

## Effect of drying temperature on physicochemical properties of cassava starch

N.A. Aviara<sup>1\*</sup>, J.C. Igbeka<sup>2</sup>, and L.M. Nwokocha<sup>3</sup>

<sup>1</sup>Department of Agricultural and Environmental Resources Engineering, University of Maiduguri, Maiduguri, Nigeria

<sup>2</sup>Department of Agricultural and Environmental Engineering

<sup>3</sup>Department of Chemistry  
University of Ibadan, Ibadan, Nigeria

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**A b s t r a c t.** Physicochemical and pasting properties of cassava (*Manihot esculenta* Crantz) starch dried in open air and at different temperatures in a tray dryer were determined and the effect of drying temperature on these properties was investigated. The open air dried starch had a moisture content of 12.5%, ash – 0.76%, protein – 0.85% and fat – 0.16%. With amylose content of 23.45%, pH of 5.88 and average granule size of 14.1  $\mu\text{m}$ , the starch exhibited the A-type crystalline diffraction pattern which was not affected by drying temperature in the range of 40-60°C when the starch was dried in a tray dryer. Starch degree of crystallinity decreased as the drying temperature increased in the above range. Water binding capacity and paste clarity increased, while swelling power and solubility, peak viscosity, breakdown viscosity, setback viscosity and peak time decreased significantly with increase in drying temperature ( $p < 0.05$ ). Freeze-thaw stability and pasting temperature decreased, while final viscosity increased significantly with increase in drying temperature, up to a point, and the trend reversed as the drying temperature increased further. Regression models that could be used to adequately express the relationships existing between the physicochemical and pasting properties of the starch and drying temperature were established.

**K e y w o r d s:** cassava starch, degree of crystallinity, physicochemical properties, pasting properties, drying temperature, tray dryer

### INTRODUCTION

Cassava (*Manihot esculenta* Crantz) is an important food crop in such tropical countries as Nigeria, Brazil, Thailand and Indonesia. The root of cassava is rich in starch, and in Nigeria which is the world leading producer of the crop (FAO, 2001), cassava is commercially processed into starch, 'lafun' and 'gari', a pre-gelatinized starchy staple food consu-

med in different parts of the country. The starch in its colloidal form is consumed as 'fufu' and the by-products of cassava processing are used as animal feed.

Presently, the production and exportation of cassava and cassava products to other parts of the World are being promoted in the country. Owing to the poor storability characteristics of cassava root in its fresh or unprocessed state, the produce is usually processed into more storable forms, one of which is starch. Cassava finds limited application as food and industrial raw material in its unprocessed state, but as starch it can be employed for its functional roles in a wide range of food and industrial operations.

The isolation of cassava starch from fresh roots is normally carried out in aqueous medium and the product has to be dried, packaged and supplied in a granular form. This makes drying a fundamental unit operation in starch processing.

The functionality of a starch type in a given application and, therefore, its quality is determined principally by the physicochemical properties. These include the water binding capacity, gelatinization temperature, swelling power and solubility, freeze-thaw stability, paste clarity, paste viscosity, retrogradation and gel strength (Nwokocha, 2002). Starches need to be dried under conditions that will not have any adverse effect on these properties as that could lead to impaired functionality. According to Zweifel *et al.* (2003), the drying conditions are determinants for the phase morphology of protein and starch in cooked pasta, which in turn governs the textural properties of the pasta. In order to control and establish the starch drying conditions that will yield the physicochemical property values needed for a particular application, there is the need to determine the response of

\*Corresponding author's e-mail: nddyaviara@yahoo.com

the properties to starch drying temperature. Odigboh and Mohsenin (1975) reported that drying temperature significantly affected the paste viscosity of native cassava starch, with the viscosity decreasing with increase in drying temperature. According to Metres *et al.* (1996), the viscosity of cassava starch decreased with increase in fermentation and sun drying. The swelling power, solubility and gelatinization temperature of corn starch were affected by drying conditions (Haros and Suarez, 1997) and starches isolated from low and high temperature dried pasta exhibited lower peak viscosities than those from ultra high temperature dried pasta (Yue *et al.*, 1997). Wang *et al.* (2001) reported that the peak viscosity of rice flours increased with increase in drying temperature, while the influence of drying temperature on rice starch properties was not evident. Starch from very high temperature dried durum semolina pasta had greater RVA peak viscosity and breakdown viscosity than that from high temperature dried pasta (Guller *et al.*, 2002). Zweifel *et al.* (2003) noted that high temperature drying of durum wheat pasta reduced the swelling power of the isolated starch.

Information on the effect of drying temperature on a wide range of the physicochemical properties of cassava starch appears to be scanty. This study was, therefore, undertaken to investigate the effect of drying temperature in a tray dryer on the physicochemical properties of cassava starch with a view to establishing the relationship existing between the properties and drying temperature.

#### MATERIAL AND METHODS

The cassava roots used for starch extraction were harvested from a farm at the Amina way, University of Ibadan, Ibadan, Nigeria, 20 months after planting.

The roots were peeled, thoroughly washed and crushed in a grating machine. The resultant pulp was mixed with sufficient amount of water to form slurry and this was sieved with the aid of a muslin cloth and 75  $\mu\text{m}$  mesh size sieve. The fibrous remnants were thoroughly washed and discarded, and the starch milk obtained was allowed to settle, then the supernatant was decanted. The starch was resuspended and washed several times with distilled water to remove the impurities and protein debris. The cassava starch obtained was divided into two portions and utilized as follows.

The first portion was dried in open air and used for proximate composition and pH determination, scanning electron microscopy, X-ray diffractometry and physicochemical characterization. The second portion was used for drying tests and in investigating the effect of drying temperature on the physicochemical characteristics of the starch.

The open air drying of starch was carried out by spreading the milk in thin layer on an aluminium tray. The set up was exposed to open air at ambient conditions of 27 to 30°C dry bulb temperature, 23 to 26°C dew point temperature and 50 to 78% relative humidity. The starch was allowed to dry in triplicate under those conditions until the surface cracked

and touching of the cake caused it to crumble into powder. This took up to two days and necessitated the introduction of a period of rest during the night.

The proximate composition of the starch, namely moisture, protein, fat, ash and crude fibre content, was determined using the AACC standard methods (AACC, 2000).

The amylose content of the starch was determined using the IITA method (IITA, 1995). Starch pH was determined using a WPA CD70 pH meter.

A starch granule micrograph was obtained using a JSM 35 Genie scanning electron microscope (SEM) at a 2000x magnification.

The moisture content at which starch drying commenced was determined and a laboratory model tray dryer fitted with a temperature control device was used. The dryer consisted of a drying chamber in which perforated trays were arranged vertically and placed horizontally, a plenum chamber where the heating elements were installed, a 0.374 kW axial flow fan that took in the drying air at 15.164  $\text{m s}^{-1}$ , passed it through an expanded duct and perforated plate and supplied it to pass over the product bed and the tray floor at 0.104  $\text{m s}^{-1}$ . The dryer also had an outlet through which the used air was discharged.

Air at an ambient condition of 27 to 30°C dry bulb temperature, 23 to 26°C dew point temperature and 50 to 78% relative humidity was heated to the drying temperatures of 40, 50, 55 and 60°C respectively. For an experimental run at each drying temperature, the fan was turned on and the dryer allowed to run empty for 2 h to enable it to stabilise at the specified air condition. 25 g of starch at the initial moisture content was then weighed in triplicate into drying dishes, spread into thin layer on the dishes and placed on the drying trays in the drying chamber. Change in sample weight during drying was monitored by periodical weighing using an electronic balance. Weighing of samples was carried out every 10 min for the first 1 h; every 30 min for the next 3 h; every 1 h for the next 3 h and every 2 h for the next 6 h. Weighing was continued until three consecutive readings gave identical mass. The test was then terminated and equilibrium with the drying environment was assumed to have been reached. The moisture content of the samples was determined at this point and taken to be the dynamic equilibrium moisture content.

The x-ray diffraction patterns of cassava starch samples dried in open air and at different temperatures in a tray dryer were determined using an MD10 2.04 diffractometer that produced a monochromatic  $\text{CuK}\alpha$  radiation (wave length =  $1.54 \times 10^{-9}$  m, voltage = 25 kV, current = 400  $\mu\text{A}$  and exposure time = 1 200 s). These diffraction patterns were used to classify the cassava starch and compute the degree of crystallinity. The degree of crystallinity of the starch samples was determined using the method of Wang *et al.* (2005).

Starch water binding capacity was determined using the method of Medcalf and Gillies (1965) as adapted by Nwokocho (2002) and Jyothi *et al.* (2006). Swelling power and solubility were determined using the method of Tattiyakul *et al.*

(2007). Freeze-thaw stability and paste clarity were determined using the method of Singhal and Kulkarni (1990) and Bello-Perez *et al.* (2000), respectively.

The pasting characteristics of starch were determined using a rapid visco-analyser (RVA) Series 4, Newport Scientific Ltd., Sydney, Australia, that employed the 12 min profile. The starch pasting properties obtained were peak viscosity, final viscosity, setback, breakdown, pasting temperature and peak time.

The data obtained were subjected to one way analysis of variance (ANOVA), Duncan multiple range test and regression analysis. The results were used to determine the effect of drying temperature on the physicochemical and pasting properties of cassava starch and to establish the relational models expressing the properties as a function of drying temperature.

## RESULTS AND DISCUSSION

The proximate composition of open air dried cassava starch at a moisture content of  $12.5 \pm 0.11\%$  is presented in Table 1. The moisture content was found to be within the range of 10-20% moisture content recommended for commercial native starches (Soni *et al.*, 1993a). The ash content ( $0.76 \pm 0.00\%$ ) was higher than the values reported by Ogunmola *et al.* (2001) and Nwokocha (2002) for starches from different varieties of cassava. The ash content of starch is usually dependent on the amount of minerals taken up by the source crop from the soil. The crude protein ( $0.85 \pm 0.02\%$ ) was higher, while the crude fat ( $0.16 \pm 0.00\%$ ) was comparable to the values reported by Osunsami *et al.* (1989), Agboola *et al.* (1990) and Charles *et al.* (2004). Although minor amounts of residual protein and lipid can influence gelatinization, the most dramatic effect of these components is on the flavour profile of the starch (Thomas and Atwell, 1999). The amylose content (23.45%) was within the range reported for different varieties of cassava starches (Defloor *et al.*, 1998 and Ogunmola *et al.*, 2001) and the crude fibre was found to be insignificant. The ratio of amylose to amylopectin within a given type of starch is of important consideration with respect to starch functionality

**Table 1.** Proximate composition and pH of cassava starch

Parameter	Value
Moisture content (%)	$12.5 \pm 0.11$
Ash (%)	$0.76 \pm 0.00$
Crude fiber (%)	NIL
Crude fat (%)	$0.16 \pm 0.00$
Protein (%)	$0.85 \pm 0.02$
Granule size range ( $\mu\text{m}$ )	6.5-19
Average granule size ( $\mu\text{m}$ )	$14.1 \pm 3.4$
Amylose content (%)	$23.45 \pm 0.03$
pH	$5.88 \pm 0.12$

in foods as it affects the granule architecture, gelatinization and pasting profiles, and textural attributes (Thomas and Atwell, 1999).

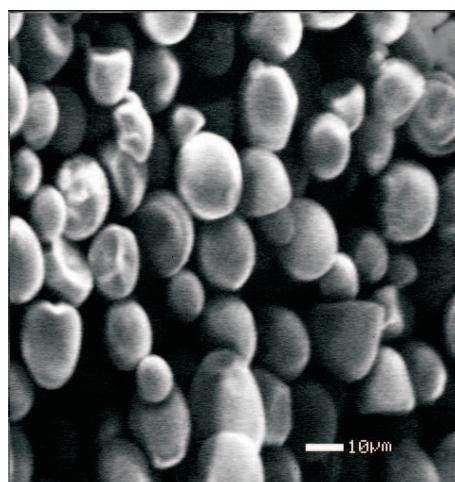
The pH of the starch dispersion was 5.88 (Table 1) and within the acceptable range for cassava starch (5-7) stated by Sanni *et al.* (2005).

The scanning electron micrograph of cassava starch is presented in Fig. 1. The starch granules are mostly spherical in shape with a few having indentations similar to an egg that has been cut at various positions. A similar observation was reported by Jane *et al.* (1994). The granule size ranged from 6.5 to 19  $\mu\text{m}$  and averaged  $14.1 \pm 3.40 \mu\text{m}$ . This lies within the same range of granule sizes reported by Defloor *et al.* (1998) for the starches of other varieties of cassava. According to Carcea and Acquisticci (1997), starch granule dimension is a characteristic peculiar to each plant species.

The initial moisture content of the wet starch was found to be  $82 \pm 0.75\%$  (d.b.). At the end of drying, the moisture content of open dried starch was 12.5% (d.b.). The dynamic equilibrium moisture content of the starch dried in a tray dryer decreased with increase in drying temperature and had a value of 7.70, 6.85, 6.40 and 1.70% (d.b.) for starch dried at 40, 50, 55 and 60°C, respectively.

Three general crystalline diffraction patterns have been identified in native starches by the method of x-ray diffractometry. These have been used to classify starch as first the A-type typical of cereal starches with major peaks at 15, 17, 18 and 23° (Manek *et al.*, 2005). The second is the B-type normally obtained in roots and tuber starches with a single strong and sharp peak at 17° and two identifiable peaks at 22 and 24° (Riley *et al.*, 2006). The third is the C-type pattern which is a mixture of the A and B types and typical of bean starches (Thomas and Atwell, 1999).

The cassava starch exhibited the A type crystalline diffraction pattern with major peaks at 15, 17, 18 and 23° and this was not affected by drying temperature (Fig. 2). Increase



**Fig. 1.** Scanning electron micrograph (SEM) of cassava starch (magnitude-2000x).

in drying temperature, however, caused the diffraction peaks to become stronger and the degree of crystallinity to decrease. Khunae *et al.* (2007) reported that the heat moisture treatment of rice starch did not alter its A-type diffraction pattern.

The mean values of the physicochemical properties of cassava starch obtained at different drying temperatures are presented in Table 2. The results show that drying temperature affected the physicochemical properties.

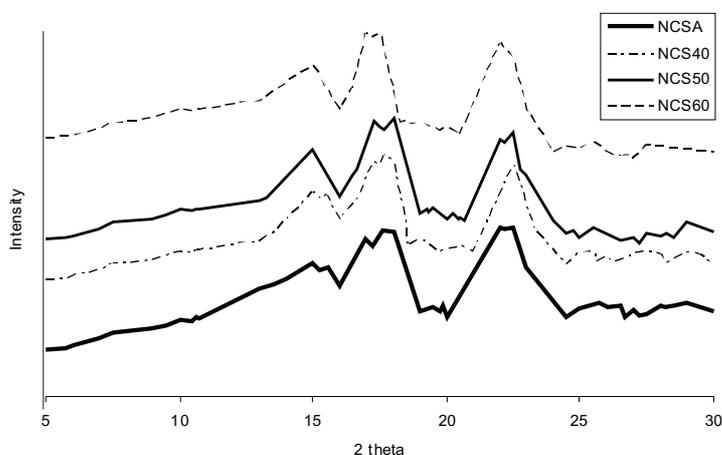
The water binding capacity of open air dried starch was significantly higher than that of starch dried at different temperatures in thin layer using a tray dryer, at 5% level. The starch dried at different temperatures, however, exhibited significant differences in water binding capacity at 5% level. Water binding capacity is a measure of the strength of starch intergranular bond. Low water binding capacity is attributable to tight association (Soni *et al.*, 1993b), while high water binding capacity is indicative of a loose association of native starch polymers or low lipid content (Nwokocho and Ogunmola, 2005). The water binding capacity of cassava starch dried in a tray dryer increased with increase in drying temperature within the range of 40–60°C. This implies that increase in drying temperature may have resulted in some weakening of the starch granule integrity. The relationship

existing between water binding capacity and starch drying temperature was found to be linear and could be expressed with the following equation:

$$\omega = 0.117T + 62.968, \quad R^2 = 0.949 \quad (1)$$

where:  $\omega$  – water binding capacity (%),  $T$  – drying temperature (°C),  $R^2$  – coefficient of determination.

The swelling power of open air dried starch was significantly higher than that of starch dried at different temperatures in thin layer using a tray dryer, at 5% level. The amylose and amylopectin components of starch granule are arranged inside in such a way that adjacent linear chains of the amylopectin molecules associate in a radial fashion to form crystalline micelles. In between the micelles are amorphous regions occupied by amylose molecules. The strength of the micellar networks decides many of the properties of starch granules. When starch is heated in an excess water system, the granules imbibe water through the amorphous region in a reversible manner and the amount of water imbibed increases with temperature until the gelatinization temperature is reached and the starch swells irreversibly with a loss of crystalline order. This irreversible swelling is accompanied by a significant uptake of water resulting in granule expansion and leaching of amylose into the solution (Pomeranz, 1990).



**Fig. 2.** X-ray diffraction pattern of native cassava starch dried at different temperatures; NCSA – dried in open air, NCS40 – dried at 40°C, NCS50 – dried at 50°C and NCS60 – dried at 60°C.

**Table 2.** Physicochemical properties of cassava starch dried at different temperatures

Drying temperature (°C)	Water binding capacity (%)	Swelling power (g g <sup>-1</sup> )	Solubility (%)	Freeze-thaw stability (%)	Paste clarity (%)
Open air	73.50±0.13a	19.54±0.73a	9.00±0.30a	18.50±0.00b	66.35±0.75c
40	67.80±0.21e	18.43±0.20b	8.60±0.40a	18.40±0.03b	54.57±0.15d
50	68.60±0.15d	17.01±0.00c	5.63±0.21b	19.75±0.17a	66.70±0.82c
55	69.22±0.03c	16.72±0.57c	4.50±0.05c	14.80±0.01c	70.60±0.01b
60	70.23±0.14b	13.27±0.35d	3.00±0.07d	9.20±0.02d	72.40±0.58a

Numbers in columns denoted by the same letter are not significantly different at 5% level.

The solubility of open air dried starch was not significantly different from that of starch dried at 40°C in tray dryer. The swelling power and solubility of the starch dried at different temperatures differed significantly, but the swelling power values of the 50 and 55°C dried starch were not significantly different at 5% level. This could be attributed to the presence of similar bonding forces within the granules of the starches dried at these temperatures. The swelling power decreased with increase in drying temperature in a relationship that was found to be polynomial of the second order and could be represented by the following equation:

$$\varphi = -0.0163T^2 + 1.3922T - 11.243, \quad R^2 = 0.932, \quad (2)$$

where:  $\varphi$  – swelling power ( $\text{g g}^{-1}$ ).

The starch solubility decreased with increase in drying temperature in a linear relationship that could be expressed using the following equation:

$$\zeta = -0.2773T + 19.645, \quad R^2 = 0.998, \quad (3)$$

where:  $\zeta$  – solubility (%).

The increase of solubility with swelling power showed that a part of the linear component of the starch was involved in the micellar network while the rest was free from entanglement and preferentially solubilized (Soni *et al.*, 1993b).

The freeze-thaw stability and paste clarity of open air dried starch were not significantly different from those of starch dried in a tray dryer at 40 and 50°C, respectively, at 5% level of significance. The freeze-thaw stability of the starch dried at different temperatures as measured by the level of syneresis, differed significantly at 5% level. A similar result was obtained for paste clarity. The results reveal that syneresis in the starch increased with increase in drying temperature up to a point, and thereafter decreased with further increase in temperature. This implies that the freeze-thaw stability of the starch decreased with increase in drying temperature up to a point and increased with further increase in drying temperature. This could be attributed to a sort of thermal breakdown of starch macromolecules when drying took place at temperatures above 55°C, which resulted in reduced reassociation of the macromolecules. The relationship

existing between percentage syneresis and drying temperature was found to be polynomial of the second order. The relationship could be represented by the following equation:

$$\tau = -0.0553T^2 + 5.0504T - 95.094, \quad R^2 = 0.988, \quad (4)$$

where:  $\tau$  – percentage syneresis (%).

Paste clarity increased with increase in drying temperature and could be due to reduction in the reassociation of solubilized molecules with increase in drying temperature. The relationship existing between starch paste clarity and drying temperature was found to be polynomial of the second order and could be expressed with the following equation:

$$\rho = -0.0331T^2 + 4.2011T - 60.602, \quad R^2 = 1.000, \quad (5)$$

where:  $\rho$  – paste clarity (%).

Pasting properties of starch are used in assessing the suitability of its application in food and other industrial products. The most important pasting characteristic of granular starch dispersion is its viscosity. High paste viscosity suggests suitability as a thickening agent in food (Rapaille and Vanhemelrijck, 1999) and as a finishing agent in textile and paper industries (Nwokocha, 2002).

The pasting curves of cassava starch dried in open air and in thin layer using a tray dryer at different temperatures are presented in Table 3. The results show that peak viscosity of open air dried starch was significantly lower than that of starch dried at different temperatures at 5% level. The peak viscosity of starch dried at different temperatures differed significantly at 5% level and the peak viscosity of cassava starch dried in a tray dryer decreased with increase in drying temperature indicating a decrease in the leaching of amylose from the granules (Gomes *et al.*, 2004). The relationship existing between the starch peak viscosity and drying temperature was found to be polynomial of the third order and could be expressed with the following equation:

$$v_p = -0.0497T^3 + 7.1613T^2 - 341.43T + 5861.9, \quad R^2 = 1.000, \quad (6)$$

where:  $v_p$  – peak viscosity, RVU.

**Table 3.** RVA pasting properties of cassava starch dried at different temperatures

Drying temperature (°C)	Peak viscosity	Final viscosity	Breakdown viscosity	Setback viscosity	Pasting temperature (°C)	Peak time (min)
Open air	397.55±0.03e	250.07±0.11a	249.13±0.02d	101.65±0.00a	72.80±0.01c	4±0.00a
40	481.92±0.07a	241.33±0.13b	307.83±0.10a	67.25±0.06c	72.90±0.01b	3.87±0.01c
50	481.08±0.07b	250.08±0.16a	300±0.00b	69±0.02b	72.80±0.02c	4±0.00a
55	477.25±0.05c	250.00±0.08a	290±0.034c	62.75±0.05d	73.50±0.01a	4±0.00a
60	421.50±0.04d	236.42±0.01c	241.67±0.02e	56.58±0.11e	73.50±0.01a	3.93±0.02b

Explanation as in Table 2.

The final viscosity of open air dried starch was significantly higher than that of starch dried at 40 and 60°C, respectively, but did not differ significantly from that of starch dried at 50 and 55°C at 5% level of significance. The variation of starch final viscosity with drying temperature shows that it initially increased with increase in temperature and, after a point, remained constant within a range and then decreased with further increase in temperature. This shows that increasing the drying temperature of starch improved the stability of the paste (Gomes *et al.*, 2004). The relationship existing between final viscosity and starch drying temperature could be expressed with the following equation:

$$v_f = -0.0105T^3 + 1.4674T^2 - 66.962T + 1245.8, \\ R^2 = 1.000, \quad (7)$$

where:  $v_f$  – final viscosity, RVU.

The breakdown viscosity of open air dried starch was significantly lower than that of starch dried at 40, 50 and 55°C, but higher than that of starch dried at 60°C at 5% level. Significant differences were found in the breakdown viscosity of starch dried at different temperatures at 5% level. The variation of breakdown viscosity with drying temperature shows that it decreased with increase in temperature in a relationship that was found to be polynomial of the third order. This relationship could be expressed using the following equation:

$$v_b = -0.0343T^3 + 4.8885T^2 - 231.68T + 3948.9, \\ R^2 = 1.000 \quad (8)$$

where:  $v_b$  – breakdown viscosity, RVU.

The setback viscosity of open air dried starch was significantly higher than that of starch dried at different temperatures at 5% level. The setback viscosity of starch dried at different temperatures differed significantly at 5% level and it varied with drying temperature according to a polynomial relationship of the second order. This relationship could be represented by the following equation:

$$v_s = -0.0643T^2 + 5.8665T - 64.457, \\ R^2 = 0.979 \quad (9)$$

where:  $v_s$  – setback viscosity, RVU.

The pasting temperature of open air dried starch was significantly lower than that of starch dried at 40, 55 and 60°C and did not differ significantly from that of the starch dried at 50°C at 5% level. There was no significant difference between the pasting temperatures of starch dried at 55 and 60°C, while significant differences were found between those of the 40 and 50°C dried starch at 5% level. The variation of pasting temperature with starch drying temperature shows that the pasting temperature initially decreased with

increase in temperature, and then increased. The relationship existing between the pasting temperature of cassava starch and drying temperature was found to be polynomial of the third order, which could be expressed with the following equation:

$$T_p = -0.0012T^3 + 0.184T^2 - 9.25T + 225.3, \\ R^2 = 1.000, \quad (10)$$

where:  $T_p$  – pasting temperature (°C).

The peak time of open air dried starch was significantly higher than that of starch dried at 40 and 60°C, but not significantly different from that of the 50 and 55°C dried starch, at 5% level. There were no significant differences between the peak times of starch dried at 50 and 55°C, while the difference between that of 40 and 60°C dried starch was significant at 5% level and it shows that the peak time of cassava starch varied with drying temperature in a relationship that was found to be polynomial of the second order. This relationship could be represented by the following equation:

$$t_p = -0.001T^2 + 0.1068T + 1.2562, \\ R^2 = 0.995, \quad (11)$$

where:  $t_p$  – peak time (min).

## CONCLUSIONS

1. The open air dried cassava starch at 12.5% moisture content and pH of 5.88, had an ash content of 0.76, 0.85, crude protein, 0.16% crude fat and 23.45% amylose.
2. The starch granules were spherical in shape and of an average size of 14.1  $\mu\text{m}$ .
3. The starch exhibited the A-type crystalline diffraction pattern which was not affected by drying temperature in the range of 40-60°C.
4. The starch degree of crystallinity decreased with increase in drying temperature.
5. The physicochemical and pasting properties of the starch were significantly affected by drying temperature. Water binding capacity and paste clarity increased, while swelling power and solubility, peak viscosity, breakdown, set back and peak time decreased with increase in drying temperature. Freeze-thaw stability and pasting temperature decreased up to a point and then increased, while final viscosity increased up to a point and decreased with further increase in drying temperature in a tray dryer.
6. The relationships existing between the physicochemical and pasting properties of the starch and drying temperature could be expressed with regression equations. These equations are applicable to starch dried in a tray dryer in the drying temperature range of 40-60°C.

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