UUPhysicochemical Properties of Sorghum (*Sorghum Bicolor* L. Moench) Starch as Affected by Drying Temperature

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ABSTRACT

Starch was isolated from white sorghum grains and its proximate composition determined. Scanning electron micrograph and x-ray diffraction pattern of the starch were obtained. The effect of drying temperature in a tray dryer on physicochemical and pasting properties of isolated starch was investigated.

The open air dried starch had a composition of 10.73% moisture, 0.30% ash, 1.06% protein and 1.07% fat. Amylose content was 21.08% and the average granule size and pH were 18.59μm and 5.45, respectively. The starch exhibited the A-type crystalline diffraction pattern, which was not altered by drying in a tray dryer at the temperature range of 40 to 60^{o}C. Water binding capacity and swelling power of the open air dried starch were not significantly different from those of starch dried at 40^{o}C ($p<0.05$). Water binding capacity increased from 79.63 to 88.5%, while swelling power, solubility and percentage of syneresis decreased from 12.01 to 8.96g/g, 7.08 to 2.85% and 14.00 to 10.80%, respectively as the drying temperature increased from 40 to 60^{o}C. Paste clarity was low (22.50 – 26.20%) but increased with increase in drying temperature up to 50^{o}C and decreased with further increase in temperature. Pasting properties of open air dried starch differed significantly from those of starch dried at different temperatures. Peak viscosity decreased from 398.75 to 325.25 RVU as the drying temperature increased from 40 to 60^{o}C. Setback viscosity increased with increase in drying temperature up to a point and decreased with further increase in temperature. Final and breakdown viscosities as well as pasting temperature and peak time had polynomial relationships of the third order with drying temperature.

Regression equations that could be used to adequately express the relationships existing between the physicochemical and pasting properties of sorghum starch and drying temperature were established. These models could be used to select the drying temperature that would yield starch of desired physicochemical properties for a functional application.

Keywords: White sorghum starch, degree of crystallinity, physicochemical properties, pasting properties, drying temperature, tray dryer.

1. INTRODUCTION

Sorghum (*Sorgum bicolor* L. Moench) belongs to the Gramineae family of crops and the Andropogoneae tribe. The species sorghum bicolor L. Moench is cultivated throughout the intertropical zone of Africa. In Nigeria, it is mainly grown in the region lying between the middle belt and semi-arid zones. Sorghum grain has a high starch content of 60 – 80% (McNell *et al*., 1975). Sorghum starch finds uses in the food, chemical, pharmaceutical and textile industries. As a result of this, interest in sorghum starch has recently been on the increase (Elkhalifa *et al*., 2004). The starch is normally isolated in aqueous medium and dried, packaged and supplied in granular form. This made drying a fundamental unit operation in starch processing. Due to the physical and chemical properties of starch, it is sensitive to heat when moist. Therefore, it requires that drying should be carried out under conditions that will not affect the functionality.

The functionality of a starch type in a given application and therefore, its quality is determined principally by the physicochemical properties and these properties include water binding capacity, gelatinization temperature, swelling power and solubility, freeze-thaw stability, paste clarity, paste viscosity, retrogradation and gel strength (Adebowale and Lawal, 2002). Elkhalifa *et al.* (2004) noted that fermentation of sorghum flour increased the solubility of its starch, decreased the water binding capacity and had no effect on the freeze-thaw stability and paste clarity. Adebowale *et al*. (2005) reported that heat moisture treatment and annealing of red sorghum starch increased the water binding capacity and pasting temperature but decreased the swelling power and solubility. According to Zweifel *et al*. (2003), the drying conditions are determinants for the phase morphology and structure of protein and starch in cooked pasta, which in turn, governs the textural properties of the pasta. Odigboh and Mohsenin (1975) reported that the drying temperature of isolated starch significantly decreased the paste viscosity of native cassava starch and this was confirmed by the findings of Aviara *et al.* (2010). Swelling power, solubility and gelatinization temperature of corn starch were reported to have been affected by drying conditions (Haros and Suarez, 1997) and starches isolated from low and high temperature dried pasta were found to exhibit lower peak viscosity than those from ultra high temperature dried pasta (Yue *et al*., 1999). Wang *et al*. (2001) reported that the peak viscosity of rice flour increased with increase in drying temperature, while the effect of drying temperature on the starch properties was not evident.

In order to establish and control the drying conditions to yield starch with physicochemical property values needed for a particular application, there is the need to determine the response of these properties to starch drying temperature. Information on the effect of drying temperature on the physicochemical properties of sorghum starch appears to be scanty; therefore, this study was undertaken to investigate the changes in these properties of the starch due to drying temperature. The properties studied include water binding capacity, swelling power and solubility, freeze-thaw stability, paste clarity and pasting properties.

2. MATERIALS AND METHODS

2.1 Isolation of sorghum starch

The grains of white sorghum from which starch was isolated were supplied from Kano and procured from an outlet in Ibadan, Nigeria.

The grains were cleaned and soaked in warm water at the temperature of 35^{o}C for 6h (Aviara, 2010) to loosen the starch granules from the protein matrix and inhibit microbial activity. The soaked grains were washed and crushed in a disc attrition milling machine (wet milling) and the paste obtained was mixed with sufficient amount of water to form slurry. The slurry was sieved through a muslin cloth and 75μm mesh size sieve. The residue was thoroughly washed and discarded, and the starch milk collected was washed several times in aqueous alkaline medium to remove the encapsulating protein matrix and allow the starch to settle. The supernatant was decanted and the starch milk obtained was washed several times with decanting of water to remove impurities and protein residue. The sorghum starch obtained was divided into two portions and utilized in further investigations.

The first portion was dried in open air and used for proximate composition and pH determination and scanning electron microscopy. The second portion was used to obtain starches dried in thin layer in a tray dryer at the temperatures of 40, 50, 55 and 60^{o}C respectively. The x-ray diffraction pattern and physicochemical properties of the open air dried starch and those dried at different temperatures were determined.

2.2 Determination of proximate composition

The moisture content of starch was determined by weighing using the AACC standard method, (AACC, 2000). Five grams of open air dried starch was weighed in triplicate into previously dried and weighed crucibles. These were placed in an oven and dried at 105^{o}C to constant weight. The crucibles and contents were allowed to cool in a dessicator and weighed. The moisture content was calculated as percentage loss in starch weight reported to dry basis.

The ash content was determined using 2g of the starch samples used in moisture content determination. This was ashed in a muffle furnace at 650^{o}C until a constant weight of ash was obtained. The ash content was calculated as weight of ash obtained per weight of starch (dry basis) expressed in percentage. This was repeated three times and the average value was determined.

The crude protein content was determined using 1.5g of starch weighed into a Kjeldahl long neck digestion flask. 10ml of concentrated H_2SO_4 was added and the mixture swirled to wet the starch. A piece of Kjeldahl tablet was added and the sample was digested over a heating mantle with occasional swirling until a clean clear solution was obtained. Heating was continued for 30 mins after which the flask and its content was allowed to cool. The content of the flask was transferred into a 500ml Quickfit distillation flask. The Kjeldahl flask was filled to 2/3 of its volume with distilled water, rinsed thoroughly and the water transferred into the Quikfit distillation flask. The rinsing and transferring of water was repeated and the volume made up to 250ml. Some pieces of zinc granules were added and 70 ml of 40% NaOH solution was ran down continuously by the side into the flask. The distillation equipment was assembled with a delivery adaptor dipping into 25ml of 2% boric acid solution containing three drops of screened methyl red indicator. It was

noted that during the distillation process, the green color of boric acid indicator and indicator mixture turned pink. The ammonia liberated into the boric acid solution was titrated with standard hydrochloric acid until the pink color turned purple.
The protein content of starch was determined using the following equation:

$$\text{Percentage protein} = \frac{(V \times N \times 1.4 \times F)}{W \times \%DM} \quad (1)$$

where
V = volume of HCl used (ml)
N = molarity of the HCl solution
F = conversion factor from Nitrogen to protein: 6.25
W = weight of starch used (g)
%DM = percentage of dry matter
A blank determination of protein content following the above procedure without starch sample was also carried out.

The crude fiber content was obtained using 2g of starch sample weighed into a 500ml conical flask and 100ml of digestion reagent added. This was boiled on an electric heater and allowed to simmer under reflux for 40 mins with the content of the flask swirled from time to time to ensure an even digestion. The flask was removed and cooled under a cold water tap. The content was filtered through a Whatman No.4 filter paper and washed several times with hot water. The paper was opened and the residue transferred into a porcelain dish. This was dried for 2 h at 100^{o}C, cooled for 3 mins in a desiccator, weighed and ashed in a muffle furnace at 600^{o}C for 30 mins. The residue was cooled in a desiccator and reweighed. The crude fiber content of starch was taken as the loss in weight on ashing, expressed as a percentage of the starch sample weight.

The fat content of the starch was determined by carrying out the ether extraction using a soxhlet extractor.

The amylose content of starch was determined using the method of IITA (1995). 100mg of starch sample (db) was weighed into boiling tubes. 1ml of ethanol (95%) was added and the mixture swirled. 9ml of 1M NaOH solution was then added. The tubes and their contents were heated in boiling water bath for 10 minutes to gelatinize the starch. The tubes were removed, cooled and their contents transferred into 100ml standard volumetric flask. The volume was made up to mark with distilled water and the flask shaken thoroughly and allowed to stand. 2.5ml of the gelatinized starch solution was poured into a 50ml volumetric flask and 0.5ml of 1M acetic acid added to acidify the solution. 1ml of iodine solution was then added. The flask was shaken and left for 20 minutes for color to fully develop. The absorbance of the solution was monitored using a SpectrumLab 752S uv visible spectrophotometer at 620 nm. Iodine solution of the same concentration as that used in color development was used with distilled water in the reference cell.

$$\text{Amylose content} = \frac{(\%\ \text{amylose of standard} \times \text{absorbance of sample})}{\text{absorbance of standard}} \quad (2)$$

2.3 Starch pH

2g of starch (db) was dispersed in 20ml of distilled water in long neck 50ml beaker for 1h at room temperature. The pH was, thereafter, measured using a WPA CD70 pH meter. This was replicated thrice and the average pH of the starch was determined.

2.4 Starch Microscopy

Granule micrographs were obtained using a JSM 35 Genie Scanning Electron Microscope (SEM). The starch was sprinkled onto a double-backed adhesive carbon tab stuck to a circular aluminium stub. The aluminium stub with the starch sample on it was placed in the vacuum chamber of a polaron PS3 sputter coater. After attaining a vacuum of 0.1 to 0.2 torr and plasma current of 42 mA, the gold coating process was carried out for 140s. The stub with gold coated starch was then placed in the SEM chamber which was evacuated before the electron beam was turned on. A 10kV, 2.05A setting was used for the subsequent imaging work on starch, the aperture size being fixed at 3. Granule size was then analyzed using a BT1600 Image Analyzer.

2.5 Thin layer drying of starch

The initial moisture content of the starch used in carrying out thin layer drying was determined in triplicate and the average value was recorded. The drying equipment used was a Laboratory model tray dryer fitted with a temperature control device. It 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 0.374kW axial flow fan that supplied the drying air at a rate of 0.238 m^3/s and an outlet for discharging the used air.

Air at the ambient condition of 27 to 38^oC dry bulb temperature, 23 to 26^oC dew point temperature and 50 to 78% relative humidity, was heated to the drying temperatures of 40, 50, 55 and 60^oC respectively. For an experimental run at each drying temperature, the fan was turned on and the dryer allowed running empty for 2 h to enable it to stabilize at the specified air condition before the test began. 25g of starch at the initial moisture content was weighed in triplicate into drying dishes and placed on the drying trays in the drying chamber with the fan running. Change in sample weight was monitored by weighing periodically using an electronic balance. Weighing of samples was carried out as follows, every 10 minutes for the first 1h; every 30 minutes for the next 3h; every 1h for the next 3h; every 2h for the next 6h. Weighing was continued until three consecutive readings gave identical weights. 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 average dynamic equilibrium moisture contents of the starch at the different drying temperatures were recorded.

2.6 X-ray diffraction

X-ray diffraction measurements were carried out on samples of open air dried starch and those dried in thin layer in a tray dryer at different temperatures, using an MD10 2.04 diffractometer that produced a monochromatic CuKα radiation (wave length = 1.54Å, voltage = 25kV, current 400μA and exposure time = 1200s).

The x-ray diffraction patterns of open air and tray dryer dried starches were used to classify the sorghum starch and compute the degree of crystallinity. The degree of crystallinity of the starch samples was determined using the method reported by Wang *et al*. (2005). This involved the connection of the peak baselines of the diffraction pattern with a smooth curve. The upper diffraction peak area and the total area over the diffraction angle, 2θ of 5 to 30^o were graphically determined and the ratio of the upper area to the total diffraction pattern area was taken as the degree of crystallinity and expressed as follows.

$$Crystallinity,\% = \frac{A_c}{(A_c + A_a)} \quad (3)$$

where
A_c = area of the crystalline region of the X-ray diffractogram
A_a = area of the amorphous region of the X-ray diffractogram.

2.7 Physicochemical and pasting properties

The physicochemical properties of open air dried starch and those dried in a tray dryer at different temperatures were determined in triplicate as follows:
Water binding capacity was determined using the method of Medcalf and Gilles (1965) as reported by Nwokocha (2002). 2g of starch was weighed into a centrifuge tube and 25ml of distilled water was added. The tube was corked and agitated on a shaker for 1h at room temperature. It was then removed and centrifuged at 6000 rpm for 10 minutes. The water released was decanted and the tube drained at a tilt angle of 45^o for 5 minutes. The tube was weighed and the amount of bound water was determined. The water binding capacity was expressed as percentage of bound water to starch by weight (db).

Swelling power and solubility of starch was determined using the method of Konik *et al*. (1993). About 0.5g of starch was weighed into centrifuge tube and 25ml of distilled water added. This was immersed in a water bath at the temperature of 85^{o}C for 30 minutes. The slurry was stirred thoroughly with glass rod throughout the period of heating. The tube was removed and cooled to room temperature and then centrifuged at 6000 rpm for 15 minutes. The supernatant was carefully sucked into a weighed crucible, weighed and evaporated over a steam bath and dried in an oven at 120^{o}C for 4 h. The paste was weighed and the swelling power calculated as weight of sedimented paste per gram of starch (db). The difference in weight after drying the supernatant gave the weight of the soluble components. Percentage solubility was calculated as weight of soluble components per weight of starch (db).

The method of Singhal and Kulkarni (1990) was followed in determining the freeze – thaw stability of starch. 0.5g of starch (db) in 10ml of distilled water in test tube was heated at 95^{o}C for 30 minutes with constant stirring. This was then removed, cooled with continuous stirring, transferred into a weighed centrifuge tube and subjected to freezing for 18 h and thawing for 3 h. It was then centrifuged at 6000 rpm for 10 minutes and the percentage of water separated to the weight of paste was taken as a measure of the freeze – thaw stability.

Paste clarity was determined using the method of Singhal and Kulkarni (1990). This involved the measurement of light transmitted by starch of a known concentration at 660 nm using a uv

visible spectrophotometer. Starch paste was prepared by heating 0.2g of starch in 20ml of distilled water in cap – sealed tubes in a water bath at 95^{o}C (with occasional shaking to avoid the formation of lumps) for 30 minutes. The light transmittance was determined with distilled water used in the reference cell.

The pasting characteristics of starch were determined using a Rapid Visco Analyser (RVA) Series 4, Newport Scientific Ltd, Sydney, Australia. 3g (db) of starch sample in 25ml of distilled water was shaken well and poured into a canister. A paddle was placed inside the canister and centrally positioned onto the paddle coupling and the set up was inserted into the RVA machine. The measurement circle was initiated by actuating the instrument motor. The profile was observed as it ran on the monitor of the computer that was connected to the instrument. The 12 minutes profile was used. The time – temperature regimes used were: idle (starting) temperature of 50^{o}C for 1 min, heating from 50^{o}C to 95^{o}C in 3 minutes, 45s, holding at 95^{o}C for 2 minutes 30s, cooling back to 50^{o}C over 3 minutes 45s and maintaining the 50^{o}C for 2 minutes. The starch pasting properties namely viscosity, final viscosity, setback viscosity, breakdown viscosity, pasting temperature and peak time were obtained.

2.8 Statistical Analysis

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 properties of sorghum starch and to establish the relationship between starch properties and drying temperature treatment.

3. RESULTS AND DISCUSSION

3.1 Proximate composition

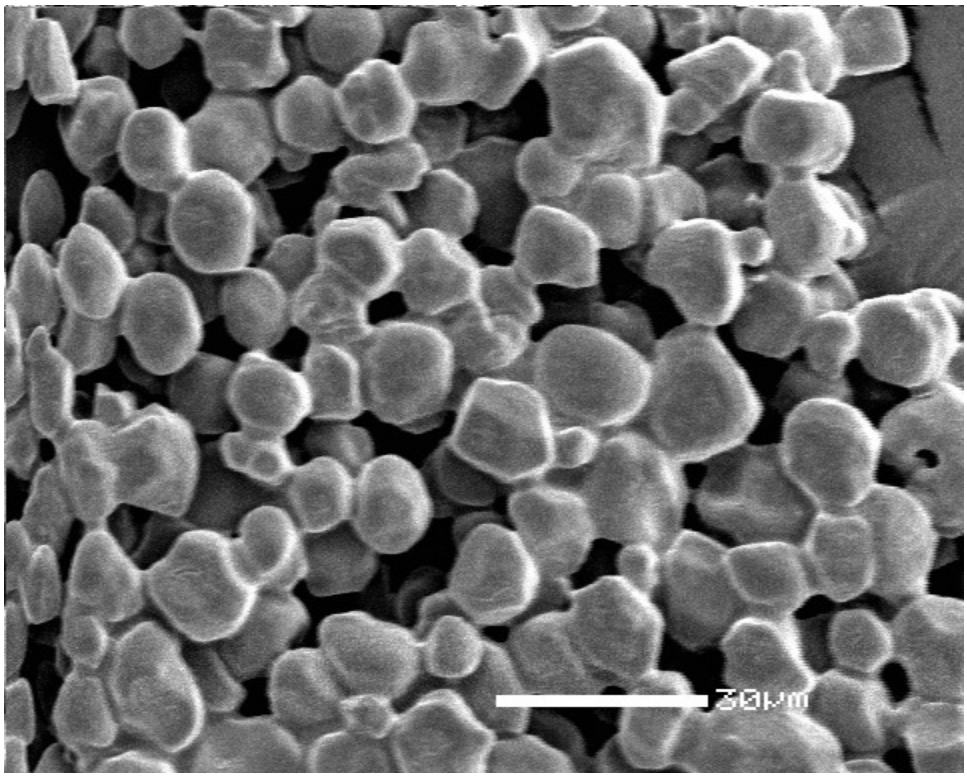
The proximate compostion and pH of the open air dried sorghum starch are presented in Table 1. The moisture content was found to be 10.73 ± 0.211% and within the range of 10 – 20% for commercial native starches (Soni *et al.,* 1993). The ash content (0.30 ± 0.011%) was lower than that of sweetsop and soursop starches (Nwokocha and Williams, 2009), higher than that of *treculia africana* starch (Nwokocha and Ogunmola, 2005) and cocoyam starch and within the same value as that of cassava starch (Nwokocha *et al.,* 2009). The protein and fat contents of 1.06 ± 0.004% and 1.07 ± 0.001% respectively were higher than those of cassava starch (Ogunmola *et al.,* 2001), but lower than those of maize starch (Soni *et al.,* 1990. The amylose content (21.08%) was within the range reported for the starches of different varieties of sorghum (Carcea *et al.,* 1992) but lower than the values reported by Chanapamokkhot and Thongngam (2007) and Boudries *et al.* (2009). The crude fiber in starch was found to be insignificant and the pH was 5.45.

Table 1. Proximate composition and pH of open air dried sorghum starch

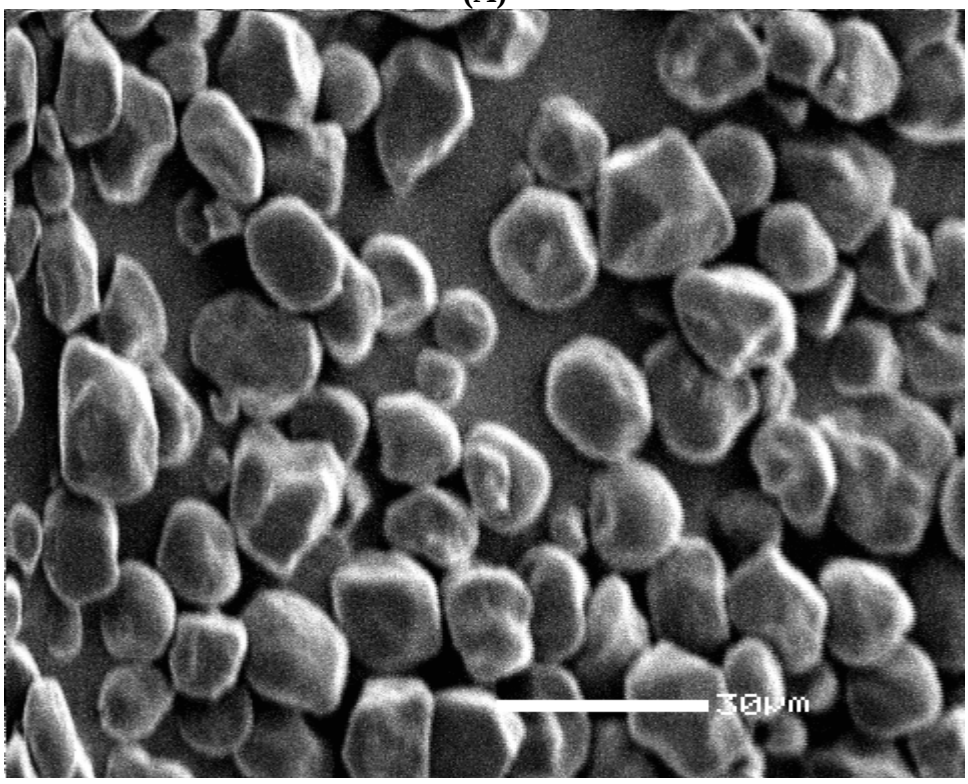
Parameter Value
Moisture content, % 10.73±0.211
Ash, % 0.3±0.011
Crude fiber, % NIL
Crude fat, % 1.07±0.001
Protein, % 1.061±0.004
Amylose content, % 21.08±0.01
pH 5.45±0.105

3.2 Scanning Electron Microscopy

The scanning electron micrographs of sorghum starch dried in open air (A) and in a tray dryer at 60^{o}C (B) are presented in Figure 1. The starch granules are irregular in shape with smoothened edges and have unimodal distribution. An examination of the micrographs A and B in Figure 1 shows that drying sorghum starch in the temperature range employed did not change the shape and surface features of the granules.



(A)



(B)

Figure 1. Scanning Electron Micrographs (SEM) of sorghum starch, A: open air dried and B: dried in a tray dryer at 60^{o}C, 1600X

The granule size analysis of the starch dried in open air (mildest drying condition) and at 60^{o}C in a tray dryer (harshest drying condition) is presented in Table 2. The starch dried in open air has a granule size range of 5.44 – 19.02μm, average granule diameter of 11.27μm, L/D of 1.18 and roundness of 0.79, while the starch dried at 60^{o}C was of 5.42 – 19.39μm granule size range, 11.62μm average granule diameter, L/D of 1.14 and roundness of 0.81. These values are within the range reported by Jane *et al.* (1994) and Choi *et al.* (2004). The granule size distributions of the starch dried in the two conditions stated above are presented in Figure 2. This figure shows that the drying of starch at the higher temperature of 60^{o}C influenced the granule size distribution. This could be attributable to a possible on set of swelling that must have occurred when the granules at the high initial moisture content were exposed to the high temperature, which was quite close to starch gelatinization temperature. Copeland *et al.* (2009) noted that the gelatinization temperature of starches generally lies between 60 and 80^{o}C.

Table 2. Granule size analysis of sorghum starch dried in open air and at 60^{o}C

Particle characteristics	open air dried starch	starch dried at 60^{o}C
Particle Number	122	125
Maximum diameter (μm)	19.02	19.39
Minimum diameter (μm)	5.44	5.42
Average diameter (μm)	11.27	11.62
Length/Diameter, L/D	1.18	1.14
Roundness	0.79	0.81

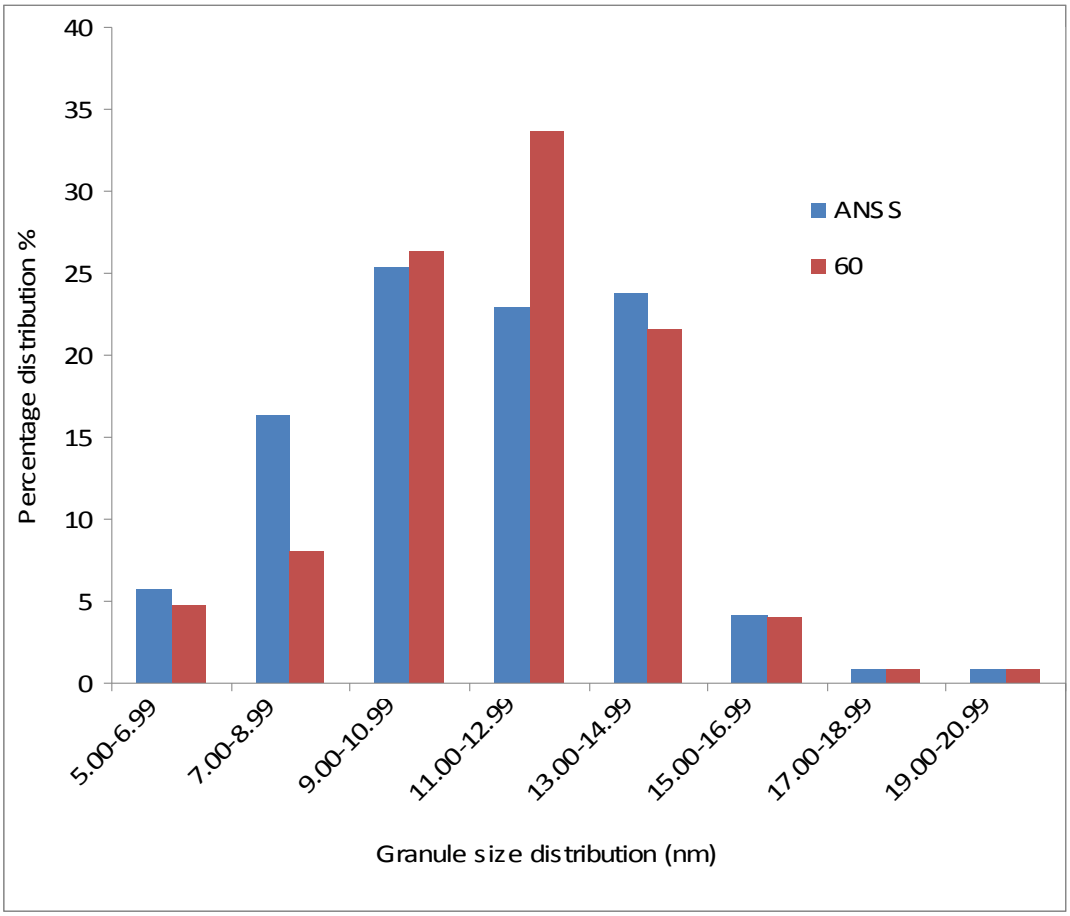


Figure 2. Granule size distribution of sorghum starch dried at different temperatures: ANSS – open air dried starch, 60 – starch dried at 60^{o}C

3.3 Starch drying

The initial moisture content of the starch was found to be 84.5% (db). The dynamic equilibrium moisture content obtained at the end of drying decreased with increase in drying temperature and had a value of 8.70, 7.60, 5.80 and 4.18% (db) for sorghum starch dried at 40, 50, 55 and 60^{o}C, respectively.

3.4 X-ray diffraction pattern

The X-ray diffraction patterns of sorghum starch presented in Figure 3 shows that the starch exhibited the A-type crystalline diffraction with major peaks at 15^{o}, 17^{o}, 18^{o} and 23^{o}. This diffraction pattern is typical of cereal starches (Manek *et al.,* 2005) and in agreement with the finding of Abd Allah *et al.* (1987). The same peaks were found for starches from sorghum varieties grown in the Sahara of Algeria (Boudries *et al.,* 2009) and Korean waxy sorghum starch (Shin *et al.,* 2004). The A-type crystalline pattern of the starch was not altered by drying temperature. Khunae *et al.* (2007) reported that the heat moisture treatment of rice starch did not alter its A-type diffraction pattern. An opposite observation was however made on slowly digestible non-pasted granular sweet potato starch (Shin *et al.,* 2005).

The relative crystallinity of sorghum starch dried at different temperatures is presented in Table 3. The physical and chemical properties of a starch type are strongly dependent on its degree of crystallinity (Frost *et al.,* 2009). Increase in starch drying temperature caused the degree of crystallinity to decrease from 36.5% for open air dried starch to 23.03% for starch dried at 60^{o}C. This could be due to the occurrence of thermal modification and annealing of starch granules as drying temperature increased, resulting in the destruction of native starch crystals. Frost *et al.* (2009) noted that the extrusion of starch to form thermoplastic starch destroyed native crystals as a result of the occurrence of starch modification.

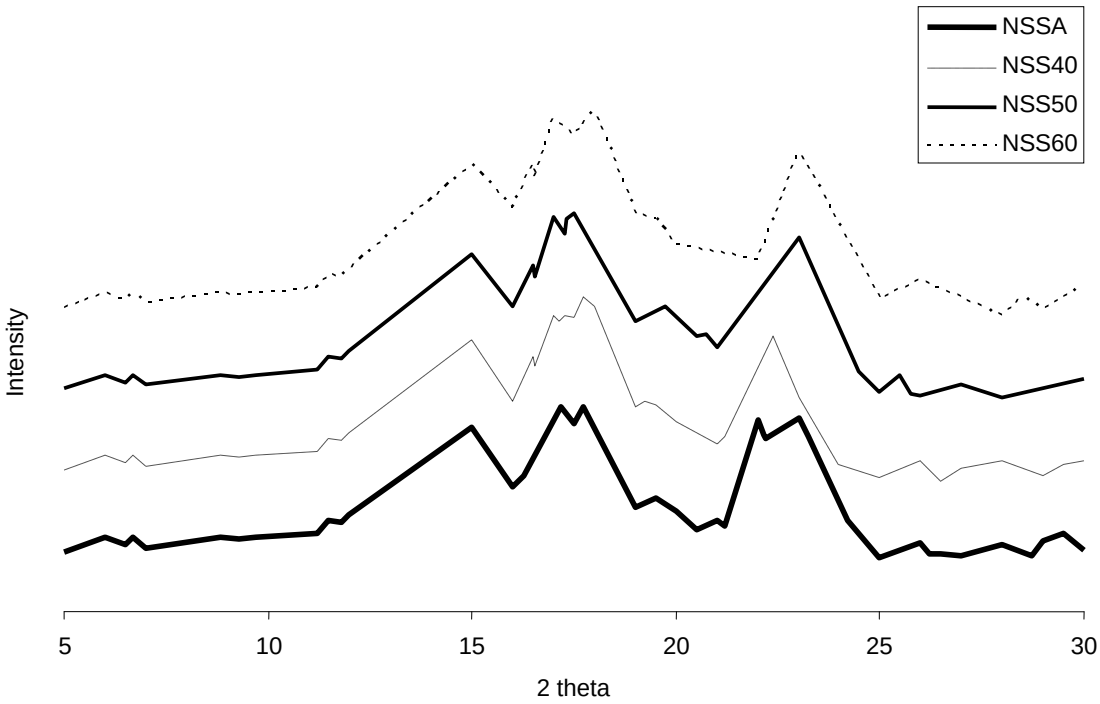


Figure3. Xray diffraction pattern of native sorghum starch dried at different temperatures: NSSA - dried in open air, NSS40 - dried at 40^{o}C, NSS50 - dried at 50^{o}C and NSS60 - dried at 60^{o}C

Table 3. Relative crystallinity of sorghum starch dried at different temperatures

Drying temperature, ^{o}C Degree of crystallinity, %
Open air
36.5
40
34.18
50

30.65
55
24.87
60
23.03

3.5 Physicochemical properties

The mean values of the physicochemical properties of sorghum starch dried at different temperatures are presented in Table 4.

Table 4. Physicochemical properties of sorghum starch dried at different temperatures

Drying temperature ^{o}C	Water binding capacity %	Swelling power g/g	Solubility %	Syneresis %	Paste clarity %
Open air	79.19±0.17d	13.006±0.975a	8.022±0.045a	15.10±0.01a	16.10±0.00d
40	79.63±0.68cd	12.01±0.614ab	7.08±0.140b	14.00±0.112b	22.50±0.75c
50	80.21±0.08c	11.23±0.25bc	6.54±0.38c	11.70±0.038c	26.20±0.19a
55	83.87±0.23b	10.76±0. 76c	3.33±0.08d	11.67±0.16c	25.00±0.04b
60	88.50±0.42a	8.96±0.182d	2.85±0.108e	10.80±0.002d	24.60±0.13b

Numbers in column denoted by the same letter are not significantly different ($p<0.05$)

3.5.1 Water binding capacity

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.,*1990), while high water binding capacity is indicative of a loose association of native starch polymers or low lipid content (Nwokocha and Ogunmola, 2005).

The water binding capacity of open air dried sorghum starch (Table 4) was not significantly different from that of starch dried at 40^{o}C in a tray dryer ($p<0.05$). The water binding capacity of the starch increased significantly with increase in drying temperature. This implies that the hydrophilic tendency of the starch increased with increase in its drying temperature. This may be due to the expansion of the amorphous region of the starch (Adebowale *et al.,* 2005) with increase in drying temperature, and as a result of the occurrence of modification in which some hydrogen bonds between the amorphous and crystalline regions got broken and the starch was opened up to water imbibition. Similar effect on starch water binding capacity has been reported as due to heat moisture treatment of chestnut starch (Singh *et al.,* 2009) and reheating of retrograded rice starch using microwave autoclave and steam (Han *et al.,* 2009).

The increase of starch water binding capacity with increase in drying temperature was linear and could be expressed with the following equation:

$$\omega = 0.6623T + 47.953, \quad R^2 = 0.98 \quad (4)$$

where
ω = water binding capacity, %; T = drying temperature, ^oC and R^2 = coefficient of determination. The water binding capacity of sorghum starch was found to be higher than that of cassava starch (Ikegwu *et al.,* 2009, Aviara *et al*., 2010).

3.5.2 Swelling power and solubility

Swelling power and solubility provide information on the nature of the associative forces within starch granules (Soni *et al.,* 1990; Ogunmola *et al.,* 2001). When starch is pasted in excess water system, the granules imbibe water through the amorphous regions in a reversible manner, and the amount of water imbibed increases with temperature until a critical temperature is reached (gelatinization temperature) at which the starch swells irreversibly with loss of crystalline order. This irreversible swelling is accompanied by significant uptake of water resulting in granule expansion and leaching of amylose into solution (Pomeranz, 1990).

The swelling power of open air dried sorghum starch was not significantly different from that of the starch dried at 40^oC ($p<0.05$), while the solubility of the open air dried starch was significantly higher than that of starch dried at different temperatures. Increase in drying temperature significantly decreased the solubility of the starch and beyond 50^oC; it significantly decreased the swelling power (Table 4). This could be due to the ordering rearrangement of starch molecules in the granules (Chung *et al.,* 2000), transformation of the amorphous amylose into helical form, increase in interaction between amylose chains or between amylose and amylopectin chains in the amorphous regions, and alteration in the interaction between crystallites and the amorphous matrix (Eerlingen *et al.,* 1997) resulting from increase in the annealing effect of starch drying as drying temperature increased. Olayinka *et al.* (2008) explained that the decrease in the swelling power and solubility of heat moisture treated sorghum starch compared with that of the native starch may have been due to granular stability resulting from unraveling of the double helices that might have been present in a crystalline array in the native granule. Decrease in swelling power and solubility could also have been due to amylose-lipid complexation (Olayinka *et al.,* 2008, Copeland *et al.,* 2009) as it has been shown that amylose inhibits granule swelling under conditions where amylose-lipid complexes are likely to be formed (Olayinka *et al.,* 2008). Gomes *et al.* (2004) and Adebowale *et al.* (2005) similarly reported that the annealing of cassava starch and heat moisture treatment and annealing of red sorghum starch, respectively decreased starch swelling power and solubility.

The relationship existing between the starch swelling power, solubility and drying temperature was found to be polynomial of the second order which can be expressed using the following equations:

$$\varphi = -0.0092T^2 + 0.7736T - 4.2615, \quad R^2 = 0.97 \quad (5)$$

$\varsigma = -0.0156T^2 + 1.3488T - 21.896,$ $R^2 = 1.00$ (6)

where
φ = swelling power, g/g, ς = solubility, %.

The above relationships show that the solubility of sorghum starch increased with increase in it swelling power. Soni *et al.* (1990), Soni *et al.* (1993) and Nwokocha and Ogunmola (2005) made similar observations on the starches of *quercus leucotrichophora* (oak), *cana edulis*, maize and *treculia africana* (African breadfruit) respectively, and attributed it to the fact that a part of the linear component of the starch may have been involved in the micellar network, while the rest was free from entanglement and therefore, was preferentially solubilized. The swelling power of sorghum starch was higher than that of cassava starch, and the solubility of the starch dried at temperatures below 50^{o}C was higher than that of cassava starch (Ikegwu *et al.,* 2009), while that of the starch dried above 50^{o}C was lower.

3.5.3 Freeze-thaw stability and paste clarity

Freezing a starch gel normally leads to the formation of ice crystals and the concentration of starch in the non-ice phase. Upon thawing, the water is easily expressed from the network, giving rise to the phenomenon known as syneresis. The ability of starch gel to withstand this phenomenon during freeze-thaw cycling (freeze-thaw stability) enhances its potential use in frozen food products (Baker and Rayas-Duarte, 1998). The level of syneresis is inversely proportional to the freeze-thaw stability of a starch gel.

The freeze-thaw stability of open air dried sorghum starch (Table 4) was significantly different from that of starch dried at different temperatures in a tray dryer ($p<0.05$). The freeze-thaw stability of starch dried at 50 and 55^{o}C respectively, as measured by the level of syneresis, was not significantly different. Syneresis in the starch was however, found to decrease logarithmically with increase in drying temperature. The relationship existing between percentage of syneresis and drying temperature could be represented by the following equation:

$\tau = -7.648\text{Ln}T + 42.066,$ $R^2 = 0.95$ (7)

where
τ = percentage of syneresis, %.

This implies that freeze-thaw stability increased with increase in drying temperature and could be attributed to changes in starch crystallinity due to partial crystallite melting, realignment of polymer chains and possible enlargement of the amorphous regions of the starch, arising from increase in drying temperature. Atichokudomchai *et al.,* (2002) reported that the freeze-thaw stability of acid-modified tapioca starch increased with annealing. Luo *et al.,* (2006) noted that the freeze-thaw stability of maize starch increased with microwave irradiation and attributed the result to increase in the magnitude of hydrogen bonding forces (interaction between amylose-amylose and amylose-amylopectin chains) within starch granules due to irradiation.

One of the major uses of starch is to impart viscosity to food, and the clarity of a starch paste is one of its important attributes in this function. Starch used to thicken fruit pie filling is preferably transparent, but starch used in spoonable salad dressing should be opaque. There are several

definitions that have been given to starch paste clarity. These include light transmittance characteristics (Hoover and Hadziyev, 1981; Maningat, 1986), light reflectance characteristics (Schoch, 1942) and degree of whiteness (Marrs *et al.,* 1977) of starch paste.

The paste clarity of open air dried sorghum starch was significantly different from that of starch dried at different temperatures in a tray dryer ($p<0.05$). The paste clarity of starch dried at 55 and 60^{o}C was not significantly different. Paste clarity increased with drying temperature up to a point and decreased with further increase in temperature. It had a relationship with drying temperature that was found to be polynomial of the second order and could be expressed with the following equation:

$$\rho = -0.0235T^2 + 2.4477T - 37.677, \qquad R^2 = 0.95 \quad (8)$$

where ρ = paste clarity, %.

This may be due to improvement of amylose leaching and enhancement of interactive bond formation between amylopectin molecules with increase in drying temperature up to a point after which granule remnants connected by a matrix of leached amylose and amylopectin increased and caused decrease in paste clarity.

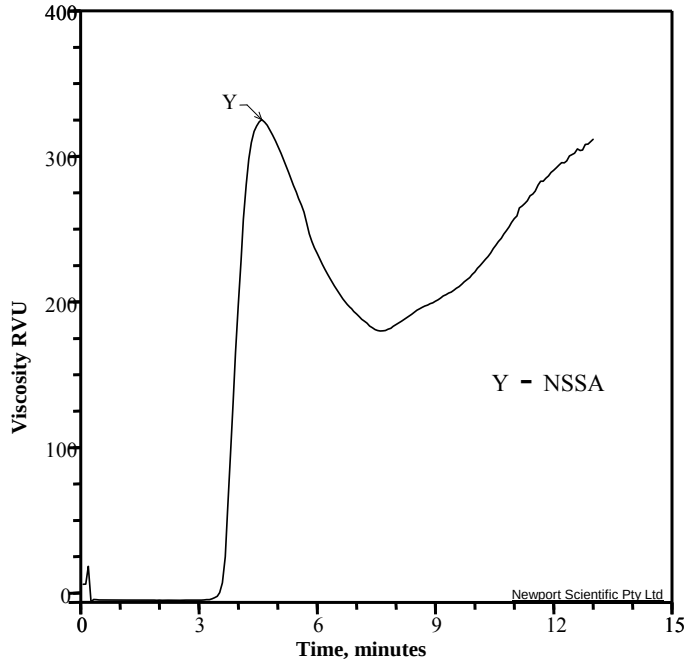
3.5.4 Pasting characteristics

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

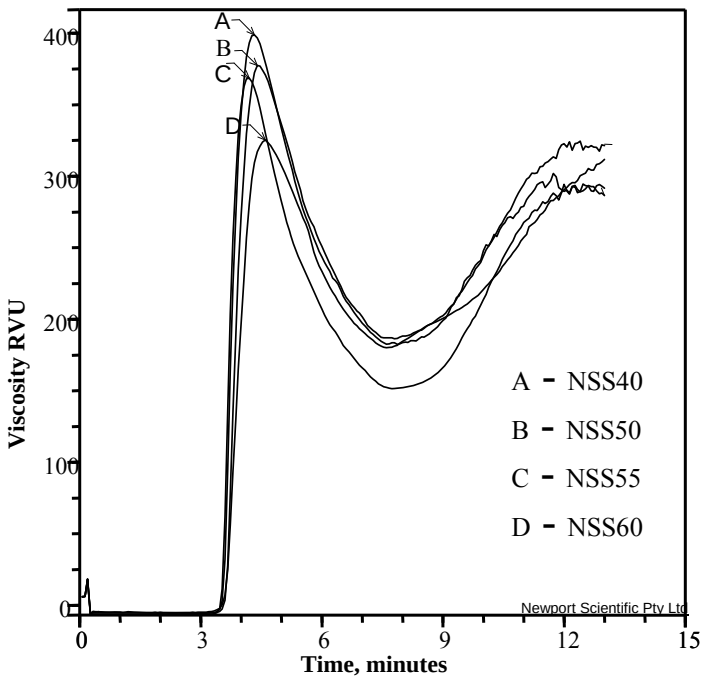
The pasting properties of sorghum starch dried in thin layer in open air and in a tray dryer at different temperatures are presented in Table 5. The pasting curves of the starch dried are presented in Figure 4.

Table 5. RVA pasting properties of sorghum starch dried at different temperatures

Drying temperature ^{o}C	Peak viscosity RVU	Final viscosity RVU	Breakdown viscosity RVU	Setback viscosity RVU	Pasting temperature ^{o}C	Peak time minutes
Open air	335.25	311.75	157.33	133.83	80.70	4.27
40	398.75	286.42	216.33	104	80.90	4.33
50	377.33	321.42	190.92	135	81.75	4.40
55	368.42	291.58	216.92	140.08	80.90	4.20
60	325.25	311.75	145.17	131.67	81.75	4.60



A



B

Figure 4. RVA pasting curves of native sorghum starch, A: NSSA - dried in open air and B: NSS40 – dried at 40^{o}C, NSS50 – dried at 50^{o}C, NSS55 – dried at 55^{o}C and NSS60 – dried at 60^{o}C in a tray dryer

Table 5 shows that the peak viscosity of open air dried starch was lower than that of starch dried at 40, 50 and 55^{o}C but higher than that of starch dried at 60^{o}C. The peak viscosity decreased with increase in drying temperature in a relationship that was found to be polynomial of the second order and could be expressed with the following equation:

$$\upsilon_p = -0.2017T^2 + 16.684T + 53.075, \quad R^2 = 0.96 \quad (9)$$

where
υ_p = peak viscosity, RVU.

According to Jacobs *et al.* (1995), both the formation of a tightly packed array of swollen and deformable granules and leaching of amylose can contribute to viscosity development in starch pastes. Decrease in swelling power with increase in starch drying temperature was expected to lead to increase in paste peak viscosity as a result of increase in the rigidity of granules (Chung *et al.,* 2000) due to insufficient gelatinization. The decrease in peak viscosity of sorghum starch with increase in drying temperature could therefore be due to increase in the annealing effect of heated air drying on the starch as the drying temperature increased. Gomes *et al.* (2004), Olayinka *et al.* (2008) and Han *et al.* (2009) respectively noted that the annealing of cassava starch, heat-moisture treatment of sorghum starch and autoclave reheating of retrograded rice starch decreased the peak viscosity.

The peak viscosity of sorghum starch used in this study was lower than that of starches from sorghum varieties grown in Zimbabwe (Beta *et al.,* 2000) and Algeria (Boudries *et al.,* 2009).

The final viscosity of open air dried sorghum starch was higher than that of starch dried at 40 and 55^{o}C respectively, but did not differ from that of starch dried at 60^{o}C and was lower than that of starch dried at 50^{o}C. The final viscosity increased with increase in drying temperature up to a point indicating that dissolved starch molecules must have increasingly formed into larger units (Adebowale *et al., 2005*) as drying temperature increased and the solution of starch was cooled. Thereafter, the final viscosity decreased with further increase in temperature as a result of the breakage of some hydrogen bonds within the starch granules. Chung *et al.* (2000) reported that the final viscosity of mung bean starch gel increased with increase in annealing temperature and thereafter, decreased with further increase in annealing temperature. The relationship existing between final viscosity and starch drying temperature was found to be polynomial of the third order which could be represented with the following equation:

$$\upsilon_f = 0.0816T^3 - 12.459T^2 + 627.22T - 10089, \quad R^2 = 1.00 \quad (10)$$

where
υ_f = final viscosity, RVU.

Breakdown viscosity is used in assessing the ability of a starch paste used as thickener to withstand severe processing conditions. During breakdown, the starch macromolecules and swollen granules undergo fragmentation resulting in lowering of viscosity. Pukkahuta *et al.* (2007) and Pukkahuta and Varavinit (2007) reported that increase in the temperature of heat-moisture and osmotic pressure treatments decreased the breakdown viscosity of starches. The breakdown viscosity of open air dried sorghum starch was lower than that of the starch dried at 40, 50 and 55^{o}C, but higher than that of starch dried at 60^{o}C. Below 60^{o}C, drying temperature did not significantly affect the breakdown viscosity of the starch. This may have been caused by the strengthening (stabilization) of the starch polymers through non-disruption of the hydrogen bonds within the granules, thereby preventing the association of amylose molecules after cooling (Gomes *et al.,* 2004). The breakdown viscosity of the starch had a relationship with drying temperature that was polynomial of the third order. This relationship could be expressed using the equation

$$\upsilon_b = -0.1236T^3 + 18.431T^2 - 907.68T + 14941, \qquad R^2 = 1.00 \quad (11)$$

where
υ_b = breakdown viscosity, RVU.

Setback viscosity is a measure of retrogradation, that is the extent to which dissolved starch macromolecules – amylose – are able to reassociate with themselves and granule fragments. During setback, the solubilized amylose molecules reassociate through the formation of a three dimensional network which results in a gel. The setback viscosity of open air dried sorghum starch was higher than that of starch dried at 40 and 60^{o}C but lower than that of starch dried at 50 and 55^{o}C. Setback viscosity increased with increase in drying temperature up to a point and decreased with further increase in temperature. It was speculated that the initial increase in setback could be due to disordered linear amylopectin chains (Tattiyakul *et al.,* 2007) being readily aligned with other adjacent linear branches to form gel with more widely spaced junction zones in the network aggregates. Decrease in setback with further increase in drying temperature may be due to the annealing effect drying at such temperature level resulting in starch stabilization and preventing the association of amylose molecules (Gomes *et al.,* 2004). Setback viscosity varied with drying temperature according to a polynomial relationship of the second order. This relationship could be represented by the following equation:

$$\upsilon_s = -0.1806T^2 + 19.476T - 386.32, \qquad R^2 = 0.99 \quad (12)$$

where
υ_s = setback viscosity, RVU.

The pasting temperature of open air dried sorghum starch was lower than that of starch dried at different temperatures in a tray dryer. There was no significant difference between the pasting temperatures of starch dried at 40 and 55^{o}C and at 50 and 60^{o}C, respectively. High pasting temperature indicates that the starch molecules may have had more mobility and undergone conformational reorganization (Petitot *et al.,* 2009), and low pasting temperature suggests that

fewer associative forces and crosslinks were present within the starch granules (Olayinka *et al.,* 2008). A combination of these phenomena may have induced the non-significant variation of the pasting temperature with drying temperature. The pasting temperature had a polynomial relationship of the third order with drying temperature, which could be expressed with the following equation:

$$T_p = 0.0025T^3 - 0.3868T^2 + 19.338T - 237, \quad R^2 = 1.00 \quad (13)$$

where

T_p = pasting temperature, ^oC.

The peak time of open air dried sorghum starch was lower than that of starch dried at different temperatures. Peak time did not significantly increase with drying temperature. Chung *et al.* (2000) reported that the annealing of mung bean starch did not significantly change the peak time. The relationship existing between the peak time of starch and drying temperature was found to be polynomial of the third order. This relationship could be represented by the following equation:

$$t_p = 0.0008T^3 – 0.1129T^2 + 5.5478T - 84.45, \quad R^2 = 1.00 \quad (14)$$

where

t_p = peak time, minutes.

4. CONCLUSIONS

The results of this study show that the proximate composition of open air dried sorghum starch was 10.73% moisture, 0.30% ash, 1.06% protein and 1.07% fat. The amylose content was 21.08% and the pH was 5.45. The granules were irregular in shape and it had unimodal distribution. The average granule size was 11.27μm. Drying of starch at the higher temperature of 60^oC slightly increased the granule size. The starch exhibited the A-type crystalline diffraction pattern which was not altered by drying temperature. The degree of crystallinity decreased with increase in drying temperature.

Increase in drying temperature significantly ($p<0.05$) increased starch water binding capacity and decreased the swelling power and solubility. Percentage of syneresis decreased implying that freeze-thaw stability increased with increase in drying temperature. Paste clarity was low but increased with increase in drying temperature up to 50^oC and decreased with further increase in temperature. Peak viscosity decreased as the drying temperature increased. Setback viscosity increased with increase in drying temperature up to a point and decreased with further increase in temperature and final and breakdown viscosities as well as pasting temperature and peak time had polynomial relationships of the third order with drying temperature.

Regression models were used to express the relationships existing between the physicochemical and pasting properties of the starch and drying temperature. These models could be used to select

the drying temperature that would yield starch with desired physicochemical properties for a functional application.

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