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Physicochemical properties of pregelatinized and microwave radiated white and red cocoyam (*Colocasia esculenta*) starches

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ABSTRACT

The need to explore potentials of pregelatinization and microwave radiation as non laborious and inexpensive processes of starch modification and to promote utilization of cocoyam starch were the reasons behind this study. Starches extracted from white and red varieties of cocoyam were modified using two physical methods: pregelatinization and microwave radiation. Functional and pasting properties of native and modified starches were evaluated using standard methods. Swelling power of the starches (1.70 – 4.10) reduced significantly ($p<0.05$) with pregelatinization but increased significantly ($p<0.05$) with microwave radiation. Water absorption capacity (1.00 – 3.20 ml/g), packed bulk density (0.56 – 0.83 g/ml) and loose bulk density (0.46 – 0.64 g/ml) of the starches increased significantly ($p<0.05$) while least gelation concentration (4.00 - 8.00%) reduced significantly ($p<0.05$) as a result of modifications. Peak, trough, breakdown, final and setback viscosity of white and red cocoyam native starches were 3687 cP and 4144 cP; 2213 cP and 2519 cP; 1474 cP and 1625 cP; 3595 cP and 4142 cP; 1382 cP and 1623cP respectively. Pregelatinization significantly increased ($p<0.05$) these pasting viscosity values in white cocoyam starch but reduced them significantly ($p<0.05$) in red cocoyam starch. Microwave radiation caused a significant increase ($p<0.05$) in the pasting viscosity values of both white and red cocoyam starches except breakdown viscosity which reduced.

Introduction

Starch is a naturally occurring and abundantly available biodegradable molecule (Ashogbon and Akintayo, 2014). The utilization of native starch in food and pharmaceutical applications is limited by some of its properties which include water insolubility, tendency to retrograde and instability under different processing conditions of temperature, pH and shear forces (Alcazar-Alay and Meireles, 2015). These limitations are mitigated by modification of starch; modification changes structural attributes of starch granules and physicochemical properties of starch, it also increases its value for food and non food industries (Lopez et al., 2010). Starch can be modified by chemical, physical, enzymatic means or their combinations. Recently, attention is shifting to the use of physical methods in starch modification. Physical methods involve treatment of native starch under different temperature/moisture combinations, pressure, shear and radiation without the use of chemical reagents (Yousif et al., 2012); it is therefore accepted as simple and cheap means of improving the physicochemical properties of starch with no associated health risk (Ashogbon and Akintayo, 2014; Majzoobi et al., 2011). Physical methods such as pregelatinization, heat moisture treatment, annealing, microwave radiation have been used to modify yam, sorghum, potato, plantain and pinhao seeds starches (Adebowale et al., 2005; Oladebeye et al., 2011; Pinto et al., 2015; Nadir et al., 2015; Olatunde et al., 2017). Cocoyam starch, apart from cocoyam flour, is an intermediate shelf stable product and a means of reducing the post harvest losses of cocoyam corms; the corms are highly perishable with a lot of post harvest losses. Cocoyam starch, unlike cassava and corn

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starches, has not found wide application in food and non food industries. The property of cocoyam starch has been investigated (Aprianita et al., 2014; Gbadamosi and Oladeji, 2013; Awokoya et al., 2012; Himeda et al., 2012; Oladebeye et al., 2009). White and red cocoyam starch samples were reported to have similar X-ray B pattern with ellipsoidal shaped granules (Awokoya et al., 2012). Mweta et al. (2008) reported that cocoyam starch had low swelling ability but high retrogradation tendency. However, there is inadequate information on the property of physically modified cocoyam starch. This study reports on the potentials of pregelatinization and microwave radiation as non laborious and inexpensive processes of modifying starches obtained from white and red varieties of cocoyam.

Materials and methods

Starch isolation

Freshly harvested matured white and red cocoyam corms (*Colocasia esculenta*) were purchased from a cocoyam farmer at Ago Aduloju, Ado-Ekiti, Nigeria. Starches were isolated separately from the two varieties of cocoyam using the method described by Gbadamosi and Oladeji (2013) with some modifications. Cocoyam corms were washed, peeled, cut into small slices and then washed again, the slices were ground in a milling machine and the resulting slurry was mixed with distilled water (1:4). The mixture was sieved through muslin bag and the starch suspension was left overnight at refrigerated temperature (4 °C). The suspension was then decanted; the white starch sediment was washed 3 to 4 times by re-suspending in distilled water, settling and decanting until the supernatant became transparent. The isolated starch was dried in a Hinotek hot air oven (DHG 9030A; Hinotek Group Ltd., China) at 40 °C for 8 hours, milled using a conventional blender (VTCL High Performance Mixer and Grinder) and sieved through 0.5mm aperture. The starch sample was thereafter packaged in a sealed high density polyethylene and stored in air tight glass jars at ambient temperature (27 °C – 31 °C).

Modification of cocoyam starches

Pregelatinization

Pregelatinization of starches of white and red cocoyam was carried out according to the method described by Waliszewskia et al. (2003). 100 g of each starch sample was suspended in 150 ml of distilled water and heated in a water bath at 80 °C for 15 minutes with slow intermittent manual mixing using stirring rod. Thin film of the pregelatinized starch on a stainless steel tray was then dried in a Hinotek hot air oven (DHG 9030A; Hinotek Group Ltd., China) at 40 °C for 24 hours, cooled, milled using a conventional blender (VTCL High Performance Mixer and Grinder) and sieved through 0.5 mm aperture. The starch sample was thereafter packaged in a sealed high density polyethylene and stored in air tight glass jars at ambient temperature (27 °C – 31 °C).

Microwave radiation

The modified method of Lewandowicz *et al.* (2000) was used for the microwave treatment of starches of white and red cocoyam. The moisture content of 100 g of each starch sample was adjusted to 20% by adding appropriate amount of distilled water. The moistened starch sample was put in a 500 ml beaker, covered with perforated plastic (Sonhart Investement Ltd., Ogun State, Nigeria) and placed in a Samsung microwave oven (Model: ME731K, 2450 MHz, 800W; Samsung Electronics Co. Ltd., USA) for 4 minutes. After microwave treatment, the starch sample was cooled, milled using a conventional blender (VTCL High Performance Mixer and Grinder) and sieved through 0.5mm aperture. The starch sample was thereafter packaged in a sealed high density polyethylene and stored in air tight glass jars at ambient temperature (27 °C – 31 °C).

Functional properties

Swelling

Swelling power of starch samples at 60 °C, 70 °C, 80 °C and 90 °C was determined according to the method described by Kaur et al. (2011). Swelling power was reported as ratio of swollen sample to dry sample.

Water and oil absorption capacity (WAC and OAC)

Water and Oil absorption capacity of the starch samples were determined by using the procedure of Sathe et al. (1982). WAC and OAC were expressed as volume (ml) of water/oil absorbed per gram of sample.

Least gelation concentration

Least gelation concentration was determined according to the method described by Onwuka (2005). 5 ml suspension of each starch sample (2 – 20% w/v) was prepared in ten test tubes; the test tubes were heated in a boiling water bath (100 °C) for 1 hour, followed by cooling in cold water bath. The samples were further cooled at 4 °C for 2 hrs after which each test tube was inverted to determine least gelation concentration The concentration at which the sample did not fall when the tube was inverted was noted to be least gelation concentration.

Loose and packed bulk densities, and powder flowability

Loose and Packed bulk densities were determined using the method of Mpotokwane et al. (2008). Loose bulk density was calculated as the ratio of the weight of the sample to the volume while packed bulk density was calculated as the ratio of the tapped weight of the sample to volume. The flow properties of the starches were determined using Hausner ratio and the Carr index. The Hausner ratio and Carr index were evaluated using the following:

$$Hausner\ Ratio = \frac{\text{Packed bulk density}}{\text{Loose bulk density}} \quad (1)$$

$$Carr\ Index(\%) = \frac{\text{Packed bulk density} - \text{Loose bulk density}}{\text{Loose bulk density}} \times 100 \quad (2)$$

Hausner ratio is used to classify flow properties as follows: free flowing (good) 1.0–1.1, medium flowing (Fair) 1.1–1.25, difficult flowing (Poor) 1.25–1.4 and very difficult flowing (very poor) above 14 (Hayes, 1987).

Pasting properties

The pasting properties were determined using Perten Rapid Visco Analyzer (RVA 4500; Perten Instruments, Sweden) and thermocline for widows (TCW) software on computer for viscometric data acquisition. The 12 minutes profile was used. The following parameters were obtained; peak viscosity, trough viscosity, breakdown viscosity, final viscosity, setback viscosity, pasting temperature and peak time.

Statistical analysis

The difference in the experimental data was tested for statistical significance p<0.05 by Statistical Analysis of Variance (ANOVA) using SPSS 21 software package.

Results and discussion

Functional properties and flow characteristics of native and physically modified starches of white and red cocoyam varieties

Functional properties and flow characteristics of native and modified starches of white and red cocoyam varieties are presented in Table 1. Swelling power at 60 °C of red cocoyam native starch was higher than that of white cocoyam native starch; however this difference was not significant (p<0.05). Amylose content and molecular organization within the starch granules affect swelling of granules. Amylose content of red cocoyam starch (25.4%) is similar to that of white cocoyam starch (24.6%) (Lauzon et al., 1995), the difference in the swelling of the starches may therefore be due to different molecular organization of the red and white cocoyam starches. It is plausible that the degree of crystalline packing within starch granules and the associative force between the granules of red cocoyam starch are probably of a lower order of magnitude than in white cocoyam starch (Alam and Hasnain, 2009). This may have enhanced the penetration of water into crystalline regions and increased the hydration of linear fragment of amylopectin leading to increase in starch granules swelling. The swelling power of cocoyam starches was lower than the range (3.15 – 3.71) reported for cocoyam starch by Ojinnaka et al. (2009) but comparable with that of plantain starch (Olatunde et al., 2017).

Table 1. Functional properties and flow characteristics of native and physically modified starches of white and red cocoyam (*Colocasia esculenta*) varieties

Variety	Modification	Swelling Power @ 60ºC	WAC (ml/g)	OAC (ml/g)	LGC (%)	Packed Bulk Density (g/ml)	Loose Bulk Density (g/ml)	Hausner Ratio	Carr Index (%)	Flowability
White	**Native**	1.82± 0.05^{cd}	1.00± 0.00^{d}	1.20± 0.10^{a}	8.00^{a}	0.59± 0.02^{d}	0.46± 0.01^{d}	1.28^{a}	28.26^{a}	Poor
	Pregelatinization	1.70± 0.10^{d}	1.20± 0.10^{cd}	1.20± 0.20^{a}	4.00^{b}	0.67± 0.03^{c}	0.55± 0.00^{c}	1.22^{bc}	21.82^{c}	Fair
	Microwave Radiation	3.70± 0.30^{b}	2.40± 0.20^{b}	1.20± 0.00^{a}	4.00^{b}	0.79± 0.02^{a}	0.61± 0.02^{b}	1.30^{a}	29.51^{a}	Poor
Red	**Native**	2.10± 0.20^{c}	1.00± 0.00^{d}	1.00± 0.00^{a}	8.00^{a}	0.56± 0.01^{d}	0.47± 0.02^{d}	1.19^{c}	19.15^{d}	Fair
	Pregelatinization	2.00± 0.05^{cd}	1.40± 0.10^{c}	1.20± 0.20^{a}	4.00^{b}	0.74± 0.02^{b}	0.59± 0.01^{b}	1.25^{b}	25.42^{b}	Fair
	Microwave Radiation	4.10± 0.20^{a}	3.20± 0.20^{a}	1.20± 0.10^{a}	4.00^{b}	0.83± 0.03^{a}	0.64± 0.02^{a}	1.30^{a}	29.69^{a}	Poor

Values are means of triplicate determination; Mean values in the same column with different superscript are significantly different at 5% level (p<0.05)

Pregelatinization reduced the swelling power of cocoyam starches while microwave radiation caused a significant increase ($p<0.05$) in the swelling power. During pregelatinization the hydrogen bonds and the crystalline nature of starch are broken (Nakorn et al., 2009) leading to increase in water absorption and swelling. The partial gelatinization of the pregelatinized starch sample might have caused the sample to have lower swelling power when compared with native starch. Similar observation of reduction in swelling power of a local variety of *Dioscorea rotundata*, plantain and corn starches as a result of pregelatinization was reported by Bakre et al. (2014), Olatunde et al. (2017) and Yousif et al. (2012) respectively. The increase in swelling power of microwave treated cocoyam starches may be attributed to the effect of the radiation on the crystallinity of starch granules. The vibration of water molecules within starch granules initiated by microwave radiation energy may have disrupted the crystalline arrangement of the granules leading to increased water absorption and swelling of starch granules (Xie et al., 2013; Karkkainen et al., 2011).

Swelling power of the starches increased as temperature increased (Fig. 1 and 2), the increase was enormous between temperature of 70 °C and 80 °C. This may be due to the fact that at high temperature, starch molecules become more thermodynamically activated with increased granular mobility which enhanced penetration of water and improved swelling ability (Awokoya et al., 2012). Additionally, the disruption of the amorphous region of the granules at high temperature range may have reduced the restraining effect of amylose on granules swelling, thus allowing the granules to swell freely (Karim et al., 2007; Nadiha et al., 2010). White cocoyam starch continued to swell after the temperature of 80 °C was reached (Fig. 1); this may suggest that the starch granules maintained their integrity up to a temperature of 90 °C. However, the swelling of red cocoyam starch reduced after the temperature of 80 °C was reached which suggest that further heating to 90 °C caused the starch granules to lose their integrity. Throughout the temperature range (60 °C to 90 °C), the swelling power of cocoyam starches modified by microwave radiation was consistently higher than that of native starches; however, the swelling power of pregelatinized cocoyam starches was lower than that of native starches except at 70 °C.

Water absorption capacity (WAC) of white and red cocoyam native starches were similar (1.00 ml/g), this value was lower than 1.23 - 1.67ml/g reported for cocoyam starch by Ojinnaka et al. (2009). However, modification significantly increased ($p<0.05$) water absorption capacity of white and red cocoyam starches. The higher WAC of modified cocoyam starches may be due to the macromolecular disorganization and fragmentation of starch granules during the treatments (Alcazar-Alay and Meireles, 2015). Similar increase in WAC as a result of pre-gelatinization was reported for corn starch (Yousif et al. 2012). The oil absorption capacity (OAC) of white cocoyam native starch was higher than that of red cocoyam native starch. The difference in the density of lipophilic residues of protein on the surface of starch granules of the two cocoyam varieties, which affect the extent of hydrophobic interaction between the lipophilic residues and the hydrocarbon chains of lipids, may have been responsible for the difference in the OAC of the starches (Eltayeb et al., 2011). Modifications increased the OAC of red cocoyam starch from 1.00 to 1.20 mL/g while that of white cocoyam starch was not affected. Swelling, WAC and OAC are important functional attributes which influence the choice of starch in foods such as baked products, extruded products and mayonnaise.

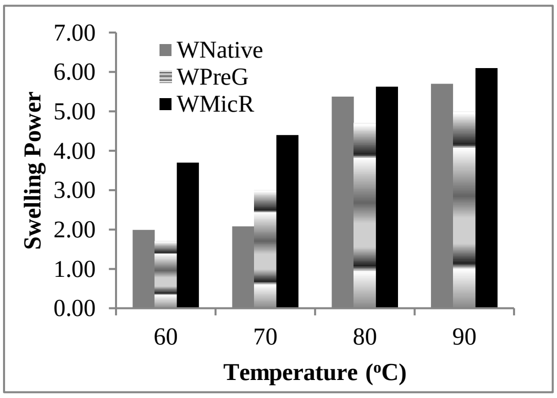

Fig. 1. Effect of Temperature on the Swelling Power of Native and Physically Modified Starch of White Cocoyam

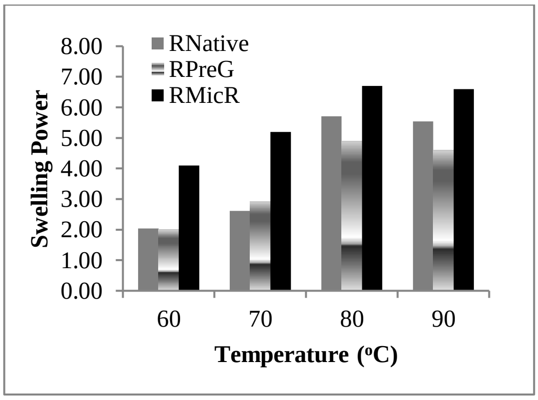

Fig. 2. Effect of Temperature on the Swelling Power of Native and Physically Modified Starch of Red Cocoyam

Cocoyam starches did not form gel until it reached a concentration of 8%; pregelatinization and microwave radiation enhanced the gelling ability of the starches with significant reduction ($p<0.05$) in least gelation concentration (LGC). The breaking of the semi-crystalline structure of pregelatinized starch may have enhanced the leaching and re association of amylose, and ultimately improved starch gelation. The disruption of crystalline structure induced by microwave radiation (Lewicka et al., 2015) may have allowed the entrance of water into granules and cause unhindered interaction between starch and water resulting in the leaching out of more amylose molecules. The self association of the leached amylose to form amorphous polymer network may have increased the gelation of microwave treated starches and thereby caused reduction in LGC (Wang and Copeland, 2012). This result suggests that modified cocoyam starch may perform better as gelling agents than native starch and may be suitable for products that require strong gelation ability especially at low concentration.

Packed and loose bulk densities of native starch of white and red cocoyam were 0.59 g/ml, 0.46 g/ml and 0.56 g/ml, 0.47 g /ml respectively. These values were lower than 0.98 g/ml and 0.76 g/ml reported for white yam (*Dioscorea rotundata*) by Falade and Aiyetigbo (2015) respectively. Pregelatinization and microwave radiation significantly increased ($p<0.05$) the packed and loose densities of the two starches with microwave modified sample having the highest values. The two physical modification methods seem to produce starch particles that were more compacted with little or no air space resulting in higher bulk density. Similar increase in packed and loose bulk densities of a local variety of white yam (*Dioscorea rotundata*) as a result of pregelatinization was reported by Bakre et al. (2014). Starch with higher density is preferable as they offer packaging and transportation advantages.

Hausner ratio is used to classify flow properties of sample; Carr index on the other hand is an index of flour compressibility, which gives an indirect measure of material fluidity, the higher its values, the more cohesive the material. Hausner ratio flowability classification can however be used to interpret the carr's index due to inverse relationship between material compressibility and flowability, and interrelationship between the values of these two indices (Igathinathane et al., 2010). Native starch of white cocoyam had higher carr index than native starch of red cocoyam, this suggests that white cocoyam native starch had better compressibility than red cocoyam native starch. However red cocoyam native starch with a lower hausner ratio had a better flowability than white cocoyam native starch. Pregelatinization significantly reduced ($p<0.05$) the hausner ratio and carr index of white cocoyam starch from 1.28 to 1.22 and 28.26% to 21.82% respectively indicating increase in flowability, while it significantly increased ($p<0.05$) that of red cocoyam starch from 1.19 to 1.25 and 19.15% to 25.42% respectively indicating decrease in flowability. Starches modified by microwave radiation had the highest hausner ratio and carr index with poor flowability and a very good compressibility.

Pasting properties of native and physically modified starches of white and red cocoyam varieties

Pasting properties of starch is very important because starch viscosity plays prominent role in starch utilization in food and pharmaceutical applications. The maximum viscosity (peak viscosity) of native starch of red cocoyam (4144 cP) was higher than that of native starch of white cocoyam (3687 cP) during the heating period of pasting test (Table 2). This is contrary to the observation of Oladebeye et al. (2013) who reported that white cocoyam starch had higher peak viscosity than red cocoyam starch. The final viscosity of red cocoyam native starch was also significantly higher ($p<0.05$) than that of white cocoyam native starch. There were significant differences ($p<0.05$) between the peak and final viscosity of native and modified starches.

Starches modified using microwave radiation had significantly higher ($p<0.05$) peak and final viscosity than native and pregelatinized starches, the high peak viscosity may probably be due to unrestricted swelling of the sample. In microwave radiation modified starches there was leaching out of more amylose molecules which re-associate through increased polymer-polymer hydrogen bonding during cooling resulting in the formation of a gel structure, this may have contributed to the high final viscosity of the starches (Ragaee and Abdel-Aal, 2006). The effect of pregelatinization on peak and final viscosity of cocoyam starches differed based on the variety; the peak and final viscosity of pregelatinized white cocoyam starch were higher than that of native starch, whereas the peak and final viscosity of pregelatinized red cocoyam starch were lower than that of native starch. Similar differential effect of pre-gelatinization on the flowability of the starches has been reported in this study. The difference in the structural integrity of starch granules of the two varieties of cocoyam may be responsible for this observation. Yousif et al. (2012) reported similar reduction in the peak viscosity of corn starch as a result of pregelatinization.

Table 2. Pasting properties of native and physically modified starches of white and red cocoyam (*Colocasia esculenta*) varieties

Variety	Modification	Peak Viscosity (cP)	Trough Viscosity (cP)	Breakdown Viscosity (cP)	Final Viscosity (cP)	Setback Viscosity (cP)	Peak Time (mins.)	Pasting Temperature (ºC)
White	**Native**	3687^{d}	2213^{e}	1474^{c}	3595^{f}	1382^{f}	4.87^{c}	84.05^{b}
	Pregelatinization	4087^{c}	2572^{c}	1515^{b}	4683^{c}	2111^{c}	4.87^{c}	84.05^{b}
	Microwave Radiation	4373^{a}	3135^{b}	1238^{d}	5944^{b}	2809^{b}	5.40^{a}	85.60^{a}
Red	**Native**	4144^{b}	2519^{d}	1625^{a}	4142^{d}	1623^{d}	4.53^{d}	80.70^{d}
	Pregelatinization	2896^{e}	2214^{e}	682^{f}	3715^{e}	1501^{e}	4.60^{d}	83.20^{c}
	Microwave Radiation	4414^{a}	3635^{a}	779^{e}	6639^{a}	3004^{a}	5.07^{b}	83.10^{c}

Values are means of triplicate determination; Mean values in the same column with different superscript are significantly different ($p<0.05$)

Peak viscosity gives an indication of the viscous load to be encountered during mixing while final viscosity defines the final textural quality of starchy foods.

The lowest paste viscosity value during isothermal holding is called trough or shear thinning. The difference between peak viscosity and trough, which is the breakdown viscosity, reflects the extent to which the starch granules can withstand heating and mechanical shear. The higher the breakdown viscosity the lower the ability of the sample to withstand heating and shear stress during cooking, and the lower the stability of the starch paste. The values of breakdown viscosity of native starches of white and red cocoyam were 1474 cP and 1625 cP respectively. The lower breakdown viscosity of white cocoyam native starch indicates that its starch granules had higher structural integrity and the paste may be more thermally stable than that of red cocoyam starch. This result corroborates the continuous increase in the swelling of white cocoyam starch during heating (Fig. 1) as against the reduction in the swelling of red cocoyam starch after 80 °C was reached (Fig. 2). Pregelatinization increased breakdown viscosity of white cocoyam starch while it reduced that of red cocoyam starch. Microwave radiation reduced breakdown viscosity of the starches; the reduction in the breakdown viscosity may be as a result of increase in amylose leaching during the isothermal holding period, such leached amylose can provide a network in which swollen starch granules may become embedded and be more resistant to shear (Jakakody et al., 2007). The breakdown values of red cocoyam modified starches were exceptionally low when compared with other starches; indicating starch paste of high thermal stability which may be suitable for foods that require long heating process at high temperature.

The setback values of white and red cocoyam native starches were 1382 cP and 1623 cP respectively. Setback viscosity, which shows the tendency of starch to re-associate and retrograde, indicates the extent to which viscosity increase during cooling period of pasting test. Higher setback value indicates greater retrogradation tendency. The result suggests that paste of white cocoyam starch would have lower tendency to retrograde, this may be of advantage in food metabolism and nutrient bioavailability since retrograded starch showed some resistance to digestive enzymes in human (Shittu et al., 2001). Microwave treatment significantly increased ($p<0.05$) setback values of the two starches, this shows that microwave radiation may have facilitated re association of starch molecules during cooling period leading to high final viscosity and setback values.

Microwave radiation significantly increased ($p<0.05$) peak time of cocoyam starches while pregelatinization had no significant effect ($p<0.05$) on the peak time. The pasting temperature of white cocoyam native starch was significantly higher ($p<0.05$) than that of red cocoyam native starch. This difference in pasting temperature may be as a result of difference in crystallinity packing within granules of the two starches. It is possible that crystalline regions of white cocoyam starch are tightly packed together more than that of red cocoyam starch (Alam and Hasnain, 2009), this will require higher heat energy before it can be disrupted. The pasting temperature of pregelatinized starch of white cocoyam was similar to that of native starch of white cocoyam, however pregelatinization significantly increased ($p<0.05$) pasting temperature of red cocoyam starch. Pasting temperature of the two starches modified using microwave radiation was significantly higher ($p<0.05$) than that of native starches.

Conclusion

Pregelatinization and microwave radiation affected functional and pasting properties of white and red cocoyam starches to different extent. Microwave radiation increased the swelling power, water absorption capacity, gelation, packed bulk density, loose bulk density, peak viscosity, trough viscosity, final viscosity and setback viscosity of the starches.

Pregelatinization increased the water absorption capacity, gelation, packed bulk density and loose bulk density of the starches while its effect on viscosity depends on the variety. Cocoyam starches modified using microwave radiation had high viscosity values and can find usefulness in extruded products. Pregelatinized red cocoyam starch with low viscosity values may be used for baby foods and similar products; the relatively high thermal stability of pregelatinized red cocoyam starch may also qualify it as ingredients in canned products.

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