Original Research Article

MORPHOLOGY, FUNCTIONAL AND PASTING PROPERTIES OF GINGER STARCHES PREPARED BY FOUR DIFFERENT DRYING METHODS

ABSTRACT

Aim: This study aims at providing information on the morphology, physico-chemical and pasting properties of the ginger starches prepared by air drying, oven drying, freeze drying and microwave drying methods with a view to improving their utilization.

Place and Duration of Study: Department of Pharmaceutics and Pharmaceutical Technology, Olabisi Onabanjo University, Nigeria and Department of Pharmaceutics, University of Ibadan, Nigeria between March 2013 and February 2014

Methodology: Starch was isolated from the rhizomes of *Zingiber* officinale and dried by oven, freeze drying, air drying and microwave drying methods. They were studied for their composition, morphological, functional and pasting properties

Result: The residual protein content of the four starches was slightly different averaging 5.06 %. The residual protein of air dried starch was the lowest and that freeze dried starch was the highest. There was no significant variation in the saponin and tannin contents of the dried samples. The amylose content of the ginger starches ranged between 20.7% and 22.1%. The rank order of the amylose content was microwave dried > freeze dried> air dried > oven dried. Drying methods had a significant effect (P<0.0001) on the solubility index and swelling capacity of the dried samples investigated. The rank order of the swelling capacity of the ginger starches was freeze dried < air dried < oven dried < microwave dried. A higher solubility index was observed among the oven-dried sample and the microwave sample compared to freeze dried and air dried samples. X-ray fluorimetric (XRF) analysis of the different starch powders showed the presence of iron, copper and zinc in trace amounts; and the absence of heavy metals like lead and mercury. All the four dried samples have fairly good flow. Significant differences were observed in individual pasting parameters of the ginger starches especially in peak viscosity, trough viscosity, final viscosity and setback viscosity. Peak viscosity was found to be lowest for freeze dried starch (300.42RVU) and highest for oven dried starch (324.25 RVU). The rank order of the final viscosity was oven dried > microwave dried > air dried > freeze dried.

Conclusion: This study revealed that the physicochemical as well as the pasting properties of the ginger starches prepared by the four drying methods make them to be considered as excellent resource with possible applications in many food and pharmaceutical processing. The results also showed that difference in drying methods during processing has an effect

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on composition and pasting properties of ginger starch.

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Keywords: Ginger starch; morphology; functional; pasting properties; drying methods

18 **1. INTRODUCTION**

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20 Starch, the main reserve carbohydrate of several crops, is highly abundant in nature and can 21 be easily extracted with high purity and low cost. Industries have been using starch for a 22 long time as an ingredient in foods, especially for its functional properties. Depending on the 23 source, starches have different applications in the food and pharmaceutical industries where 24 they are used as fillers, glidants, thickners, binders, disintegrants as well gelling, bulking and 25 water retention agents [1]. Starch which is a major dietary source of carbohydrate, is the most abundant storage of polysaccharide in plants, and occurs as granules in the chloroplast 26 27 of green leave and the amyloplast of seeds, tubers and rhizomes. Starch consist of two molecules- the linear and helical amylose, and the highly branched amylopectin. They 28 usually consist of 10-38 % amylose although the proportion often varies depending on the 29 30 botanical source of the starch [2]. Tropical roots and tubers, grains, cereals and fruits are crops that have served as staple foods for millions of people throughout the world for many 31 32 centuries and they generally have a high starch content which has made them potential 33 sources of industrial starch.

34 Ginger is a perennial creeping plant, with thick tuberous rhizome, producing an erect stem 35 30 - 100 cm (1-3 ft) tall. The lance-shaped leaves are bright green, 15 - 20 cm (6-8 in) long, 36 with a prominent longitudinal rib, enclosing conical clusters of small yellow-green flowers 37 marked with purple speckles. It is propagated from rhizome cuttings, planted on rich, well drained loam. The underground rhizomes are the medicinal and culinary useful part of the 38 39 plant. Ginger is widely used as a flavoring agent in beverages and many food preparations. 40 Ginger helps relieve indigestion, gas pains, diarrhoea and stomach cramping. The primary 41 known constituents of ginger root include starch, gingerols, zingibain, bisabolenel, 42 oleoresins, essential oil (zingiberene, zingiberole, camphene, cineol, and borneol), mucilage, 43 and protein.

44 Starch isolated from ginger rhizomes has been investigated for its use as pharmaceutical 45 excipients [3-6] but it appears little or no work has been done to improve their functional 46 properties and enhance their industrial uses in Pharmacy as thickener, binder, filling material 47 and encapsulating agent. The increased requirements for starches in different applications, 48 such as functional and healthier food, or applications in pharmaceuticals have thus forced a steady development of new starch types and the crucial need to understand the properties of 49 the existing ones. This study was conducted to provide information on the morphology, 50 51 physico-chemical and pasting properties of the ginger starches prepared by air drying, oven 52 drying, freeze drying and microwave drying methods with a view to improving their utilization.

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54 2. MATERIAL AND METHODOLOGY

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56 2.1 MATERIALS

57 Ginger starch was extracted from ginger rhizome at the Department of Pharmaceutics and 58 pharmaceutical technology laboratory of Olabisi Onabanjo University, Nigeria. Water was 59 double distilled and all other chemicals were of analytical grade

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61 2.2 METHODOLOGY

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63 **2.2.1 Extraction of Ginger starch from ginger rhizome**

64 Rhizomes of ginger (Zingiber officinale Roscoe, Family Zingiberaceae) was washed with water, peeled and grated to produce ginger slurry. The grating was intermittently done to 65 prevent the starch from heating up due to heat from grater. Enough quantity of distilled water 66 was added to soak the material for 5 hours. The slurry was then thoroughly washed with 67 68 water onto the muslin cloth into a collecting vessel to release the starch granules embedded 69 in the parenchyma cells. The content of the collecting vessel was the allowed to settle for $\frac{2}{2}$ 70 hrs and the yellowish supernatant was decanted. Series of re-dispersion and decanting were done to remove impurities. The settled starch was scrapped off and divided into four equal 71 72 parts, each of which was subjected into different drying methods which are; freeze drying, oven drying, microwave drying and open air drying. 73

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75 2.2.2 Determination of Functional properties

76 77 2.2.2.1 Determination of bulk and tapped density

78 This was carried out for each sample using granules of mesh sizes of 500 - 1000 microns. A 79 50g sample was poured into a 50ml measuring cylinder and the bulk volumes were noted. The bulk density was calculated from the ratio of the mass to the bulk volume. The 80 81 procedure was repeated and result obtained in triplicate. Tapped density determination was 82 carried out by tapping the cylinder 100 times and the tapped volume obtained was used to 83 calculate the tapped density.

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85 2.2.2.2 Determination of particle density

86 The particle densities of the dried ginger starch powders were determined by the 87 pycnometer method using liquid immersion technique with benzene as the displacement liquid. A 50 mL pycnometer bottle was weighed when empty (w). This was filled with 88 89 benzene to the brim till it overflows. The excess was wiped off the bottle and its contents were weighed (W1). The difference between the two weights recorded was calculated (W2). 90 A 2 g quantity of the ginger starch powder was weighed (W3) and quantitatively transferred 91 into the pycnometer bottle. The excess solvent was wiped off and the bottle weighed again 92 93 (W4). The particle density, ρ (g/cm3), was calculated from the following:

- 94 ρ = W2. W3
- 95

50 (W1+W3-W4)(1) 96

97 2.2.2.3 Determination of flow properties

A 30 g sample of the powders was poured into a plugged glass funnel with the tip. 10 cm 98 above the flat surface of the bench. The granules were allowed to flow freely through the 99 orifice of the funnel to form a heap whose height and diameter were determined. The angle 100 101 of repose was calculated using the equation below:

- $Tan \theta = h/r....(2)$ 102
- 103 Where h = height and r = radius of circular heap

104 The hausners ratio was calculated as the ratio of the tap density to the bulk density of the 105 samples while the compressibility Index (C %) was calculated using bulk and tap densities data when fitted into the equation below: 106

107 Compressibility index = (tapped density-bulk density) / tapped density X 100 %......(3) 108

109 2.2.2.4 Determination of Swelling capacity and solubility Index 110 The method described by Leach et al., 1959 [7] was used with slight modifications. Starch 111 blends (1 g) were weighed and transferred into a clean dry test tube and weighed (W1). The 112 mix was then dispersed in 50 mL of distilled water using a magnetic stirrer. The resulting 113 slurry was heated at 80°C for 30 minutes in a thermo stated water bath. The mixture was 114 cooled to room temperature and centrifuged at 15 minutes. 5 mL of aliguot of the 115 supernatant was dried to a constant weight at 120°C. The residue obtained after drying represented the amount of starch solubilised in water. Solubility was calculated as per 100g 116 117 of starch on dry basis. The residue obtained after centrifugation with the water it retained 118 was transferred to the clean dried test earlier and re-weighed (W2).

119 Swelling of starch = (W2 - W1) / weight of powder x 100 (4)

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121 2.2.2.5 Determination of moisture content

A 5g powder was weighed in a tarred crucible and dried in hot air oven (Memmert Model 200) at 100±5°C till a constant weight is obtained. The moisture contents were calculated by the formula given below. The mean of three determinations was taken as the final moisture content

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$$MC = W_0 - W_1 / W_0 \dots \dots \dots \dots \dots (5)$$

127 Where MC is the moisture content and w_0 , w_1 are initial and final weights of the starch 128 respectively.

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130 2.2.2.6 Determination of protein, tannin and saponin content

131 The protein and tannin content of the dried ginger starch samples were determined by 132 established methods [8, 9]

133 2.2.2.7. Analysis of elemental constituents

Elemental analysis was carried out by the methods of Mineral Analysis using Atomic
 Absorption Spectrophotometer. Weight was recorded to the nearest 0.001g

136 2.2.2.8. Fourier Transform Infrared Spectroscopy (FT-IR)

The FTIR spectrum of the starches was recorded with a Perkin Elmer RXI
spectrophotometer (Connecticut, USA). The dry powder was mixed with KBr and pressed
into pellets. The spectrum was obtained by scanning between 4000 and 500/cm.

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141 **2.2.3 Determination of Pasting properties**

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Pasting characteristics were determined with a Rapid Visco Analyser (RVA Super 3, Newport Scientific Pty. Ltd, Australia). A 3 g sample was dissolved in 25 ml of water in a sample canister. The sample was thoroughly mixed and fitted into the RVA as recommended (Newport Scientific, 1998). The slurry was heated from 50 to 95°C with a holding time of 2 min followed by cooling to 50°C with another 2 min holding time. The 12 min profile was used and the rate of heating and cooling was at a constant rate of 11.25°C/min. Corresponding values for peak viscosity, trough, breakdown, final viscosity, setback, peak time and pasting temperature from the pasting profile were read from a computer connectedto the RVA.

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154 **3. RESULTS AND DISCUSSION**

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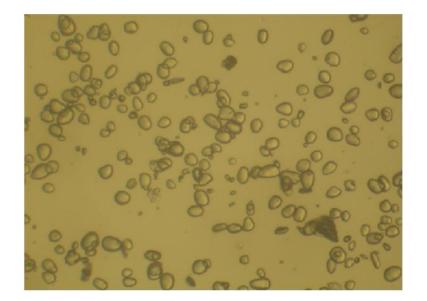
156 **3.1 Morphology and Composition of ginger starches**

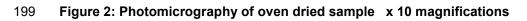
157 The diversity of the shapes of the granules in ginger starches is shown in Figure 1-4. The polyhedral/rounded/oval 158 shapes sample: range from in air dried 159 irregular/orbicular/oval/reniform in freeze dried starch; oval/ellipsoidal/rounded in microwave 160 dried starch and being oval/cordate/polyhedral in oven dried starch. Table 1 presents the composition and functional properties of the ginger starches obtained from the four drying 161 method. The residual protein content of the four starches was slightly different averaging 162 163 5.06% (Table 1). The residual protein of air dried starch was the lowest while freeze dried 164 starch had the highest. There was no significant variation in the saponin and tannin contents 165 of the dried samples. The amylose content of the ginger starches ranged between 20.7% 166 and 22.1%. The rank order of the amylose content of the starches was microwave dried > 167 freeze dried > air dried > oven dried. The amylose portion of the starch affects its swelling and hot-paste viscosities. Shimelis and Rakshit (2005) stated that as the amylose content 168 169 increases, the swelling tends to be restricted and the hot paste viscosity stabilizes. Based on 170 the classification of starches by amylose content [11], all the four dried samples are 171 intermediate (20-25% amylose).

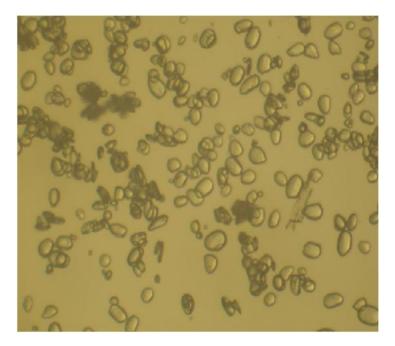
172 **3.2 Functional properties of ginger starches**

173 Swelling capacity and solubility index provide evidence of the magnitude of interaction 174 between starch chains within the amorphous and crystalline domains and also evidence of 175 association bonding within the granules of starches [12]. Drying methods had a significant 176 effect (P<0.001) on the solubility index and swelling capacity of the dried samples 177 investigated. The difference in swelling capacity among starches from different drying 178 methods indicates variation in the strength of associative bonding forces within the granules 179 [7]. The rank order of the swelling capacity of the ginger starches was freeze dried < air 180 dried < oven dried < microwave dried. The highest swelling capacity shown by microwave ginger starch might be indicative of weak bonding forces within its granules and the fact that 181 182 it is less compact when compared to the other starch granules. The difference in amylose 183 content and starch granular properties may also have affected the swelling capacity and 184 solubility of the ginger starches. A low swelling capacity observed in the freeze dried sample 185 may be due to stronger bonding force in its starch granules. A higher solubility index was 186 observed among the oven-dried sample and the microwave sample compared to freeze 187 dried and air dried samples. High solubility indices in starches has been attributed to the 188 easy solubility of the linear fraction (amylose) which is linked loosely with the rest of the 189 macro molecular structure, and released or leached out during the swelling process [18]. 190 Microwave dried sample with the highest amylose content had the highest solubility index 191 and swelling capacity. Starch granules become increasingly susceptible to shear 192 disintegration as they swell and starches with lower amylose content swell more than those 193 with higher amylose content.

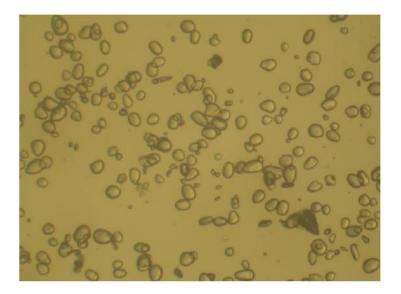
195 Figure 1: Photomicrography of freeze dried sample x10 magnification







205 Figure 3: Photomicrography of microwave dried sample x 10magnifications



208 Figure 4: Photomicrography of air dried sample x10magnification

212 Table 1: Functional properties of dried samples

Drying Methods	Bulk Density (g/cm ³)	Tapped Density (g/cm ³)	Particle Density (g/cm ³)	Angle of Repose (⁰)	Carrs Index (%)	Swelling Capacity	Moisture Content (%)	Solubility (%)	Protein content	Tannin content (mg/100g)	Saponin content (mg/100g)	Amylose content (%)
Freeze dried	0.418	0.589	1.420	41.3	29.25	10.57	10.7	9.87	4.725	6.030	0.125	21.17
Oven dried	0.400	0.584	1.420	43.3	31.51	10.86	10.3	10.17	5.270	5.850	0.155	20.67
Microwave dried	0.383	0.563	1.420	42.7	31.97	10.93	10.8	10.28	4.885	5.870	0.135	22.06
Air dried	0.383	0.563	1.420	42.3	31.97	10.65	10.6	9.97	5.345	6.310	0.145	21.14

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222 FTIR is a powerful technique to examine the formation of inter- and intra- molecular 223 hydrogen bonds in starches. Fourier Transform Infrared Spectroscopy revealed the presence of O-H and C-O absorption bands in all the samples. Freeze dried sample had O-224 H and C-O absorption bands distinct at 3350.38 cm⁻¹ and 1061.52 cm⁻¹ respectively. Oven 225 dried sample had 16 peaks while the air dried sample had 18 peaks. The British 226 227 Pharmacopoeia, 2005 gives limit tests for a number of possible contaminants in 228 pharmaceutical raw materials which may be introduced into the finished product during processing. Such tests include those for lead, arsenic, calcium, iron, potassium, aluminum, 229 230 halogens and a host of others.

Elements	Freeze dried	Oven dried	Microwave dried	Air dried	
Fe	0.195	0.215	0.235	0.185	
Р	121.055	112.260	109.46	120.145	
Са	5.230	5.345	5.175	6.350	
Na	1.680	1.490	1.750	1.635	
Mg	3.470	2.860	3.240	3.770	
Zn	0.435	0.435	0.415	0.415	
Mn	31.055	33.245	29.430	36.295	
Al	0.000	0.000	0.000	0.000	
К	21.470	20.45	23.59	24.230	
Pb	0.000	0.000	0.000	0.000	
Nickle	0.000	0.000	0.000	0.000	
Selenium	0.000	0.000	0.000	0.000	
Arsenic	0.000	0.000	0.000	0.000	
Silicon	0.000	0.000	0.000	0.000	
Cu	0.003	0.001	0.002	0.000	

231 Table 2: Elemental constituent of dried samples

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234 Although the pharmacopoeial requirements are not categorical on the exact tolerable level of 235 any possible contaminant, it should not be presumed that unusual impurities are tolerated. 236 X-ray fluorimetric (XRF) analysis of the different starch powders (Table 2) showed the 237 presence of iron, copper and zinc in trace amounts. Heavy metals like lead and mercury 238 were absent. The presence of heavy metals in formulated products is highly undesirable as 239 they form stable covalent or co-ordinate complexes with body protein and can also act as 240 catalyst (due to their variable valency state) to induce auto -oxidative reactions. The 241 Pharmacopoeia has therefore placed strigent limits on the amount of lead and other heavy 242 metals that may be present in pharmaceutical products [14]. Angle of repose, compressibility 243 index and Hausner ratio determines the flow character or properties of powder particles. For 244 an excellent flow, angle of repose must fall within the range of 25-30 degrees. All the four 245 dried samples have fairly good flow which can be improved by the addition of a glidant if they 246 are to be used in tablet formulation and production

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Drying	Peak	Trough/Hold	Final	Breakdown	Setback	Peak	Pasting
Methods	(RVU)	(RVU)	Viscosity (RVU)	(RVU)	(RVU)	Time (min.)	Temp (⁰C)
Freeze dried	300.42	151.75	222.00	148.67	70.25	5.02	82.45
Oven dried	324.25	200.00	290.33	124.25	90.33	4.66	81.48
Microwave dried	306.42	163.42	236.00	143.00	72.58	4.58	82.66
Air dried	322.33	159.83	235.92	162.55	76.09	4.75	82.35

Table 3: Pasting properties of Ginger starch powders

3.3 Pasting properties of ginger starches

256 Table 3 highlights the pasting properties of the starches. Pasting temperature of different 257 ginger starches ranged from 81.5 to 82.7°C; the highest for microwave starch and the lowest 258 for oven dried starch. Peak viscosity is the maximum viscosity attained by gelatinized starch 259 during heating in water. It indicates the water binding capacity of the starch granule. Peak 260 viscocity was found to be lowest for freeze dried starch and highest for oven dried starch. 261 The rank order of the peak viscosity was oven dried > air dried > microwave > freeze dried. 262 Trough viscosity was found to be the lowest for freeze dried starch (200.0 RVU) and the 263 highest for oven dried starch (151.75RVU). Holding strength measures the ability of starch 264 to remain undisrupted when paste is subjected to a long duration of high, constant 265 temperature during the process of steaming. Oven dry method produced starch with the 266 highest holding strength. Setback is a measure of re-crystallization of gelatinized starch 267 during cooling. There are significant differences in the values of setback viscosity for the 268 dried starches. The difference in setback among different starches may be due to the 269 amount and the molecular weight of amylose leached from the granules of the gelatinized 270 starch [15]. Oven dried sample had the highest final viscosity. Final viscosity indicates the 271 ability of the starch to form a viscous paste. The rank order was oven dried > microwave 272 dried > air dried > freeze dried sample. The increment in the final viscosity of paste made 273 from oven-dried sample during cooling indicates formation of a firm gel after cooling, rather 274 than a viscous paste as in the case of other-dried sample. The values of breakdown 275 viscosity of the starch samples vary significantly. Breakdown viscosity is a measure of the 276 vulnerability or susceptibility of the cooked starch to disintegration. The higher the 277 breakdown in viscosity, the lower the ability of the starch sample to withstand heating and 278 shear stress during cooking [16]. Therefore, oven dried starch (124.25 RVU) and microwave 279 (143.0 RVU) starches might be able to withstand more heating and shear stress compared 280 to starches from freeze dried starch (148.67 RVU) and air dried sample (162.55 RVU) 281 because of their lower breakdown value. Starches with high paste stability or breakdown 282 have very strong cross-linking within the granules [17]. It therefore means that there is 283 stronger cross-linking within the granules of air-dried starch powder compared to the other 284 dried samples.

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4. CONCLUSION

287 The pasting and physicochemical properties obtained indicate that all the starches have 288 useful technological properties for many applications. The data showed that the ginger starch prepared by oven drying can be used as a functional ingredient in food systems. This study revealed that the physicochemical as well as the pasting properties of the ginger starches prepared by the four drying methods make them to be considered as excellent resource with possible applications in many food and pharmaceutical processing.

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294 **AUTHORS' CONTRIBUTIONS**

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Author BOA and BLG designed the study and wrote the protocol. Author BOA, BLG and FY managed the literature search and performed the statistical analysis. Author BLG, BOA and FY managed the analyses of the study while author BLG wrote the first draft of the manuscript. All the authors read and approved the final manuscript.

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