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MODIFICATION OF BITTER YAM (*Dioscorea dumetorum*) STARCH BY OCTENYL SUCCINIC ANHYDRIDE AND MICROWAVE IRRADIATION TREATMENT: PREPARATION, MORPHOLOGY, STRUCTURE, AND FUNCTIONALITY

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ABSTRACT

Starch was extracted from bitter yam (*Dioscorea dumetorum*) and subsequently modified through esterification (succinylation) and microwave treatment. The functional properties of the starches were then examined. Proximate composition analysis showed that, after modification, the succinylated (OSABS) and microwave-treated (MTBS) starches exhibited lower moisture content but higher levels of protein, ash, crude fiber, and fat compared to the untreated bitter yam starch (UBS). Both modifications substantially improved the swelling power and solubility profile of OSABS and MTBS at 80 °C. SEM micrographs revealed no surface damage for the OSABS sample, but showed major granule damage for the MTBS, with the granules appearing shriveled and collapsed. The starches were further characterized using FTIR, TGA, XRD, and light spectroscopy. Gelation studies indicated that the UBS sample is a superior gelling agent compared to the modified derivatives. All forms of modification raised the pasting temperature but reduced the peak, trough, breakdown, setback, and final viscosities. Additionally, the modified starches demonstrated excellent paste clarity and pasting properties, making them suitable for specific applications in the industries. Overall, modifying bitter yam starch resulted in changes to its properties, thus, using octenyl succinic anhydride and microwave treatment may be a superior alternative to conventional modification methods.

Keywords: Bitter yam starch, Microwave treatment, Octenyl succinic anhydride, Physicochemical properties, Structure characteristic.

INTRODUCTION

The Dioscorea family includes the bitter yam *(Dioscorea dumetorum)*. Although the bitter yam is a rich source of starch and phyto-proteins, it remains one of the many tropical tubers that are underutilized and at risk of extinction. This tuber is commonly referred to as African bitter yam. It is also known as cluster yam, wild yellow yam and three-leaved yam (Egbuonu *et al*., 2014; Oke *et al.,* 2020; Nwosu-Obieogu *et al.,* 2022). Dried tubers of bitter yam have found usefulness in breweries, serving as a base in beer production. Like most yams, bitter yam tubers can be processed into instant flour for baking, yam flakes and starch for various pharmaceutical applications (Nwosu-Obieogu *et al.,* 2022).

The diverse domestic and industrial uses of starch make this biopolymer a crucial biomaterial for economic progress, especially in developing countries worldwide. Due to its soil adaptability, relative abundance, biodegradable properties and distinct physiochemical characteristics, starch is extensively used in food processing (Zhang *et al*., 2017a; Liu *et al*., 2014; Zhang *et al*., 2017b). While starch has long been a staple food ingredient, it has also demonstrated versatility across various industries, including pharmaceuticals, textiles, cosmetics, paper, plastics, and adhesives. It serves as a viscosity enhancer, stabilizer, thickener and bulking agent, among other applications (Bergthaller, 2005; Hemamalini and Dev 2018). Nevertheless, natural starches have several limitations, including strong hydrophilicity, weak mechanical strength, high solubility in water, low heat stability, poor compatibility with hydrophobic synthetic polymers and limited processability. These limitations restrict their use in various applications (Azwar and Hakkarainen 2012; Priya *et al.,* 2014). In addition, native starch possesses a complex semi-crystalline structure that necessitates energy for its gelatinization (Jane 1992). To address these drawbacks, starch can be modified using various methods, including physical, chemical, or enzymatic approaches. Alteration of the granular structure of starch, as well as its transformation into cold water-soluble variant have been achieved by chemical or physical modification of native starch (Jane 1992). Chemical methods of modification such as esterification, etherification, oxidation, acid hydrolysis and crosslinking have been reported as being very effective in enhancing starch properties (Santacruz *et al.*, 2002). Modification by octenyl succinic anhydride (OSA) has been extensively studied over the past six decades. The standard process involves esterification of starch with OSA in a mild alkaline solution (scheme 1). With this method, OSA becomes substituted onto the starch and, in the process, imparts hydrophobicity to the starch granules (Timgren *et al.,* 2011; Rayner *et al.*, 2012; Sweedman *et al.*, 2013; Bello-Pérez *et al.,* 2015; Simsek *et al.,* 2015; Lv *et al.*, 2018).

Physical modifications which include multiple cycles of deep freezing, thermally inhibited treatment and thawing, superheated methods, osmotic pressure treatment, iterated syneresis, pulsed electric fields (PEF) treatment, corona electrical discharges and microwave treatment have garnered significant attention (Nemtanu and Minea, 2006; Maache-Rezzoug *et al.*, 2009; Han *et al.,* 2009; Babu *et al.*, 2018; Awokoya *et al.*, 2020). Among these, microwave treatment (MT) is notable, being very effective in modifying native starch for enhanced functional properties and broader potential applications (Awokoya *et al.,* 2020). Microwaves are electromagnetic waves within the frequency range of 300–300,000 MHz. Microwave energy is absorbed by polar molecules in the starch, causing them to align with the electric field. This results in rapid reorientation and the generation of heat by molecular friction (Sumnu, 2001). The MT ensures bulk heating and a faster heating rate relative to other conventional heating methods – achieving up to 50–70% efficiency, as against just 10% efficiency associated with conventional ovens (Mermelstein, 1997).

This report focuses on the effects of esterification, as well as microwave treatment on bitter yam starch. While earlier studies on esterification and microwave treatment have focused on starches extracted from maize, potato, rice, wheat, cassava, sorghum, canna and water chestnut: widely available sources; to the best of our knowledge, no research has been conducted to explore starch extracted from bitter yam, an unconventional starch source. This study, therefore, aimed at isolating and modifying bitter yam starch through esterification and microwave treatment, and evaluating the impact of these methods of modification on its properties.

MATERIALS AND METHODS

Materials

Bitter yam tubers (*Dioscorea dumetorum*) were sourced from Akinola food market located in a suburb of Ile-Ife in Osun State, Southwestern region of Nigeria. The esterifying agent, octenyl succinic anhydride (molecular formula $C_{12}H_{18}O_3$ was obtained from Sigma-Aldrich (Steinheim, Germany). All reagents utilized in the study were of analytical grade.

Starch isolation and purification

Bitter yam tubers were first washed in a bowl containing water, after which those that were free from any signs of spoilage were selected for use. Starch isolation was performed as described by Awokoya *et al.* (2020), although with slight modifications. A total of 38.6 kg of bitter yam tubers were hand-peeled and washed with water. Upon grating, distilled water (9 L) was mixed with the resulting pulp and then left for about 6 h to allow the separation of starch. Starch was isolated from yam fiber by filtering the suspension through a muslin cloth. To ensure a complete starch separation, the fiber residue was thoroughly rinsed and re-filtered. The supernatant (starch in solution) was left undisturbed for about 14 h to allow sedimentation. The clear liquid was decanted. To eliminate the proteins and fibers content, the isolated starch was repeatedly washed three times, with about 9 L distilled water (each time). Further purification of the starch was performed by re-suspending the starch in distilled water, decanting, and allowing it to sediment for another 14 h. The final starch product was air-dried for approximately 48 h at around 27 °C and stored in an airtight plastic bag until use. Figure 1 illustrates the stepwise isolation of the starch.

Preparation of OSABS (starch octenyl succinate)

Octenyl succinate modification of the prepared starch (Scheme 1) was performed as outlined by Sathe and Salunkhe (1981) with slight adjustments. Initially, 100 g of starch was weighed into a conical flask containing 300 mL distilled water. The flask was placed on a magnetic stirrer and stirred at 200 rpm for 1 h at room temperature. The resulting slurry was adjusted to pH 9.0 with NaOH (1 M). Starch octenyl succinate (designated as OSABS) was produced by adding 5 g octenyl succinic anhydride (OSA) to the slurry. The pH was maintained within 8.0 – 9.0 range, and the reaction was allowed to complete for 2 h. The pH of the slurry was then lowered to around pH 4.0 using HCl (0.5 M). The OSABS produced was filtered out of solution, washed 6 times with distilled water and air-dried for about 36 h.

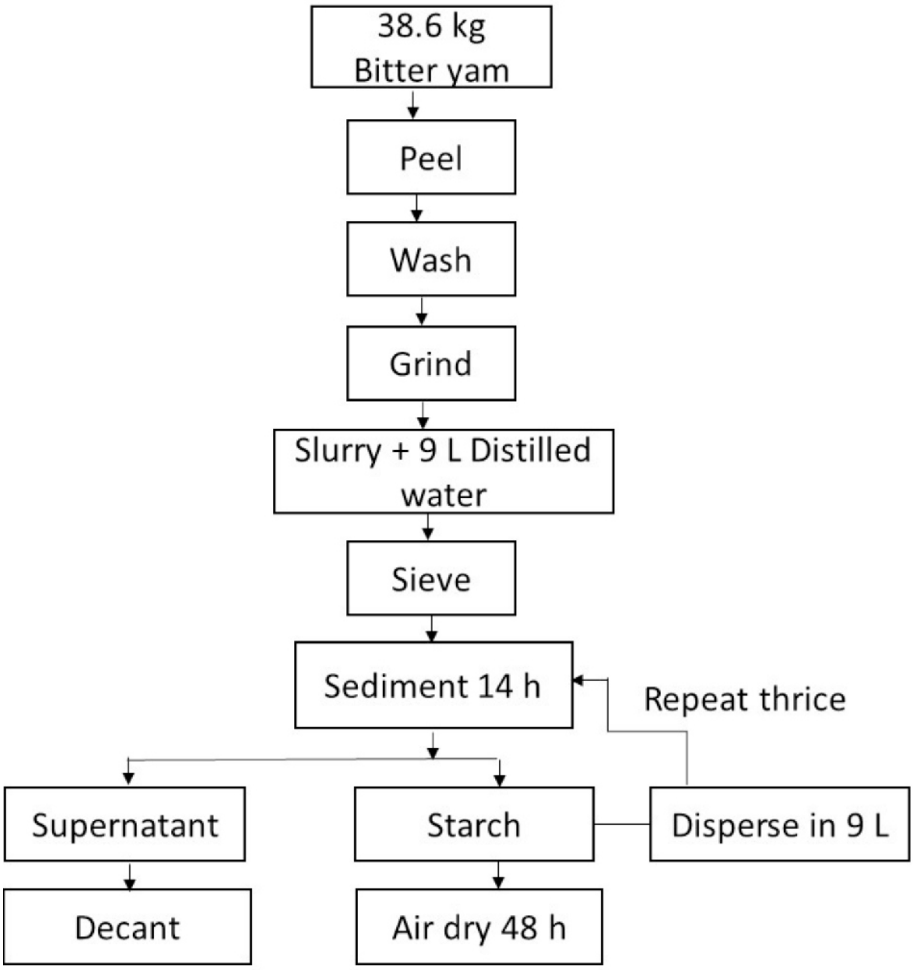
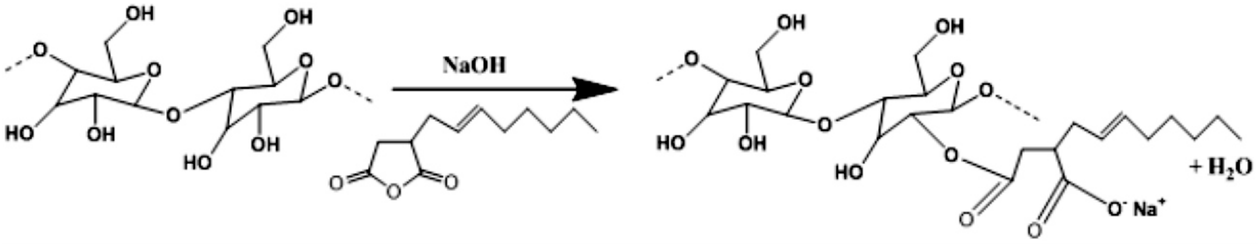


Figure 1. Schematic diagram for extraction of bitter yam starch.



Scheme 1: Synthesis of Starch Octenyl Succinate.

Instrumentation

Characterization of the starch samples was undertaken by the following equipment: Fourier transformed infrared spectrometer (FTIR-Nicolet 330 spectrometer, Thermo Fisher Scientific, USA) within the range of 4000 - 400 cm^{-1}, to identify the functional groups; scanning electron microscope (SEM-EDX, Phenom ProX, Thermo Fisher Scientific, USA), to analyze the morphology and surface texture; standard optical microscope (OMAX trinocular light microscope A35140U, China); thermogravimetric and differential thermal analyzers (TGA/DTA) (PerkinElmer TGA-4000 analyzer, Waltham, MA, USA) and; X-ray diffractometer (XRD) (Rigaku MiniFlex-600 model), with diffractograms captured over a 2θ range of 2° to 70°.

Preparation of microwave-treated bitter yam starch

The microwave treatment method was largely adopted from Ma *et al.* (2015). Approximately 150 g bitter yam starch was dispersed in 500 mL distilled water in a three-necked round bottom flask (1000 mL capacity) and then refluxed. This was followed by heating, at 70 °C for 1 h, in a microwave synthesizer (MAS-II mono-made, Shanghai, China). Energy level was set at 400 W. After 1 hour, the sample was rapidly cooled and left in an ice bath for about 20 min. The suspension was filtered and air-dried for 36 h and afterwards stored in a sealed glass ware for further use. The ensuing product was labelled microwave-treated bitter-yam starch (MTBS). Untreated bitter yam starch (UBS) served as the control.

Physicochemical and functional properties of US, OSABS and MTBS samples

Proximate composition

The standard methods adopted by the Association of Official Analytical Chemists (AOAC, 1996) were used to estimate the proximate composition of the starch samples. The % carbohydrate composition was quantified by subtracting the sum of the percentages of all other contents (such as moisture, crude fat, ash and crude protein) from 100.

Gelation studies

The least gelation concentration (LGC) of the starch was determined as described by Coffman and Garcia [1977]. Different quantities of the starch were placed in different test tubes containing 5 mL distilled water. The test tubes were thoroughly mixed for 10 min using a Whirlmix mixer (Cenco Instrumenten, Breda, The Netherlands). This gave starch concentrations ranging from 2 to 18% (w/v). The test tubes were arranged and heated at 80°C for 30 min in a water bath. Afterwards, the samples were rapidly cooled and left for 2 h in ice-water bath. The LGC was identified as the lowest concentration at which the starch sample remained stable without falling or slipping upon the inversion of the test tube containing it.

Light transmittance measurement

In accordance with the technique described by Rafiq *et al.* (2016), a slurry containing 1% (w/v) starch in distilled water was heated (with intermittent shaking) for 30 min in a boiling water bath. The test tube was subsequently allowed to cool for 1 h, at room temperature. Measurement of the transmittance (at λmax 620 nm) of the paste was performed with a UV-vis spectrophotometer (UV-1800 Shimadzu, Kyoto, Japan).

Determination of swelling power and solubility

Determination of the swelling power and solubility of the starch samples were performed in accordance with the methods of Leach *et al.* (1959). Starch sample (0.1 g) was weighed into different dry test tubes. The weight of each test tube + sample was taken as W_1. Distilled water (10 mL) was measured into each test tube. This was followed by a 30 s vigorous mixing on a Whirlmix mixer. The resulting slurries were heated for 30 min, at temperatures ranging from 40 – 80 °C, in a temperature-regulated water bath (GFL, Burgwedel, Germany). At its experimental temperature, each sample was withdrawn, allowed to cool at room temperature, and then centrifuged for 15 min at 6000 rpm. The final weight was determined and designated, W_2.

$$Swelling\ of\ starch = \frac{W_2 - W_1}{Weight\ of\ starch\ (g)} \qquad 1$$

Upon centrifugation, an aliquot (5 mL) of the supernatant was dried to a constant weight, at 110 °C, in a Gester GT-D10 drying oven (Fujian, China). The residue recovered from the dried 5 mL aliquot is a measure of the quantity of starch solubilized in water. The solubility of the starch was expressed in grams per 100 grams of starch on a dry weight basis.

Determination of pasting properties

The pasting profile of the starch samples was analyzed using a Rapid Visco Analyser Super 4 model (RVA, Perten Instruments, Kurva, Sweden). The moisture content of the sample was first estimated for accurate determination of sample weight and the required quantity of water needed. A portion of the starch was added to 25 mL deionized water in an RVA aluminum test canister. A standard procedure which involved stirring, heating- (from 50 to 95 °C), and cooling was applied. The total analysis duration was 12.5 min. The resulting data were plotted as paste viscosity in centipoise (cP) versus time.

Statistical analysis

SPSS was used in the statistical analyses. The results were reported as mean values and standard deviations. Additionally, analysis of variance (ANOVA) was conducted by means of a statistical software suite written in the R programming language (R Core Team). The significantly different means detected by the ANOVA was identified by Duncan's multiple range test, with a significance level set at 5% ($p < 0.05$).

RESULTS AND DISCUSSION

Characterization of UBS, OSABS and MTBS samples (SEM/SOM, TGA/DTA, XRD and FTIR)

Scanning electron micrographs and light micrographs of the starch samples are shown in Figures 2A and 2B. The granules of the OSABS were microscopically very similar to the UBS sample. The starch granules of both UBS and OSABS were spherical to ovoid and uniformly sized, indicating that the appearance of the starch was not significantly altered by the esterification reaction. Nevertheless, surface damage to the granules was observed in the MTBS-modified sample, showing severe deformation that resembled wringing and collapse of the starch granules. According to Lewicka *et al.* (2015), this deformation could be attributed to the temperature effects during microwave irradiation. In the observed light micrographs (Figure 2B), the granules of both UBS and OSABS appeared somewhat oval with minimal differences in their granular appearance. In contrast, the granules of MTBS showed more drastic changes, appearing shriveled and collapsed. These results are consistent with the scanning electron microscopy data.

The TGA and DTA curves depicting the thermal degradation process of UBS, OSABS, and MTBS starches are illustrated in Figures 3A and 3B, respectively. Thermogravimetric analysis is a crucial technique that offers valuable insights into the thermal stability of starches, which is essential for their industrial applications. Based on the TGA profiles (Figure 3A), the degradation processes for the UBS, OSABS, and MTBS samples occur in three, three, and two main steps, respectively. The initial weight loss for all three samples took place between 25 and 125 °C, corresponding to the initial vaporization of water (both free and bound). (Khawas and Deka, 2017). The second step of weight loss occurred around 390 and 370 °C for both the UBS and OSABS samples, sequentially, while for the MTBS sample, it was around 350 °C. These steps correspond to the primary decomposition of starches, that is depolymerization of starch chains (Jumaidin *et al.*, 2017). Additionally, along with the micro-organic components, this stage indicates the thermal degradation of the amylose and amylopectin parts of the starch (Pineda-gomez *et al.,* 2021). The third stage of weight loss in the UBS and OSABS samples commenced from around 400 °C. Overall, it was observed that the thermal stability of UBS was higher than those of the OSABS and MTBS. Figure 3B displayed the DTA profiles of the starches, clearly indicating endothermic patterns for all samples. One major endothermic effect was observed individually for MTBS, OSABS, and UBS at approximately 350, 375, and 390 °C, respectively. It is noteworthy that UBS and OSABS exhibit very similar thermal characteristics. The endothermic peaks observed in the DTA profiles at temperatures above 300 °C may be linked to the breaking of bonds within the structure of the starch samples (Kaczmarska, 2019).

The FTIR spectra of the UBS, OSABS and MTBS samples are shown in Figure 4A. The prominent absorption band between 3379-3414 cm^{-1} in all the samples corresponds to the –OH group vibration frequency of glucose residues. The degree of formation of inter- and intra-molecular hydrogen bonds is indicated by the width of this peak. Additional vibrational peaks at 1022, 1080, and 1157 cm^{-1} were attributed to the angular deformation of the C-C and C-O bonds, together with the skeletal vibration of the α-(1→ 4) glycosidic linkage (Qiu *et al.,* 2015; Wang *et al*., 2010). Generally, the infrared spectra showed no significant changes following the modifications. This suggests that the esterification reaction and microwave treatment merely altered the starch chain but did not damage its underlying chemical structure. The only observed change was a shift in the absorption band from 3379 to 3414 cm^{-1} after the microwave treatment.

The crystalline structures of bitter yam starches were investigated using XRD. As shown in the diffractograms (Figure 4B), the untreated bitter yam starch (UBS) exhibited prominent diffraction peaks at 18.28 and 23.52^{0}, characteristic of a B-type starch structure. Following succinylation (OSABS), the intensity of these peaks remains intact. After microwave treatment (MTBS), the two peaks gradually vanished and a new peak appeared around 20.34^{0}, suggesting that the microwave process disrupted the crystalline regions of the UBS (native) starch.

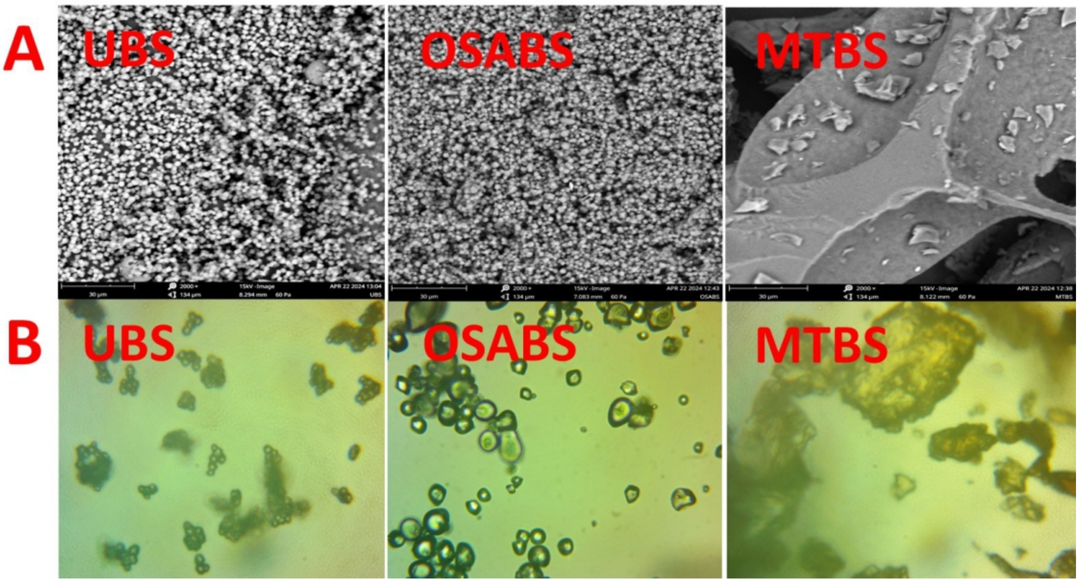


Figure 2: Scanning electron microscopy (SEM) images **(A)** and standard optical light micrographs **(B)** of UBS, OSABS and MTBS starch samples.

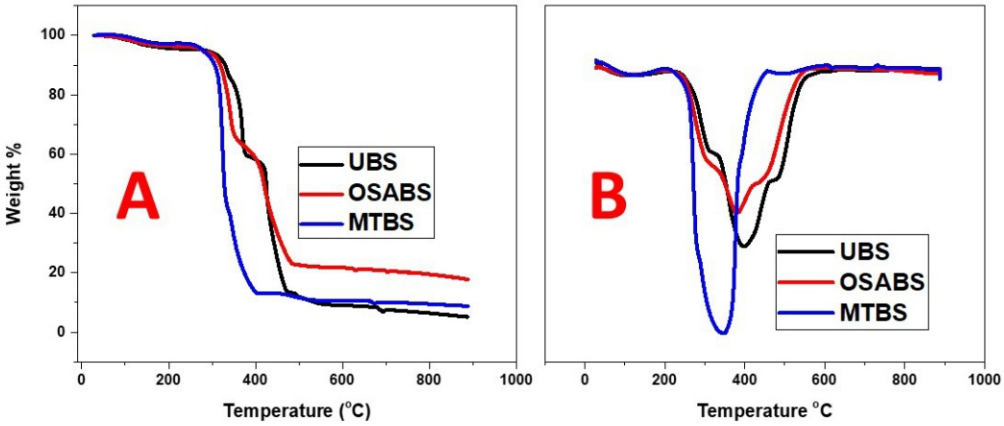


Figure 3: **(A)** Thermogravimetric analysis curves and **(B)** Differential thermal analysis profiles of UBS, OSABS and MTBS starch samples

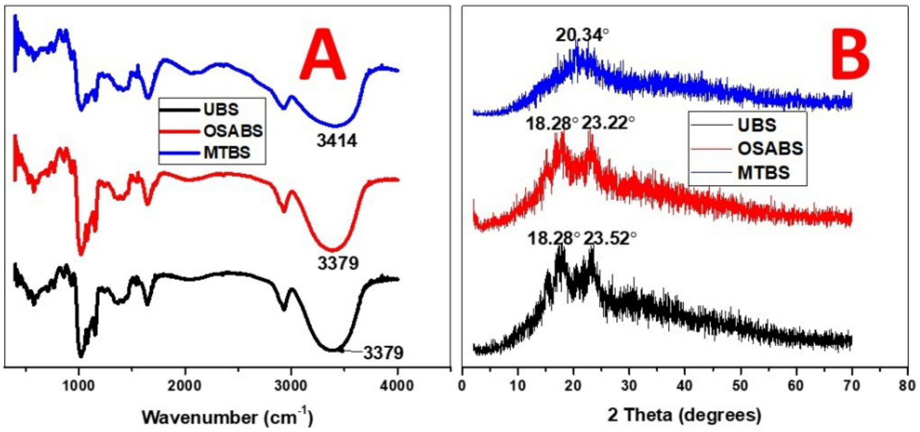


Figure 4: (A) FTIR spectra and **(B)** X-ray diffraction patterns of UBS, OSABS and MTBS starch samples

Proximate composition of UBS, OSABS and MTBS samples

Table 1 presents the proximate composition of the starches. The moisture content of the UBS sample was 12.08%, while it ranged from 11.02 to 11.11% for the modified starches. The observed reductions in moisture content after modifications could be attributed to the substituted hydroxyl groups in OSABS. For the MTBS, it might most likely be due to the evaporation of water during drying upon microwave treatment. The moisture contents in both the untreated and modified starches were within a desirable range and are very important for the prolongation of the shelf life of the starch. This range of moisture content is analogous to the one reported by Olayinka *et al.* (2013). Starches having moisture content of below 14% are most likely able to resist microbial growth, which in turns leads to the extension of their shelf lives (Hayma, 1995). The native starch sample had a carbohydrate content of 85.17%. Both physical and chemical modifications significantly reduced ($p<0.05$) the carbohydrate content from 85.17 to 82.43% for OSBAS and 84.09% for MTBS. These results suggest that all the starch samples are rich in carbohydrates and can serve as important energy sources when included in diets. The protein, fat, fiber, and ash contents of all the samples increased after modifications. The purity of the starches was indicated by the lower percentages of fat, protein, and ash in the starch samples (Adebowale *et al.*, 2005). The low non-starch components (ash, protein and fat) of the starch make it valuable for certain industrial applications. For example, the low protein content of bitter yam starch could be advantageous in its application in the pharmaceutical industries, in bakery and in the production of yogurts (Onuegbu *et al.*, 2011).

Table 1: Proximate composition of UBS, OSABS and MTBS derivatives of *Dioscorea dumetorum.*

Starch	Protein	Fat	Fibre	Ash	Moisture	Dry matter	Carbohydr.
UBS	1.13±0.04^{a}	0.61±0.06^{a}	0.75±0.04^{a}	0.26±0.04^{a}	12.08±0.04^{a}	87.92±0.06^{a}	85.17±0.01^{a}
OSABS	2.08±0.01^{b}	1.17±0.09^{b}	1.92±0.08^{b}	1.29±0.04^{b}	11.11±0.05^{b}	88.89±0.07^{b}	82.43±0.05^{a}
MTBS	1.17±0.01^{b}	1.04±0.08^{a}	1.42±0.02^{b}	1.26±0.01^{b}	11.02±0.01^{c}	88.98±0.04^{b}	84.09±0.04^{a}

All values represent the means of triplicate determinations ± standard deviation. Means within columns marked with different superscripts (a, b, c) are significantly different according to LSD ($P < 0.05$). Abbreviations: UBS – Untreated bitter yam starch; MTBS – Microwave treated bitter yam starch; OSABS – Octenyl succinic anhydride bitter yam starch; Carbohydr – Carbohydrate.

Gelation properties of UBS, OSABS and MTBS samples

The gelation properties of untreated, octenyl succinylated, and microwave-treated bitter yam starches are shown in Table 2. The least gelation concentration (LGC) reflects the gelation capacity; therefore, a lower least gelation concentration enhances the gelling ability of starches. The results showed that LGC increased after succinylation. The rise in LGC after succinylation was likely caused by granular disintegration and structural fragmentation during the modification process. However, both UBS and MTBS samples could form a better gel at a lower concentration (8%). At 6% (w/v), the UBS was viscous, while the MTBS starch remained liquid. Additionally, a firm gel formed in UBS at concentrations ranging from 12-18% (w/v). These observations indicated that the UBS possessed better gelation properties than the MTBS. Similar results have been reported for the native and modified (physical and chemical) breadfruit starch (Adebowale *et al.,* 2005).

Table 2: Gelation properties of UBS, OSABS and MTBS derivatives of *Dioscorea dumetorum.*

Concentration (%w/v)	**Starch sample**		
	UBS	**OSABS**	**MTBS**
2	-Liquid	-Liquid	-Liquid
4	-Viscous	-Liquid	-Liquid
6	-Viscous	-Liquid	-Liquid
8	+Gel	-Liquid	+Gel
10	+Gel	-Viscous	+Gel
12	+Firm gel	+Gel	+Gel
14	+Firm gel	+Gel	+Gel
16	+Firm gel	+Gel	+Gel
18	+Firm gel	+Gel	+Gel
LGC[a]	8	12	8

UBS – Untreated bitter yam starch; MTBS – Microwave treated bitter yam starch; OSABS – Octenyl succinic anhydride bitter yam starch.

[a] Least gelation concentration.

Transparency for UBS, OSABS and MTBS samples

The clarity (light transmittance) of starch paste, an essential factor affecting its applications, can significantly impact the sensory qualities and appearance of starch products. Among the samples, OSABS exhibited the highest paste clarity at 65.54%, surpassing UBS at 59.21% and MTBS at 56.23% (Figure 5). The results indicate that the chemical substitution of hydroxyl groups with octenyl succinic anhydride groups disrupts the ordered structure of the starch paste, thereby inhibiting retrogradation. This alteration results in a more fluid paste with improved clarity, enhancing the transparency of OSABS paste. This finding agrees with the observations of Huang *et al.* (2016), on octenyl succinic anhydride-modified starch prepared using a non-conventional technology. In contrast, microwave treatment may increase the mobility of starch molecules, resulting in reduced light transmittance of the MTBS sample compared to the UBS sample.

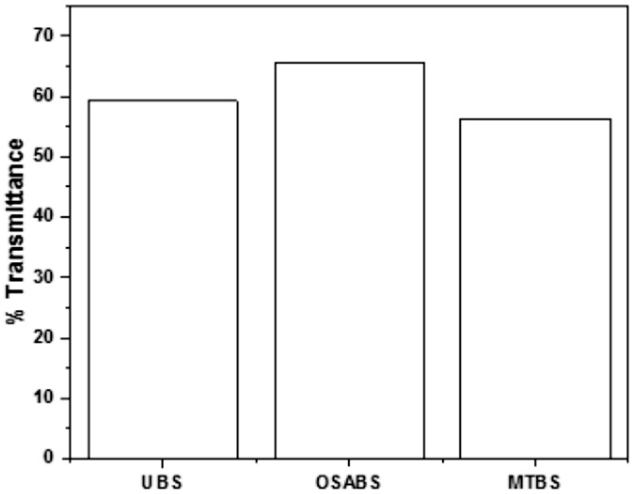


Figure 5: Light transmittance of UBS, OSABS and MTBS derivatives of *Dioscorea dumetorum*.

Swelling power and solubility for UBS, OSABS and MTBS samples

Figures 6 and 7 illustrate the swelling and solubility capacities of UBS, OSABS, and MTBS samples, respectively, evaluated at five different temperatures. The swelling index indicated that both UBS and OSABS samples exhibited a significant increase in swelling power, aligning with the findings of Ignot-Gutiérrez *et al.* (2020). UBS showed a swelling power range from 2.95 to 9.25 g/g, while OSABS ranged from 1.25 to 13.05 g/g across the various temperatures studied. Notably, OSABS demonstrated a superior swelling power compared to UBS, particularly at 70 and 80°C. The improvement in swelling power could be due to enhanced water percolation into the particles, facilitated by the repulsion effects of the introduced octenyl succinic anhydride. Additionally, it is possible that at these temperatures, the intermolecular association forces of the starch granules were weakened, contributing to the increase in swelling power (Ignot-Gutiérrez *et al.* 2020). Conversely, the swelling power of MTBS decreased with rise in temperature. The swelling power of MTBS ranged from 6.51 to 10.81 g/g at the different temperatures examined. This decrease in swelling power could be attributed to the interactions between amylose-amylose and amylopectin-amylopectin chains (Olayinka *et al.,* 2008). In Figure 7, the solubility profile was dependent on temperature, with solubility gradually increasing as the temperature rose for all samples investigated. The UBS sample exhibited higher solubility than OSABS and MTBS at 60 and 70°C, while the MTBS sample had the highest solubility at 80°C. OSABS showed the highest solubility at 40°C and 50°C. This solubility indicates the degree of dissolution during the starch swelling process (Carcea & Acquistucci, 1997). The solubility index ranged from 12.37 to 15.06% for UBS, 13.06 to 15.54% for OSABS, and 12.72 to 16.13% for MTBS. The solubility results were consistent with the findings reported by Lawal *et al.* (2005).

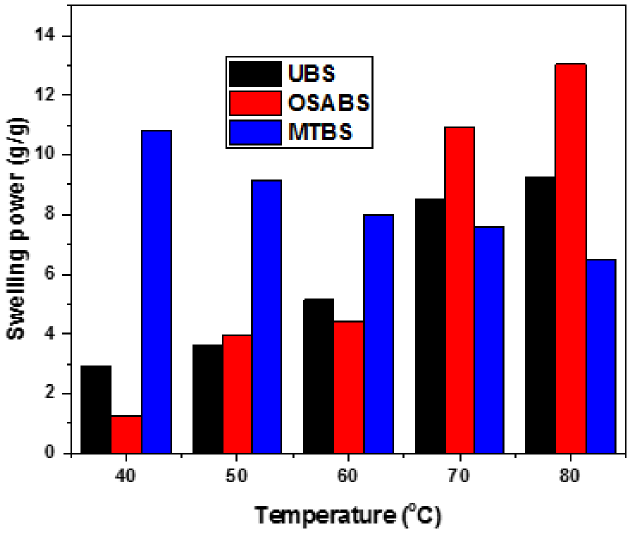


Figure 6: Effect of temperature on swelling power of UBS, OSABS and MTBS derivatives of *Dioscorea dumetorum.*

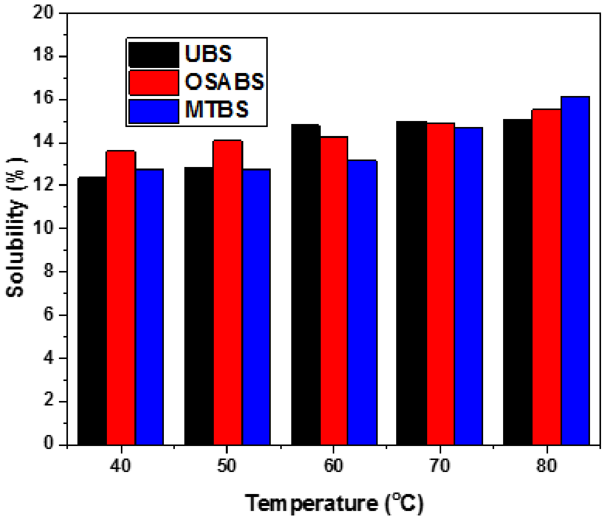


Figure 7: Effect of temperature on solubility of UBS, OSABS and MTBS derivatives of *Dioscorea dumetorum.*

Pasting characteristics of UBS, OSABS and MTBS derivatives of *Dioscorea dumetorum*

Table 3 outlines the variations in pasting behavior among the UBS, OSABS, and MTBS samples. Pasting temperature, the temperature at which starch granules begin to swell, was significantly different for the modified samples (OSABS and MTBS) compared to the untreated bitter yam starch (UBS). Both modifications resulted in a substantial increase in pasting temperature, likely due to alterations in the crystalline regions and reorientation of starch granule chains (Sun *et al.,* 2014).

Peak viscosity, reflecting the thickening power of the starch, was notably higher in UBS than in the other samples. Final viscosity, which measures the starch's ability to form a thick paste or gel after cooking and cooling, ranged from 1,557 to 1,909 cP across all samples. The MTBS sample exhibited the lowest final viscosity, while the UBS sample had the highest. Setback viscosity, indicating the recrystallization of gelatinized starch, varied from 524 to 727 cP, with lower values observed in OSABS and MTBS.

Breakdown viscosity, which reflects starch fragility under heat and shear, decreased following modifications. All modified starches showed reduced breakdown viscosity, suggesting increased thermal stability. The hierarchy of breakdown viscosity was UBS > MTBS > OSABS. This reduction in breakdown viscosity in the modified starches is likely due to the rearrangement of starch granules, enhancing resistance to heat and shear (Stute, 1992), and possibly the formation of lipid-amylose complexes during modification, as noted by Pukkahuta *et al.* (2008).

Table 3: Pasting characteristics of UBS, OSABS and MTBS derivatives of *Dioscorea dumetorum.*

Parameters	**Starch sample**		
	UBS	**OSABS**	**MTBS**
PV (cP)	1374	1291	1209
TV (cP)	1182	1124	1083
BV (cP)	192	167	176
FV (cP)	1909	1683	1557
SV (cP)	727	559	524
P_{Temp} (°C)	76.26	78.14	81.44

PV: Peak viscosity; TV: Trough viscosity; BV: Breakdown viscosity; FV: Final viscosity; SV: Setback viscosity; P_{Temp}: Pasting temperature

CONCLUSION

The impact of chemical modification via succinylation and physical modification through microwave treatment on the functional, physicochemical and morphological properties of bitter yam starch has been studied. Octenyl succinylated starch demonstrated greater apparent swelling power and paste clarity. Additionally, the modified starches were examined using FTIR, TGA, XRD, SEM, and optical microscopy. It was concluded that the microwave treatment method regions could alter the granular integrity. XRD analysis confirmed the disruption of the crystalline of the starch sample after microwave treatment. The findings revealed that the native bitter yam starch (UBS) had lower levels of fat, protein, fiber, and ash compared to the modified starches, while its moisture content was higher. The modifications notably reduced the pasting and gelation properties of the native starch. Both modification treatments enhanced the swelling and solubility capacities of OSABS and MTBS, respectively, at 80 °C. The present study's increased scientific knowledge on the effects of succinylation and microwave treatment on bitter yam starch, and thus, will expand its applications in both the food and non-food industries. This expansion is driven by the specific characteristics desired, such as crystallinity, granular surface, thermal behavior, pasting, swelling power, and solubility. Further investigation into the modification of bitter yam starch is needed to optimize its functionality for use in food and related industries.

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