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To cite this article: H.K. Tihamiyu, J.M. Babajide & S.A.O. Adeyeye | (2017) RETRACTED ARTICLE: Some quality attributes of heat-moisture treated water yam (*Dioscorea alata*) starch, Cogent Food & Agriculture, 3:1, 1278192, DOI: [10.1080/23311932.2016.1278192](https://doi.org/10.1080/23311932.2016.1278192)

To link to this article: <https://doi.org/10.1080/23311932.2016.1278192>



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Published online: 02 Feb 2017.



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Received: 25 October 2016  
Accepted: 27 December 2016  
First Published: 04 January 2017

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

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## FOOD SCIENCE & TECHNOLOGY | RETRACTION

# RETRACTED ARTICLE: Some quality attributes of heat-moisture treated water yam (*Dioscorea alata*) starch

H.K. Tiamiyu<sup>1\*</sup>, J.M. Babajide<sup>2</sup> and S.A.O. Adeyeye<sup>3</sup>

**Abstract:** This study investigated the effect of autoclave and oven heat-moisture treatment on some quality attributes of water yam starch. The water yam starch was adjusted to moisture content of 15–35% and subjected to heat-moisture treatment (HMT) using autoclave (at 120°C for 1 h) and oven (at 120°C for 12 h). Physicochemical, functional and thermal properties of the modified starches formed were carried out. Increase in amylose and resistant starch content of the samples occurred after the treatment. The water and oil absorption capacities also increased after HMT from 74.40 to 135.46% and 0.65 to 1.37 ml/g respectively. Swelling power was reduced while the solubility increased compared to native starch. Pasting time and temperature increased after HMT. A reduction in peak viscosity (643.03–80.82 RVU), trough (441.33–65.79 RVU), breakdown (203.13–93.58 RVU), and final viscosity (549.29–112.75 RVU) and set back (108.17–40.17 RVU) was noticed. Moisture content above 15% resulted in biphasic endotherms in both treatments,  $T_0$ ,  $T_p$ ,  $T_c$  and  $T_c - T_0$  also increase. The temperature of the heating medium was found to affect the RS content of the starches.

**Subjects:** Environment & Agriculture; Bioscience; Food Science & Technology

**Keywords:** autoclave; heat-moisture treatment; oven; resistant starch; water yam

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### PUBLIC INTEREST STATEMENT

Despite the high levels of yam production in the yam zone of West Africa and the high starch content of yam (60–80%) on a dry basis, yams are yet to be harnessed among the most common sources of industrial starch unlike corn, potato, wheat, tapioca and rice. This is primarily due to difficulty in the extraction of pure starches and its derivatives using affordable and domestic approaches that could be adopted by small and medium scale enterprises. Starch modification impacts some qualities into native starch which improve its stability during processing. Modifying yam starch by heat-moisture treatment could increase its competitiveness in international market. In addition, the use of this modified yam starch in food formulation would help to improve consumers' health and sensory acceptability due to the enhanced resistant starch.

## 1. Introduction

Yams belong to the genus *Dioscorea* spp. of which water yam, *Dioscorea alata* is one of the six yam species of economic importance, having high yield, high nutritive value, high multiplication ratio and better storability compared to other yams species but yet has been of low commercial quality probably because of its perceived unimpressive food quality traits (Wireko-Manu, Ellis, Oduro, Asiedu, & Maziya-Dixon, 2011). Due to the high starch content (75–84%) of the *D. alata* tubers they provide a good source of dietary carbohydrate in the tropics and subtropical regions (Osagie, 1992). Starch, an important carbohydrate in higher plants, is widely used in food, pharmaceutical and other industry because of its adhesive, thickening, gelling, swelling and film forming properties (Kunle, Ibrahim, Emeje, Shaba, & Kunle, 2003), deserves detailed investigation to understand better its structural, physicochemical and functional characteristics. The physicochemical, morphological, thermal and crystal properties of starch obtained from yam using ordinary method have been reported to boost its competitiveness on an industrial scale (Oke, Awonorin, & Workneh, 2013; Otegbayo, Oguniyan, & Akinwumi, 2014; Wang et al., 2006). However, the properties of starch obtained from the yam using heat-moisture treatment have not been fully investigated.

Heat-moisture treatment (HMT), a physical modification technique for starch is carried out at limited moisture contents (<35% moisture w/w) for a certain time period (15 min–16 h) and at temperatures (84–120°C) above glass transition temperature ( $T_g$ ) but below the gelatinization temperature (Chou, Wu, Nurtama, & Lin, 2010). During HMT, changes in X-ray patterns, granule swelling, thermal, pasting properties and reduction in enzyme susceptibility occurred depending on the conditions applied during treatment. Recent interest in resistant starch in food formulation has been on the increase due to its various health benefits in the diets which include: improving glucose and lipid metabolism, reducing the risk of the development of type 2 diabetes mellitus, obesity, coronary and inflammatory bowel diseases, and gastrointestinal disorders (Gelencser, 2009). Many researchers have demonstrated how heat-moisture treatment (HMT) could be used to increase the yield of RS (Chou et al., 2010; Chung, Hoover, & Liu, 2009; Chung, Liu, & Hoover, 2009; Li & Gao, 2010). Studies of HMT effect on starch pasting characteristics as reported by Stute (1992) reveal a higher onset temperature of viscosity development, a lower peak viscosity, and a higher or lower end viscosity. Adebawale, Henle, Schwarzenbolz, and Doert (2009) also reported decreased in swelling and solubility but increased water absorption of African yam bean starch after HMT.

Modified starches are gaining wider applications due to the improved properties impacted in them. Owing to its safety and cheapness, physically modified starches are highly preferred in most industries (especially pharmaceutical and food industries). The aim of this study was to determine the effect of autoclaving and oven moisture heat treatment on some quality attributes of water yam (*D. alata*) starch to enhance utilization, demand and market value of the species.

## 2. Materials and methods

### 2.1. Materials

Water yam tubers of the white variety were purchased from Kuto market in Abeokuta, Ogun State, Nigeria. The resistant starch kit used in this study was purchased from Megazyme International, Ireland, and all chemicals and reagent used were of analytical grade.

### 2.2. Methods

#### 2.2.1. Extraction of starch

Yam starch was extracted by the method described by Moorthy (2002). The tubers were peeled, washed and cut into small cubes and homogenize in a warring blender model number (model number (Philips-HR, 2001), for about two minutes at the minimum speed setting available. The blending was done intermittently to prevent the starch from eating up. The resultant slurry was packed into a muslin cloth and lowered into distilled water inside a bucket. The content in the cloth was continuously squeezed to expel the starch into the water. The starch was allowed to settle and the supernatant

decanted. The sediment (starch) was washed several times with distilled water to remove impurities from the starch. The starch was dried in a cabinet drier at a temperature of 45°C overnight and the dried starch was milled and stored in an airtight container until used.

#### 2.2.2. Preparation of heat-moisture treated water yam (*D. alata*) starch

Heat-moisture treatment was carried out using two different heating methods (Autoclave and oven heat treatment). The moisture content (MC) of starch samples were adjusted to 15, 20, 25, 30, 35% (the moisture content of the raw starch was pre-determined) by adding appropriate amount of distilled water. The starch/water mixtures were mixed thoroughly. For heat-moisture treated (HMT) using autoclave heat treatment, the method described by Lim, Chang, and Chung (2001) was followed. The starch samples were transferred into different pressure-resistant glass bottle (500 ml) and sealed with a cork and were then autoclaved at 120°C for 1 h. The heat-treated starches were cooled to room temperature and the samples were oven dried (40°C) to moisture level of ~13%. For HMT using oven method, the samples were sealed into different stainless steel containers and kept for 24 h at ambient temperature. The sealed samples were heated in a thermostatically controlled convection oven at 120°C for 12 h and cooled to room temperature. The starch samples were removed from the containers and dried at 45°C to uniform moisture content (~8%) (Li & Gao, 2010). All the samples were ground and screened through a 70 mesh size, then stored. Based on the treatment the starch sample were labelled as AHMT15, AHMT20, AHMT25, AHMT30 and AHMT35; OHMT15, OHMT20, OHMT25, OHMT30 and OHMT35.

#### 2.3. Resistant starch determination

The method for determining resistant starch (RS) contents was according to the analysis procedure provided by the Resistant Starch Assay Kit (Megazyme International Ireland). Starch and 4 ml enzyme mixture were added to each test tube, and then incubated in a shaking water bath for 16 h (37°C, 200 strokes/min) to hydrolyze digestible starch. The resistant portion was deposited with 95% ethanol and residue obtained was washed with 50% ethanol twice, and then treated with KOH solution (4 M, 2 ml) to solubilize RS. RS solution obtained was adjusted to pH 4.75 with 1.2 M sodium acetate buffer (8 ml, pH 3.8) incubated with amyloglucosidase (0.1 ml, 3,300 U/ml) at 50°C for 30 min. Samples were centrifuged at 1,500 g for 10 min. Aliquot (0.10 ml) of the supernatant was added with 3 ml of GOPOD and the mixture was incubated at 50°C for 20 min. Absorbance was measured using a spectrophotometer (Model 722, Shanghai No.3 Analytical Instrument Company, Shanghai, China) at 510 nm.

#### 2.4. Amylose content determination

Amylose was determined by using the method of Williams, Kurzina, and Hlynka (1970). Starch sample of 0.1 g was weighed into a 100 ml conical flask and dissolved with 1 ml of 95% ethanol and 9 ml of 1N NaOH was added to hydrolyse the starch. The flask was transferred to a boiling water bath for 10 min, removed and distilled water was added to make the volume 100 ml. About 5 ml of the sample was taken from 100 ml into another flask and 1 ml of acetic acid was pipetted out into each plus 2 ml iodine solution to change the colour. Distilled water was added to make up to 100 ml and the absorbance was read at 620 nm on the Spectrophotometer. The amylose content was calculated as thus:

$$\text{Amylose (\%)} = 3.06 \times A \times 20$$

where A = absorbance.

#### 2.5. The water and oil absorption capacities

These were determined using the method of Medcaf and Gillies (1965). About 2.5 g of each starch samples were weighed into a centrifuge tubes and 15 ml of distilled water/oil was added and then centrifuged at 3,000 rpm for 10 min. The water or oil was then decanted, and the tubes drained for 10 min. Quantity of water or oil bound by the samples was then determined by obtaining the weight difference, and the water or oil absorption capacity calculated thus:

$$\text{Water absorption capacity} = \frac{\text{Grams of Bound Water}}{2.5 \text{ g}} \times 100\%$$

$$\text{Oil absorption capacity} = \frac{\text{Grams of Bound oil}}{2.5 \text{ g}}$$

### 2.6. pH determination

5 g of yam starch was weighed and mixed with 50 ml of distilled water to obtain slurry. The pH was then determined using a Fisher Science Education pH meter (Model S90526, Singapore) meter by inserting the pH probe into the slurry.

### 2.7. Swelling power and solubility index

This was determined by the Takashi and Seib (1988) method. About 1 g of the starch sample was weighed into a centrifuge tube. About 10 ml of distilled water was then added and mixed gently. The slurry was heated in a water bath at 70, 80, 90 and 100°C respectively for 15 min. During heating, the slurry was stirred gently to prevent clumping of the starch. On completion of 15 min, the tube containing the paste was centrifuged at 3,000 rpm for 10 min after which the supernatant was decanted immediately. The sediment was then weighed and recorded. The decanted supernatant was then evaporated at 105°C for 3 h. Swelling power and solubility was calculated from the equations:

$$\text{Swelling power (g/g)} = \frac{\text{Weight of the wet mass of sediment}}{\text{Weight of sample}}$$

$$\text{Solubility index (\%)} = \frac{\text{Weight of dry supernatant}}{\text{Weight of sample}} \times 100$$

### 2.8. Pasting properties

Pasting characteristics were determined with a Rapid Visco Analyser (RVA Super 3, Newport Scientific Pty. Ltd, Australia). About 3 g sample starch was dissolved in 25 ml of water in a sample canister. The sample was thoroughly mixed and fitted into the RVA as recommended. The slurry was heated from 50 to 95°C with a holding time of 2 min followed by cooling to 50°C with another 2 min holding time. The 12 min profile was used and the rate of heating and cooling was at a constant rate of 11.25°C/min. Corresponding values for peak viscosity, trough, breakdown, final viscosity, setback, peak time and pasting temperature from the pasting profile were read from a computer connected to the RVA.

### 2.9. Starch morphology

Granule morphology was examined using a DSM 9872 GEMNI Scanning Electron Microscope (SEM). A thin layer of starch granule was mounted on the aluminum specimen holder by double-sided tape. It was coated with gold/palladium, with a thickness of about 30 nm. The size of the starch granules was recorded at desired magnification.

### 2.10. Polarized light microscopy

Birefringence of native and HMT starch granules were observed under an optical microscope (model BH-2, Olympus, Japan). The images were recorded at the same magnification (400×) for all starch samples (1.0% starch suspension).

### 2.11. Thermal properties

The gelatinization characteristics of the native and HMT starches were measured using differential scanning calorimetry (DSC6100, Seiko Instruments, Chiba, Japan). A sample (3 mg) was placed in an aluminum pan (Seiko Instruments, Chiba, Japan), and distilled water (7 µl) was added. The sample pan was sealed, allowed to equilibrate at room temperature for 2 h, and heated from 20 to 130°C at a rate of 5°C/min. An empty pan was used as a reference. The onset ( $T_o$ ), peak ( $T_p$ ) and conclusion ( $T_c$ ) temperatures were determined from the thermograms.

### 3. Results and discussion

#### 3.1. Influence of heat-moisture treatment on resistant starch formation

The resistant starch results presented in Table 1 show significant differences ( $p \leq 0.05$ ) among the starches. After HMT, oven heat-moisture treated starches (OHMT) had higher RS than the autoclaved heat-moisture treated (AHMT) starches for the same moisture content. The RS values for AHMT and OHMT starches ranged from 11.65 to 48.72 and 21.73 to 57.62% respectively and 36.97% in native starch. The highest value was observed in OHMT15 and lowest in AHMT35. An increase in resistant starch (RS) values of the starches after heat-moisture treatment was observed. Researchers like Sankhon, Amadou, Yao, Wang, and Mlyuka (2014), Chou et al. (2010), Li and Gao (2010) and Kim, Mullan, Hampson, and Pluske (2006) had reported increase in RS after HMT for varying level of moisture content for different starch source. This was associated with amylose content of starch because amylose have a high degree of polymerization and forms enzyme resistant double helices which were stabilized by hydrogen bonds resulting in increasing RS during HMT under non-gelatinized conditions.

However, a decrease in RS occurred for AHMT starch than the native starch as moisture content increased from 25 to 35% and the same was observed for OHMT at 35% moisture content. A decrease in RS after HMT for jackfruit starch was reported, where starch treated at MC of 30% and 35% at temperature 100 and 120°C for 12 and 16 h showed diminished RS content (Kittipongpatana & Kittipongpatana, 2015). Similarly, Ambigaipalan, Hoover, Donner, and Liu (2014) had also reported significant decrease in RS content of Faba beans at 120°C, indicating that temperature and moisture content play a crucial role in RS formation.

This opinion was also reported by Eerlingen, Jacobs, Van Win, and Delcour (1996) that both the moisture content and the temperature used in HMT could affect the organization of the crystalline portions in the starch granules by allowing more access of the enzymes into the granules. The decrease in RS content observed in samples treated at 25–35% MC and high (120°C) temperatures could also be a result of partial gelatinization. Higher RS degradation in autoclaved HMT than Oven HMT could be due to severity of pressure and temperature operating under autoclave heat treatment.

**Table 1. Some physicochemical properties of native and HMT *D. alata* starch**

Samples	RS content	Amylose content	Water binding capacity	Oil absorption capacity	pH
NS	36.98 <sup>c</sup>	34.05 <sup>a</sup>	74.40 <sup>a</sup>	0.65 <sup>a</sup>	6.87 <sup>d</sup>
AHMT15	48.72 <sup>d</sup>	39.97 <sup>f</sup>	113.86 <sup>b</sup>	0.77 <sup>d</sup>	7.03 <sup>j</sup>
AHMT20	43.18 <sup>cd</sup>	38.66 <sup>e</sup>	109.06 <sup>b</sup>	0.68 <sup>c</sup>	6.98 <sup>h</sup>
AHMT25	34.14 <sup>c</sup>	36.68 <sup>c</sup>	113.06 <sup>b</sup>	0.83 <sup>f</sup>	6.91 <sup>f</sup>
AHMT30	22.35 <sup>b</sup>	35.50 <sup>b</sup>	135.46 <sup>c</sup>	1.06 <sup>i</sup>	6.90 <sup>e</sup>
AHMT35	11.65 <sup>a</sup>	37.16 <sup>c</sup>	128.53 <sup>bc</sup>	1.37 <sup>k</sup>	6.81 <sup>b</sup>
OHMT15	57.62 <sup>e</sup>	40.04 <sup>f</sup>	113.86 <sup>b</sup>	0.88 <sup>b</sup>	7.02 <sup>i</sup>
OHMT20	47.99 <sup>d</sup>	38.83 <sup>e</sup>	114.93 <sup>b</sup>	0.66 <sup>b</sup>	6.93 <sup>g</sup>
OHMT25	41.95 <sup>cd</sup>	37.55 <sup>cd</sup>	110.93 <sup>b</sup>	1.20 <sup>j</sup>	6.85 <sup>c</sup>
OHMT30	38.12 <sup>c</sup>	35.27 <sup>b</sup>	115.73 <sup>bc</sup>	0.84 <sup>g</sup>	6.93 <sup>g</sup>
OHMT35	21.73 <sup>b</sup>	37.35 <sup>c</sup>	118.40 <sup>bc</sup>	0.80 <sup>e</sup>	6.69 <sup>a</sup>

Notes: NS = Native starch; AHMT15 = Autoclave heat-moisture treatment at 15% MC; AHMT20 = Autoclave heat-moisture treatment at 20% MC; AHMT25 = Autoclave heat-moisture treatment at 25% MC; AHMT30 = Autoclave heat-moisture treatment at 30% MC; AHMT35 = Autoclave heat-moisture treatment at 35%MC; OHMT15 = Oven heat-moisture treatment at 15% MC; OHMT20 = Oven heat-moisture treatment at 20% MC; OHMT25 = Oven heat-moisture treatment at 25% MC; OHMT30 = Oven heat-moisture treatment at 30% MC; OHMT35 = Oven heat-moisture treatment at 35% MC.

Means values with different letters within each column are significantly different ( $p < 0.05$ ).

### 3.2. Effect of heat-moisture treatment on amylose content

The amylose content of HMT *D. alata* starch increased after modification compared to native starch (34.05%), as observed in Table 1. There was significant differences ( $p < 0.05$ ) between amylose of starches regardless of the heat-treatment method used. The amylose content after HMT was in the range of 35.50–39.97% for AHMT starches and 35.87–41.04% for OHMT starches. Previous reports (Noda, Takahata, & Nagata, 1992; Riley et al., 2004; Riley, Wheatley, & Asemota, 2006) have shown that the amylose content also plays a key role in the digestion of starches, as starches with low amylose contents were found to be more digestible than starches with high amylose content.

The increase in resistant starch had some conjunction with amylose content. Amylose contents increased in all the treated starches and HMT15 had the largest of all samples, meanwhile, its RS level was at the maximum of 57.62% correspondingly. Li and Gao (2010) postulated that, it is likely that augmentation in amylose formation was susceptible to form longer or more ordered helical segments by amylose–amylose (AM–AM) and/or amylose–amylopectin (AM–AMP) interactions formed on HMT, which resulted in increased colour of the iodine solution during colorimetry determination.

### 3.3. Water and oil absorption capacities

Significant differences ( $p \leq 0.05$ ) existed in the water binding (WBC) and oil absorption (OAC) capacities of *D. alata* starch on heat-moisture treatment (Table 1). There was an increase in WBC from 74.4% in the native starch to 109.07–135.47% for the AHMT starches and 110.93–118.40% for OHMT starches. WBC increased with moisture content and was highest for AHMT30 and AHMT35. The increase in WBC implies that the hydrophilicity of *D. alata* starches was increased with HMT. These results are consistent with earlier reports on the water absorption properties of the HMT starches. Adebowale, Olu-owolabi, Olayinka, and Lawal (2005) reported that HMT linearly increased the WBC of red sorghum starch, which implies that hydrophilic tendency increased with moisture treatment.

The OAC also increased from 0.65 g/g in native starch to 0.68 and 1.37 g/g in AHMT starches and 0.66 and 1.20 g/g in OHMT starches. Increase in OAC is directly proportional to moisture content in AHMT starches while in OHMT starches increment did not follow increasing level of moisture content. According to Babu and Parimalavalli (2012) the increasing of the oil absorption capacity in HMT starches could be due to lipophilic nature on the granule surface and interior during HMT which were influenced for functional properties of starches.

### 3.4. Effect of HMT on pH

The mean values for pH are presented in Table 1. Significant differences ( $p < 0.05$ ) existed in pH among the starches. It was noticed that reduction in pH value occurred as moisture content increased though higher than native (6.87) except at HMT35% moisture content where values were lower (6.81 and 6.69). pH is an essential measurement of eating quality since it contributes to taste (Addy, Wireko-Manu, & Oduro, 2014). Slight increase in the pH of some HMT starches was noticed. The pH of both native and HMT starches were in line with WHO value 6.8–7.2 (Malami & Thompson, 2012).

### 3.5. Effect of temperature on swelling power and solubility index of native and heat-moisture treated *D. alata* starch

The effect of temperature on swelling power (SP) and solubility index (SI) of all the starches is shown in Table 2. With increment in temperature (70–100°C), the swelling power of the native and HMT starches were raised. Rapid increase was noticed in the swelling power of all the starches studied at 90°C, and a slow or almost constant rate at 100°C for HMT starches. The swelling power of native starch was higher than those of HMT starches and was significantly different expect at 70°C, this was consistent with previous studies involving some starches, for instance mung beans (Li & Gao, 2010), African locust bean (Sankhon et al., 2014), corn (Olu-Owolabi, Afolabi, & Adebowale, 2010), sorghum (Adebowale et al., 2005) and corn, pea and lentil (Chung, Hoover, et al., 2009), where the swelling capacity of starch were reported to be diminished by HMT.

**Table 2. Swelling power and solubility index of native and HMT *D. alata* starch**

Sample	SP70°C	SP80°C	SP90°C	SP100°C	SI70°C	SI80°C	SI90°C	SI100°C
NS	3.83 <sup>b</sup>	6.78 <sup>d</sup>	15.46 <sup>e</sup>	16.91 <sup>e</sup>	2.10 <sup>a</sup>	6.28 <sup>a</sup>	9.33 <sup>a</sup>	10.99 <sup>a</sup>
AHMT15	3.12 <sup>ab</sup>	4.27 <sup>ab</sup>	8.26 <sup>abc</sup>	9.11 <sup>ab</sup>	5.65 <sup>b</sup>	8.03 <sup>b</sup>	10.55 <sup>ab</sup>	13.62 <sup>b</sup>
AHMT20	3.52 <sup>ab</sup>	3.48 <sup>a</sup>	9.25 <sup>abc</sup>	9.76 <sup>abc</sup>	7.78 <sup>cd</sup>	9.94 <sup>cd</sup>	10.94 <sup>ab</sup>	15.18 <sup>b</sup>
AHMT25	3.60 <sup>ab</sup>	4.00 <sup>ab</sup>	10.05 <sup>ab</sup>	10.01 <sup>abc</sup>	6.64 <sup>bc</sup>	8.73 <sup>bc</sup>	12.64 <sup>cd</sup>	18.44 <sup>d</sup>
AHMT30	3.60 <sup>ab</sup>	3.86 <sup>a</sup>	7.80 <sup>a</sup>	8.70 <sup>a</sup>	9.39 <sup>ef</sup>	12.43 <sup>ef</sup>	13.95 <sup>de</sup>	17.60 <sup>de</sup>
AHMT35	3.63 <sup>ab</sup>	3.64 <sup>a</sup>	12.54 <sup>d</sup>	11.10 <sup>bcd</sup>	10.72 <sup>fg</sup>	13.94 <sup>f</sup>	19.39 <sup>f</sup>	18.58 <sup>de</sup>
OHMT15	2.79 <sup>ab</sup>	3.45 <sup>c</sup>	7.42 <sup>ab</sup>	8.50 <sup>a</sup>	7.06 <sup>bcd</sup>	9.31 <sup>bc</sup>	11.61 <sup>bc</sup>	14.41 <sup>b</sup>
OHMT20	2.65 <sup>a</sup>	4.78 <sup>b</sup>	9.70 <sup>abc</sup>	10.38 <sup>abc</sup>	9.30 <sup>de</sup>	11.39 <sup>de</sup>	15.66 <sup>e</sup>	16.61 <sup>cd</sup>
OHMT25	2.78 <sup>ab</sup>	3.94 <sup>a</sup>	10.35 <sup>c</sup>	11.41 <sup>cd</sup>	8.74 <sup>f</sup>	11.22 <sup>de</sup>	13.84 <sup>de</sup>	15.60 <sup>cd</sup>
OHMT30	2.74 <sup>ab</sup>	3.45 <sup>a</sup>	8.48 <sup>abc</sup>	9.50 <sup>abc</sup>	10.40 <sup>fg</sup>	12.66 <sup>ef</sup>	14.43 <sup>de</sup>	18.12 <sup>cd</sup>
OHMT35	3.52 <sup>a</sup>	5.59 <sup>c</sup>	12.47 <sup>d</sup>	12.48 <sup>d</sup>	11.72 <sup>fg</sup>	14.09 <sup>ef</sup>	18.51 <sup>f</sup>	19.73 <sup>cd</sup>

Note: Means values with different letters within each column are significantly different ( $p < 0.05$ ).

The lower swelling power of HMT starches could be attributed to restriction in the percolation of water within the starch matrices due to increased starch crystallinity after modification (Sankhon et al., 2014). Gunaratne and Hoover (2002) reported that additional interactions which may have occurred between amylose-amylose (AM-AM) and amylose-amylopectin chain during heat-moisture treatment may be partly responsible for the observed decrease in SP. They stated further that the decrease in SP on heat-moisture treatment could also be due to a decrease in granular stability, resulting from unraveling of double helices that may have been present in a crystalline array in the native starch. The reduction in the swelling capacity could also be due to the formation of amylose-lipid complexes within the starch granule (Tester & Morrison, 1990 in Gunaratne & Hoover, 2002). The swelling power of the AHMT and OHMT starches were noticed to decrease with decreasing moisture content.

The solubility of all the starches increased with temperature, and compared to the native starch were significant. The solubility of HMT starch was increased; however, the change in starch solubility of heat moisture treated mixture did not increase as the moisture content increased over the range of 15–35%. Other researchers have also reported increase in solubility on HMT, indicating that HMT starches had a higher solubility than native starch. The solubility increased as the temperature increased because of an increase in the mobility of the starch molecules, which facilitated dispersion of the starch molecules in water (Adebowale et al., 2005).

### 3.6. Pasting properties

Heat moisture treatment affected the pasting properties of the starches. There was a decrease in peak viscosity (PV), trough viscosity (TV), final viscosity (FV), break down (BD), and set back (SB) after HMT (except for the setback of OHMT15 and OHMT20), while the pasting temperature (PT) and peak time increased when compared with the native starch as shown in Table 3. These results indicated that heat moisture treatment had minimal effect on the granular arrangement of the starch with resultant lower swelling power when compared with their native starch. The result concurred with the report of (Adebowale et al., 2009; Zavareze et al., 2012). The reduction in the viscosity value as a result of HMT is a thinning effect which might be due to the weakening of bonding forces within the granules and their breakdown (Rasper, 1980).

The Peak viscosity (PV) indicates the ability of starch to swell freely before their physical breakdown and also the water binding capacity of starches. It was of the range 643.63 RVU for native starch, 87.58–151.96 RVU for AHMT starches and 94.58–243.08 RVU for OHMT starches. The reduction in PV after HMT was assumed to be a result of the reorganization within the granule of the modified starches. This led to low restricted swelling capacity and only a small amount of amylose was able



**Table 3. Pasting properties of native and HMT *D. alata* starch**

Sample	Peak viscosity (RVU)	Trough (RVU)	Breakdown (RVU)	Final viscosity (RVU)	Setback (RVU)	Peak time (Min)	Pasting temperature (°C)
AHMT15	151.96 <sup>e</sup>	98.46 <sup>a</sup>	53.50 <sup>d</sup>	164.79 <sup>a</sup>	66.33 <sup>a</sup>	6.94 <sup>d</sup>	86.35 <sup>c</sup>
AHMT20	94.50 <sup>ab</sup>	74.29 <sup>a</sup>	20.21 <sup>bc</sup>	119.92 <sup>a</sup>	45.63 <sup>a</sup>	7.00 <sup>d</sup>	89.18 <sup>d</sup>
AHMT25	105.38 <sup>bc</sup>	100.17 <sup>a</sup>	5.21 <sup>a</sup>	144.50 <sup>a</sup>	40.17 <sup>a</sup>	6.87 <sup>d</sup>	92.03 <sup>e</sup>
AHMT30	80.92 <sup>a</sup>	65.79 <sup>a</sup>	14.96 <sup>abc</sup>	112.75 <sup>a</sup>	46.83 <sup>a</sup>	7.00 <sup>d</sup>	95.08 <sup>f</sup>
AHMT35	87.58 <sup>ab</sup>	67.96 <sup>a</sup>	19.63 <sup>bc</sup>	128.46 <sup>a</sup>	60.50 <sup>a</sup>	7.00 <sup>d</sup>	95.10 <sup>f</sup>
OHMT15	243.08 <sup>g</sup>	140.38 <sup>a</sup>	96.21 <sup>e</sup>	474.13 <sup>c</sup>	330.54 <sup>b</sup>	5.94 <sup>a</sup>	83.88 <sup>b</sup>
OHMT20	184.42 <sup>f</sup>	170.08 <sup>a</sup>	14.33 <sup>abc</sup>	298.13 <sup>b</sup>	128.04 <sup>a</sup>	6.60 <sup>c</sup>	86.38 <sup>c</sup>
OHMT25	137.92 <sup>de</sup>	119.33 <sup>a</sup>	18.58 <sup>bc</sup>	207.08 <sup>ab</sup>	87.75 <sup>a</sup>	5.24 <sup>b</sup>	90.88 <sup>e</sup>
OHMT30	94.50 <sup>ab</sup>	84.92 <sup>a</sup>	9.58 <sup>ab</sup>	135.50 <sup>a</sup>	94.75 <sup>a</sup>	7.00 <sup>d</sup>	94.98 <sup>f</sup>
OHMT35	123.63 <sup>cd</sup>	98.33 <sup>a</sup>	25.04 <sup>c</sup>	161.17 <sup>a</sup>	63.25 <sup>a</sup>	6.97 <sup>d</sup>	94.99 <sup>f</sup>
NS	643.63 <sup>h</sup>	441.33 <sup>a</sup>	203.13 <sup>f</sup>	549.29 <sup>d</sup>	108.17 <sup>b</sup>	5.17 <sup>ab</sup>	80.75 <sup>a</sup>

Note: Sample means for each sensory attribute with the same letter are not significantly different at  $p > 0.05$ .

to leach into the medium to elevate its viscosity (Horndork & Noomhorm, 2007). Lower value of PV of the HMT starches may also be attributed to increase in amylose content of starch after modification.

HMT has been reported to increase the amylose content of starches with resultant lowering of PV. Trough value (TV) is the minimum viscosity value attained in the constant temperature phase of the RVA profile. It measures the ability to withstand breakdown during cooling. The trough viscosities (TV) were lower in the HMT starches than native starch.

Breakdown (BD) is an important criterion that decides the applicability of starch in food and industry. The breakdown viscosity of the HMT starches ranged between 5.21 and 53.50 RVU for autoclaved treatment and 9.58 and 96.21 RVU for oven treatment. The decrease in BD of HMT starches indicated that the granules were strong and resisted breakdown under shear and heat, a result also noted by Singh, McCarthy, and Singh (2006). It was speculated that HMT makes the granules resistant to deformation by strengthening the intragranular binding forces (Adebowale et al., 2009; Singh et al., 2006).

Setback, which is an indication of starch retrogradation tendency after gelatinization was not significant ( $p > 0.05$ ) following HMT among the starch samples (though the values obtained for the HMT starches were lower than the native starch) except for OHMT15 which showed significantly ( $p < 0.05$ ) higher value than the native. Higher setback value for OHMT15 indicates that the starch will retrograde easily. Anderson and Guraya (2006) also found high SB value for microwave heat moisture treated waxy and non-waxy rice. Rahman (2000) reported that starches with higher amylose content exhibited higher setback value, more hardness and less stickiness, thus the setback was considered as an important criterion for starch selection for many food industries such as noodles meaning that the high set for OHMT15 starch could be of value in such industry.

The pasting time and temperature of the heat moisture treated starches increased significantly possibly because of increase in crystallinity, as a result of reorientation of the starch granules and the increase in pasting temperature is proportional to moisture content. The differences noticed in the pasting properties of the autoclaved and oven HMT starches could be due to the severity of disruption of the organized crystalline molecules during HMT which occurred more during autoclave treatment.

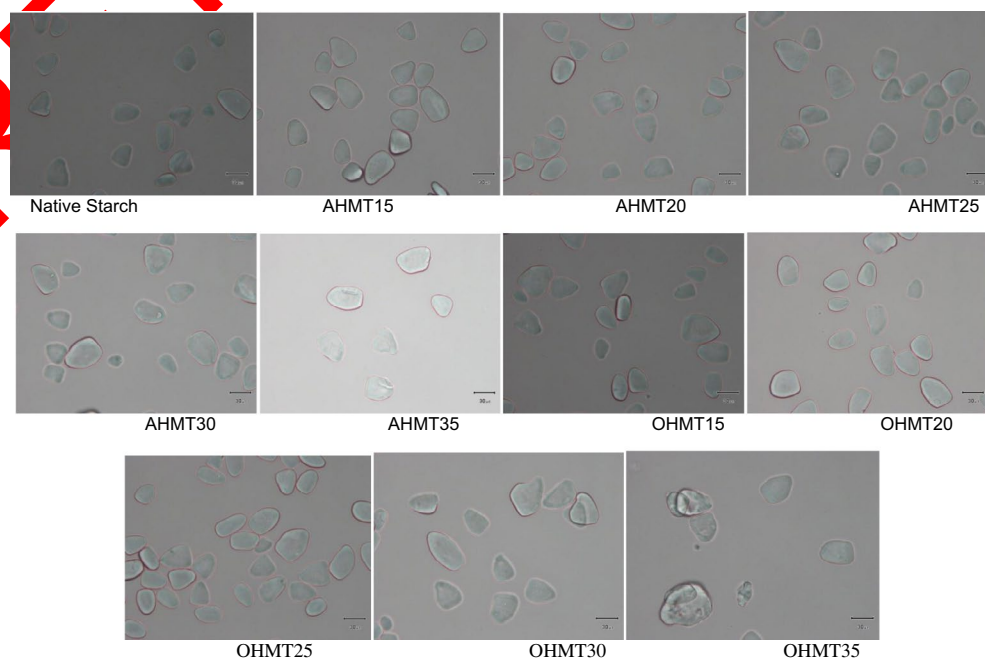
### 3.7. Granule morphology of native and heat-moisture treated starches

The shape and surface characteristics of native and modified starches are shown in Plate 1. Variable shapes were observed with the granules of the native and modified starches, consisting of small and large granule shapes which were oval, round, triangular or elliptical with smooth surfaces. The result revealed that for each moisture content the granules shape did not changed from the native except at 35% moisture content where some roughness and/or disintegration occurred on the granules surface. The disintegration of the granule at MC 35% noticed in the light micrograph of the starches may result from partial gelatinization. Slight aggregation of the granules was also noticed, which was more pronounced in AHMT25 and OHMT25. This had also been reported by Anderson and Guraya (2006) for normal and waxy rice starches granules after microwave HMT. This study showed that HMT had not altered the shape of the *D. alata* starches. And this concurred with the results presented for HMT of Taro starch (Yusnita & Siti, 2012) and waxy and normal starches (Jiranuntakul, Puttanlek, Rungsardthong, Pancha-arnon, & Uttapap, 2011), suggesting that changes in starch granular physical structure may not be necessary for internal recrystallization processes. Stute (1992) in Anderson and Guraya (2006) reported that gelatinization and damage to starch granules with respect to size, shape or birefringence do not occur during controlled application of heat/moisture to starches.

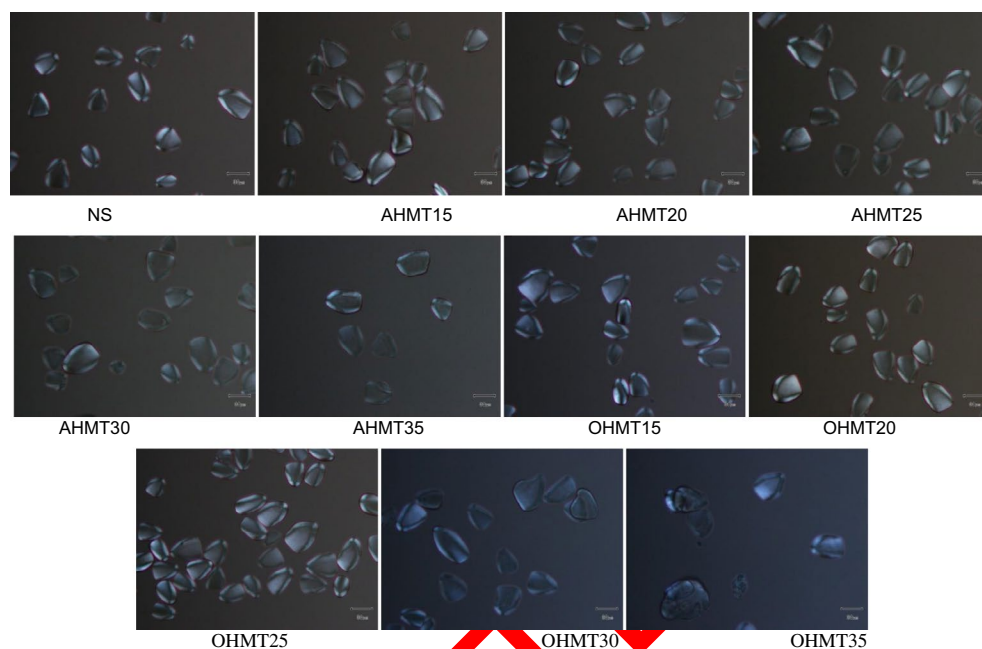
### 3.8. Polarized light microscopy of native and heat-moisture treated starches

Birefringence (Maltese cross) is indicative of the spot (hilum) where starch deposition originated during development in the leucocyte, which reflects the average radial orientation of helical structures (Chung, Hoover, et al., 2009; Chung, Liu, et al., 2009; Riley et al., 2006). Starch granules without a hilum generally do not show birefringence. The intensity of birefringence is influenced by granule shape and on the orientation of the granules with respect to the light beam. All the starches studied displayed birefringence upon exposure to polarized light though to varying degree. Native water yam starch displayed a strong birefringence pattern (Plate 2), which according to Chung, Hoover, et al. (2009), Chung, Liu, et al. (2009) and Sivak and Preiss (1998) is indicative of a great degree of order in the molecular orientation, a characteristic that is independent of crystallinity. The birefringence at the periphery and at the centre remained unchanged on HMT except at MC 30 and 35% where disappearance of the birefringence both at centre and periphery occurred in some granules. The decreased birefringence intensity at the granule center suggests that the thermal energy imparted to the double helices (forming the crystallites) during HMT may have increased their mobility thereby resulting in a loss of radial orientation. The retention of birefringence at periphery suggests that starch chains at the granule center are less organized than those at the periphery, and are

Plate 1. Light micrographs of native and HMT *D. alata* starch.



**Plate 2. Photomicrographs of native and HMT *D. alata* starches.**

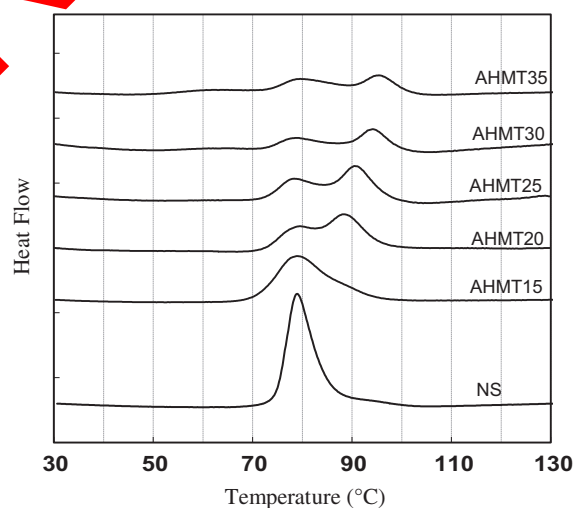


therefore more susceptible to reorientation during HMT (Chung, Hoover, et al., 2009; Chung, Liu, et al., 2009; Vermeulen, Goderis, & Delcour, 2006). Loss of birefringence at centre and/or periphery after HMT have also been reported for mung bean starch (Li & Gao, 2010); corn, pea and lentil (Chung, Hoover, et al, 2009); potato (Vermeulen et al., 2006)

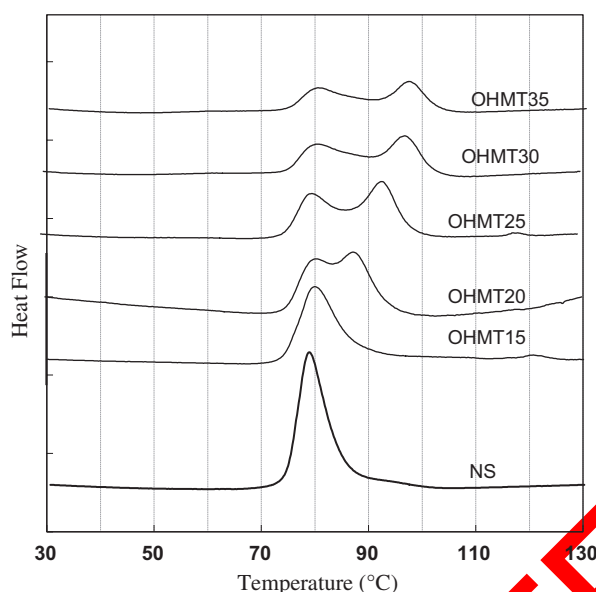
### 3.9. Thermal properties of native and heat-moisture treated *D. alata* starches

Figures 1 and 2 show the DSC of the HMT water yam starch at various moisture contents. The thermal parameters—Onset ( $T_o$ ), Peak ( $T_p$ ), Conclusion ( $T_c$ ) temperatures and transition temperature range ( $T_c - T_o$ ) are summarized in Table 4. The first peak of the thermogram on the DSC graph represents the gelatinization temperature of the starches. As moisture content increases above 15%, a second peak (biphasic transition) appears as a shoulder on the right side of the main peak in both heat treatments. This phenomenon have been observed for waxy and normal starches (Jiranuntakul et al., 2011), waxy and long grained rice (Shih, King, Daigle, An, & Ali, 2007), rice starch (Horndork & Noomhorm, 2007). The biphasic transition formed indicated that two crystalline regions with

**Figure 1. DSC thermograms of native and autoclave heat moisture treated *D. alata* starches.**



**Figure 2. DSC thermograms of native and oven heat moisture treated *D. alata* starches.**



**Table 4. Thermal transitions characteristics of native and HMT *D. alata* starch**

Sample	Oven					Autoclave				
	$T_0$	$T_{p1}$	$T_{p2}$	$T_c$	$T_c - T_0$	$T_0$	$T_{p1}$	$T_{p2}$	$T_c$	$T_c - T_0$
NS	72.5	78	-	87.5	15	72.5	78	-	87.5	15
HMT15	73.0	80	-	92.5	19.5	71.5	78.75	-	95	23
HMT20	73.5	80.8	87.5	95	21.5	73.0	79.5	88.75	100	27
HMT25	74.2	79.75	92.5	100	25.8	73.5	78.80	91.25	100	26.5
HMT30	74.6	81.2	96.25	105	23.8	74	79.75	93.75	102.5	28
HMT35	75	81.5	97.5	105.5	24	74.5	80	95	102.5	28.5

Notes: NS = Native starch; HMT15 = heat-moisture treatment at 15% moisture content; HMT20 = heat-moisture treatment at 20% moisture content; HMT25 = heat-moisture treatment at 25% moisture content; HMT30 = heat-moisture treatment at 30% moisture content; HMT35 = heat-moisture treatment at 35% moisture content.

different heat stabilities were formed during HMT (Jiranuntakul et al., 2011), which has been attributed to inhomogeneous distribution of moisture during the HMT process (Shih et al., 2007). Jiranuntakul et al. (2011) argued that the biphasic might be due to different susceptibility of the distinct two-type blocklet that construct starch granules to the HMT, resulting in two crystalline regions that exhibited different melting temperatures.

The gelatinization temperatures range from 72.50 to 87.50°C (native starch), 71.50 to 103.0°C (AHMT starches) and 73.0 to 105.50°C (OHMT starches), It was observed that HMT increased  $T_0$ ,  $T_p$ ,  $T_c$  and  $T_c - T_0$ , and this trend increased from HMT15 to HMT35% in both heat treatments. Increased  $T_0$ ,  $T_p$ , and  $T_c$  on HMT have been partly attributed to structural changes within the starch granules, which involves interaction between amylose-amylose (AM-AM) and/or amylose-amylopectin chains (AM-AMP), and to the formation of additional complexes between amylose and starch lipids (Chung, Hoover, et al., 2009). These interactions consequently reduced the mobility of the amylopectin chains, leading to increases in  $T_0$ ,  $T_p$  and  $T_c$  (Sankhon et al., 2014). Yusnita and Siti (2012) and Adebowale et al. (2009) were also of the opinion that increase in  $T_0$ ,  $T_p$  and  $T_c$  might also be due to the limited moisture level applied which may restrict the gelatinization as most of the starch granules remained intact and less disrupted. The increase in  $T_c - T_0$  after HMT indicating that HMT allowed the formation of new starch crystallites with different heat stabilities (Hoover & Manuel, 1996).

#### 4. Conclusion

Modification of Water yam starch (*D. alata*) at various moisture contents using heat-moisture treatment resulted in structural rearrangement within the starch granules. This significantly affected physicochemical properties of the starch. After Heat-moisture treatment, an increase in the amylose and RS content of the starches were noticed. The moisture content and the temperature of treatment significantly affected the resistant starch content. Higher moisture contents and/or temperature resulted in decreases in RS. The oven heat treatment showed increased RS than the autoclave heat treatment. Oven heat treated starch at 15% moisture content had the highest RS content. Increase in moisture content enhances the water absorption capacity of the starches. The pasting viscosities were reduced and pasting time and temperature increased. The reduced setback viscosities of the HMT starches could be of use in food where retrogradation is not required.

#### Funding

The authors received no direct funding for this research.

#### Competing Interest

The authors declare no competing interests.

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#### Citation information

Cite this article as RETRACTED ARTICLE: Some quality attributes of heat-moisture treated water yam (*Dioscorea alata*) starch. H.K. Tiamiyu, J.M. Babajide & S.A.O. Adeyeye, *Cogent Food & Agriculture* (2017), 3: 1278192.

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