significantly ($p \le 0.05$) increased the pH of TNNS. SPNS had the highest pH value, followed by the pH of the blends of starch (T75:S25, T50:S50, T25: S75). Tigernut native starch had the least pH value. Solubility and swelling power are pH dependent. Pronounced solubility and swelling power had been recorder at pH greater than or equal to 6 in native and modified jack bean starches (Yusuf et al., 2007), native and modified water yam starches (Awolu & Olofinlae, 2016), and commercial starch (Awolu & Olofinlae, 2016). In addition, low pH starches have commercial application as a thickener/emulsion stabilizer in cosmetic formulations.

All modification techniques and mixture of blends (with the exception of T25:S75) significantly ($p \le 0.05$) reduced the swelling capacity of tigernut native starch (Figure 1a). Awolu and Olofinlae (2016) also observed the same trend from the swelling power of native and modified water yam starches. Swelling power had been found to be as a result of the associated binding within starch granules which is a function of its amylose content. Low amylose content results in high swelling power. From the results, sweet potato native starch (SPNS) had the highest swelling capacity value while heat-moisture treated tigernut starch (THMT) had the least value. The low value for heat-moisture treated tigernut starch could be due to the effect of heat treatment disrupting the structure of the starch granules and reducing the swelling capacity of its native starch.

3.2. Pasting properties of native and modified starches

The pasting properties of starch has a high influence on aesthetic considerations and quality in the food industry as they affect the texture, digestibility and end use of starch based food commodities. The results of pasting characteristics of the samples are shown in Table 1. The final viscosity of TNNS was 240.4 RVU. Modifications (THMT, TANN and TNAS) significantly (≤ 0.05) reduced the final viscosity. Tigernut and sweet potato blend (T75:S25) however had final viscosity that was significantly (≤ 0.05) higher than TNNS. Incorporation of sweet potato starch (final viscosity of 280.4 RVU) accounted for the high final viscosity of T75:S25. As the potato starch contents in the blends reduced, the final viscosity of the blends also reduced (T50:S50; T25:S75). This same trend (reduction in final viscosity in blends compared to native starch) was also reported by Adebowale et al. (2011) when

Sample	Final viscosity (RVU)	Peak viscosity (RVU)	Setback (RVU)	Breakdown (RVU)	Peak temperature (°C)	Peak time (min)
TNNS	240.4	270.5	85.3	115.3	82.4	4.9
THMT	149.2	85.5	2.2	0.3	0.0	6.3
TANN	164.2	164.2	50.0	18.0	95.3	6.3
TNAS	188.3	131.3	81.4	24.5	79.8	5.9
T75:S25	256.9	325.3	74.1	142.4	82.4	4.7
T50:S50	217.2	214.3	86.2	83.3	79.8	5.0
T25:S75	236.7	260.6	80.9	104.8	82.4	4.9
SPNS (Control)	280.4	395.3	60.3	186.3	82.4	4.5

Key: TNNS = native tigernut starch, THMT = heat moisture treated tigernut starch, TANN = annealed tigernut starch, TNAS = acetylated tigernut starch, T75:S25 = 75% tigernut starch and 25% sweet potatoes, T50:S50 = 50% tigernut and 50% sweet potatoes native starch, T25:S75 = 25% tigernut starch and 25% sweet potatoes, and SPNS = native sweet potatoes starch.

the mixture of cassava starch and sweet potato showed significant (≤ 0.05) reduction in the final viscosity of native cassava starch. Final viscosity is an indication of the starch to form viscous paste. It indicates the stability of the cooked paste in actual use. Awolu and Olofinlae (2016) showed that modification using acetylation and oxidation increased final viscosity of native water yam starch while acid-thinning reduced the final viscosity. In addition, a high final viscosity of starch indicates that the paste is more resistant to mechanical shear and may easily form a more rigid gel (Zhang, Gu, Hong, Li, & Cheng, 2011). The high final viscosity values of sweet potato starch could be attributed to its higher amylose content (Miles, Morris, Orford, & Ring, 1985) which is more than amylose content of tigernut starch. A high final viscosity is desirable in many food products (soups, sauces and dressings); they can be utilized in wet stage production of paper and the textile industry where high viscosity is required (Moorthy, 2002).

TNNS had peak viscosity value of 270.5 RVU. As previously observed with final viscosity, sweet potato starch had the highest peak viscosity. In addition, modifications significantly (≤ 0.05) reduced the peak viscosity. Peak viscosity, which is the maximum viscosity attained during or soon after cooking is an indication of the water binding capacity of the starch (Adebowale et al., 2011). It indicates the strength of the pastes which are formed from gelatinization during food processing. It has been found to correlate with final product quality (Maziya-Dixon, Dixon, & Adebowale, 2007), while higher peak viscosity has been found to correlate to a higher thickening power of the starch (Swinkels, 1985).

The setback value of SPNS was significantly (≤ 0.05) lower than that of the native tigernut starch. The higher the setback value the lower the rate of synresis and weeping (Maziya-Dixon et al., 2007). Modifications (THMT, TANN, TNAS) and starch mixtures (except T50:S50) significantly (≤ 0.05) lowered the setback value of the native starch. Starch blend (T50:S50) had the highest setback value which is an indication of lower retrogradation value.

The breakdown viscosity of the TNNS was reduced by modifications and blend mixture except in T75:S25 blend. Starch with lower breakdown viscosity has higher capacity to withstand heating and shearing during cooking. In this instant, modification greatly and significantly (≤ 0.05) reduced breakdown viscosity of TNNS. Native starches have low capacities to withstand heating and shearing. This is one of the benefits of modifying starches. Modification enhances the ability of starches to withstand heating and shearing. The high thermal stability of the heat-moisture treated starch

could be utilized in canned foods and those products that require sterilization (Novelo-Cen & Betancur-Ancona, 2005).

There were no significant (≤0.05) differences in the pasting temperature of TNNS, SPNS, T75:S25 and T25:S75. Starch mixture (T50:S50) however had a reduced pasting temperature. THMT had the least pasting temperature. Pasting temperature is the temperature at which viscosity starts to rise (Swinkels, 1985). It ensures swelling, gelatinization and gel formation during processing (Eke-Ejiofor, 2015). Higher pasting temperature indicates lower swelling capacity. High pasting temperature could be an advantage in canned and sterilized foods which require the use of high temperatures (Otegbayo et al., 2013).

Peak time is the measure of the cooking time (Adebowale et al., 2005). It is the time taken for the starch to reach highest viscosity. Peak time of the starch samples ranged from 4.5 to 6.3 min. Sweet potato native starch and tigernut native starch had pasting time of 4.5 and 4.9 min respectively. Blending of the starches, heat-moisture treatment, annealing, and acetylation treatments increased pasting temperature of the starch samples.

3.3. Morphological characteristics of native, modified and mixed starches

The scanning electron micrograph of the native and modified starches are shown in Figures 2a–2f. Majority of the granules occurred singly; with few clusters. Clusters were observed in heat-moisture modified (THMT) starch granules (Figure 2b) and acetylated starch (TNAS) granules (Figure 2d). These clusters might be as a result of severe temperature and acidic concentration of THMT and TNAS respectively. Residual protein and drying conditions have been reported as possible causes of clusters of starch granules (Ashogbon & Akintayo, 2012). The TNNS granule (Figure 2a) had oval, smooth-surface granules with varying sizes. Oval and round shaped granules were reported by Lawal and Adebowale (2005) for native jack bean starch. Modifications changed the shapes from

Figure 2a. Scanning electron microscopy of TNNS sample.

Key: TNNS = native tigernut starch.

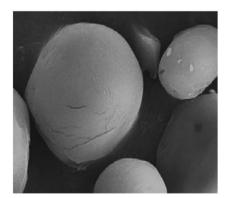


Figure 2b. Scanning electron microscopy of THMT sample.

Key: THMT = heat moisture treated tigernut starch.

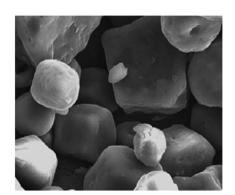


Figure 2c. Scanning electron microscopy of TANN sample.

Key: TANN = annealed tigernut starch.

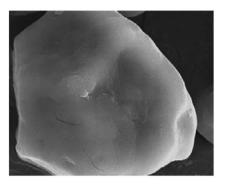


Figure 2d. Scanning electron microscopy of TNAS sample.

Key: TNAS = acetylated tigernut starch.

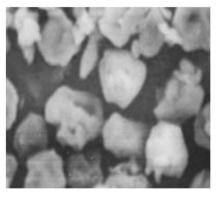


Figure 2e. Scanning electron microscopy of T75:S25 sample.

Key: T75:S25 = 75% tigernut starch and 25% sweet potatoes

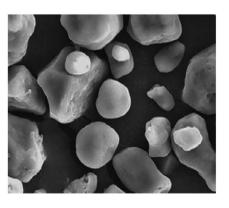
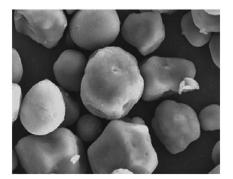


Figure 2f. Scanning electron microscopy of T50:S50 sample.

Key: T50:S50 = 50% tigernut and 50% sweet potatoes native starch.



oval to polygon shapes; hexagonal and pentagonal shapes being prominent. Sirivongpaisal (2008) also reported smaller sizes for round shaped granules; which was also observed in this work (Figures 2b–2f). Highest deformation of granules structure was observed in acetylated starch. Mixture of structures (Figures 2e and 2f) also altered the native starch granules shapes.

4. Conclusions

The WAC, pH and swelling capacity of TNNS compares favourably with SPNS while the OAC of the TNNS was however better than that of the native sweet potato starch. For modified starches, heat moisture treatment significantly ($p \le 0.05$) increased the WAC. All modifications and mixture also compared favourably with native sweet potato starch in terms of functional properties. Modification reduced the breakdown viscosity and pasting temperature. Low breakdown viscosity and pasting temperature are required for quality starches. The granule shape of the TNNS was oval, while modifications altered the granule structures. It was found that mixture of starches also altered the native starch granule shapes. In general, all the samples had good and acceptable pasting characteristics.

Funding

The authors received no direct funding for this research.

Competing Interests The authors declare no competing interest.

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Citation information

Cite this article as: Comparative analyses of functional, pasting and morphological characteristics of native and modified tigernut starches with their blends, Olugbenga Olufemi Awolu, Modupe E. Ojewumi, John Isa, Deborah O. Ojo, Hellen I. Olofin & Stella O. Jegede, *Cogent Food & Agriculture* (2017), 3: 1306934.

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