

Pasting And Functional Properties Of Native And Modified Pupuru

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Abstract—The purpose of this work is to investigate the effect modification on functional and pasting properties of pupuru. Pupuru produced from fermented cassava was subjected to chemical modification by acetylation and phthalation. Modified and native pupuru were characterized by Fourier Transform Infrared (FTIR), Functional and pasting properties of the native and modified pupuru were determined. FTIR analysis shows the presence of C = O ester (carbonyl group) for the modified pupuru, implying that the hydroxyl group contents in pupuru were reduced after phthalation and acetylation reaction. . Acetylation increased the water absorption capacity, oil absorption capacity, swelling properties and water solubility. Phthalation reduced the water absorption capacity, oil absorption capacity, swelling properties and water solubility. Modification reduced gelation and bulk density of the pupuru. Modification reduced the peak viscosity, break down viscosity, and pasting temperature. Acetylation reduced setback and peak time values while phthalation increased setback and peak time value. The modified pupuru could more effectively meet the functional and pasting property demands in food and non-food industries.

Keywords—Pupuru; Acetylation; Phthalation; Modification; Gelation

I. INTRODUCTION

An estimated fifty million people in Africa Derived more than 500 kcal per day from cassava [1] . (Manihot esculenta Crantz) roots are widely used as food in Nigeria. It is one of the most important root crops in most tropical countries and it is estimated that foods processed from cassava roots are staple for over 800 million people in the tropics [2]. Uses of cassava roots as an important food has been predominant in many countries in the tropic in the past decade [3].

Pupuru is a dried fermented product from cassava, it is consumed as cooked dough in eateries and at homes with different soups in West African regions where they are identified with different ethnic or tribal names. People living in the riverine areas of the southern, western, eastern and the middle belts of Nigeria, commonly consumed pupuru which is also known as "Ikwurikwu" [4]. [5], reported that processing of cassava to "pupuru" plays a vital role in ensuring food security and is a major income earning venture for some people in Nigeria. The smoking of the fermented cassava roots makes "pupuru" processing unique as the fermented cassava mash is molded into balls and dried using smoke heat which will impact some characteristic flavour and aroma to this product ([4]; [6]). As increase in our population cause more demand for food, more people may have to depend on "pupuru" to supplement their carbohydrate intake [6]. Currently, "pupuru" processing is entirely traditional and the product consumption is more confined to the indigenous areas [4]. Although, pupuru is mostly used as food, but it can be biologically or chemically modified into different important and various products, namely, adhesives, beverages, confectioneries, paper, textiles, plastics and pharmaceuticals. Native pupuru could have limited application because of the reduced functional properties. Therefore, modification is important to improve the functional groups and to widen its applications especially in industries in modified forms. Modification of pupuru could be chemical; physical; or enzymatic; it involves changing the physicochemical properties of unmodified (native) pupuru to obtain more useful derivatives which have an edge over their native forms [7].

II. MATERIALS AND METHODS

• Pupuru Processing

The traditional method of "pupuru" production as described by [8] was employed. Fresh cassava roots, Manihot esculenta were peeled, soaked (5 days), decorticated, dewatered, molded into balls and dried in an oven at temperature Of 50°C. The cooled

“pupuru” balls were scrapped milled and packed in different packaging materials

- Preparation of Acetylated Pupuru

The method of [9] was used. 100g of pupuru was dispersed in 500ml of distilled water, the mixture was stirred magnetically for 20 mins. The pH of the slurry obtained was adjusted to 8.0 using 1M NaOH. Acetic anhydride (10.2g) was added over a period of 1h, while maintaining a pH range 8.0-8.5. The reaction proceeded for 5 mins after the addition of acetic anhydride. The pH of the slurry was adjusted to 4.5 using 0.5M HCl. The precipitate obtained was filtered, washed four times with distilled water and air-dried for 48 hrs to give acetylated pupuru.

- Preparation of Pregelatinized Pupuru Phthalate

The method of [10] was used. pregelatinized pupuru phthalate was obtained from two steps: gelatinization and esterification. Gelatinization process was performed by heating starch solution above 70°C and drying it into flakes which were then milled and sieved. The obtained praelatinized pupuru powder was then dispersed in purified water and reacted with solution of phthalic anhydride in ethanol 96%. Solution NaOH 10 M was continuously dropped during reaction to keep alkaline condition in pH 8-10. Sodium sulphate anhydrous was also added during reaction to absorb the excessive water. Reaction was conducted under 1000 rpm stirring speed of homogenizer. The stirring was continued until 30 minutes after reaction was completed and the solution was allowed to stand overnight. The solution was then neutralized by adding HCl solution to reach pH 6.5 – 7.

- Fourier Transform Infrared (FT-IR) Analysis

The functional groups of native and modified pupuru were obtained using Fourier Transform Infrared (Shimadzu Model FTIR – 8201PC).

III. FUNCTIONAL PROPERTIES

- Water absorption capacity (WAC)

Water absorption capacity was carried out using the method described by [11]. One gram of the sample was mixed with 10 mL distilled water for 5 min. The sample was allowed to stay for 30 min, centrifuged at 3000 rpm for 30 min, the volume of the supernatant was measured. Assuming the density of distilled water was 1 g mL⁻¹.

- Swelling power and solubility

Swelling power and solubility were determined using the method described by [12]. One gram of the sample was weighed and transferred into a clean and dried test tube (W₁). The native starch was dispersed in distilled water (20 mL). The obtained slurry was heated at 60°C for 30 min in a calibrated water bath. The mixture was centrifuged at 3000 rpm for 20 min, the supernatant was decanted and the swollen granules were weighed (W₂). 10 mL of the

supernatant was oven-dried at 120°C. The residue obtained on drying the supernatant indicates the quantity of pupuru solubilised. The swelling and solubility are calculated as follows:

$$(\%) \text{Swelling capacity} = \frac{W_2 - W_1}{\text{Weight of Pupuru}} \times 100$$

$$\text{Solubility of Pupuru (\%)} = \frac{\text{weight of dried aliquot}}{\text{weight of Pupuru}}$$

- Gelatinization temperature (GT)

Gelatinization temperature was determined by the method described by [13] About 1 g of pupuru sample was transferred into a beaker and 10 mL of distilled water was added. The dispersion was heated on a hot plate. The gelatinization temperature was then taken with a thermometer suspended in the slurry.

- Determination of least gelation concentration

The method of [9]. was used with slight modification. Appropriate sample suspensions of 2, 4, 6, 8, 10, 12, 14, 16 w.v⁻¹ were prepared in 5 mL distilled water. The test tubes containing the suspensions were heated for 1 h in boiling water cooled under running tap water. The least gelation concentration was determined as concentration when the sample from the inverted test tube did not fall down or slip.

- Determination of bulk density

Bulk density was determined using the procedure of [14]. with slight modification. Sample (10g) was put into a 100 mL graduated cylinder. The cylinder was tapped forty times and the bulk density was calculated as weight per unit volume (g.mL⁻¹)

$$\text{Bulk density (g/mL)} = \frac{W_2 - W_1}{V}$$

- pH Determination

The pH was determined using the procedure of [15]. 20 g of the sample was shaken in 100 mL of distilled water for 5 min and the pH was determined using a pH meter.

IV. Pasting Properties

Pasting characteristics were determined with a Rapid Visco Analyzer (RVA). (Model RVA 3D+, Network Scientific, Australia); the starch (2.5g) sample were weighed into a dried empty canister; 25ml of distilled water was dispensed into the canister containing the sample. The solution was thoroughly mixed and the canister was well filled into the RVA as recommended. The slurry was heated from 50°C to 95°C with a holding time of 2 minutes followed by cooling to 50°C with 2mins holding time. The rate of heating and cooling were at constant rate of 11.25°C/mins. Peak viscosity trough, breakdown, final viscosity, setback, peak time and pasting temperature

were read from the pasting profile with the aid of thermocline for windows software connected to a computer [16]

III. RESULTS AND DISCUSSION

The FTIR wavelengths of native, acetylated and phthalated pupuru are presented in Fig. 1a, b, c. In spectrum of native pupuru (Fig.1a), the peak at 3425.58 cm^{-1} and 2926.07 cm^{-1} correspond to O-H and C-H stretching, while the peaks at 1654.92 cm^{-1} and 1438.18 cm^{-1} correspond to O-H and C-H bending. Acetylated pupuru (Fig.1b), show new strong absorption bands at 1703.14 cm^{-1} ; this indicates (C=O stretching of acetyl group) Phthalated pupuru (Fig.1c), showed new absorption bands at 1932.67 cm^{-1} , due to the carbonyl group of the ester (evidence that phthalation reaction has occurred).

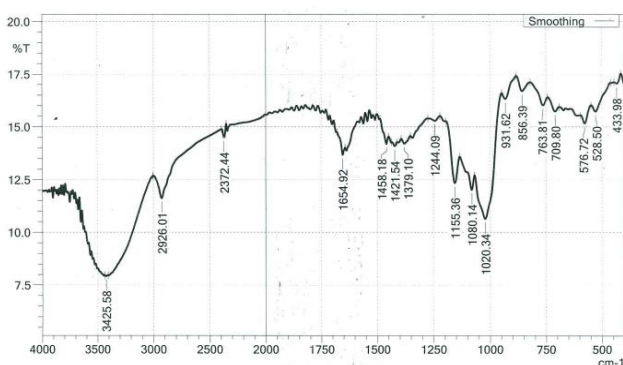


Fig. 1a FTIR spectrum of native pupuru

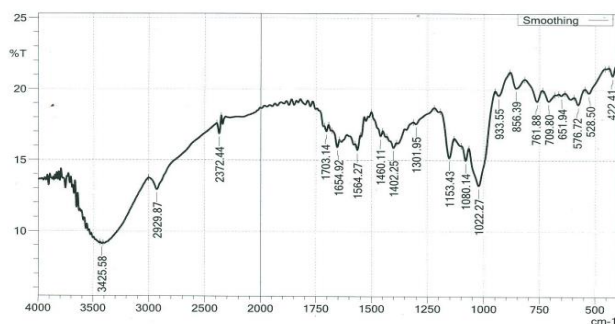


Fig. 1b FTIR of acetylated pupuru

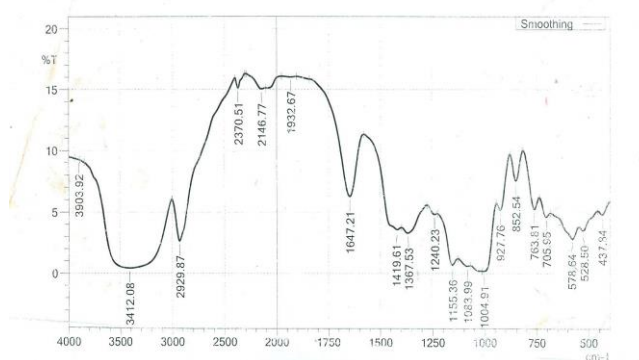


Fig.1c. FTIR of phthalated pupuru

The Functional Properties of Native Starches and their Derivatives are presented in Table 1

The water absorption capacity (WAC) of the native pupuru ($60.70\pm 0.12\%$) was increased ($93.00\pm 0.14\%$) after acetylation and decreased after phthalation ($53.30\pm 0.11\%$). pupuru showed the highest water absorption capacity and phthalated pupuru showed the least WAC. This increase in the WAC in acetylated pupuru may be associated with the introduction of acetyl groups that impeded intermolecular chain associations, causing a structural disorganization that facilitated water access in the amorphous region. Decrease in WAC in phthalated pupuru could be that phthalation reinforces the structure of the granules and limit water absorption which restricts the mobility of the chain in the amorphous region (Gunaratne and Corke, 2007)[17]. The oil absorption capacity (OAC) of the native pupuru ($42.30\pm 0.17\%$) was increased (43.30 ± 0.17) after acetylation and reduced after phthalation ($36.70\pm 0.23\%$) after phthalation. Acetylated pupuru showed the highest oil absorption capacity and phthalated pupuru showed the least OAC. This result suggested that acetylation enhanced the hydrophobic tendencies of the pupuru. Similar results were recorded by Uzomah and Ibe (2011)[18], who indicated that Acetylated starches had the strongest affinity for oil absorption.

The swelling power of the native pupuru increased after acetylation and decreased after phthalation. Acetylated pupuru showed the highest swelling power while ($38.50\pm 0.06\%$) while phthalated pupuru ($21.80\pm 0.11\%$) showed the least swelling property. Increase observed in the swelling power of acetylated pupuru may be due to weakening and disrupting of intra- and inter molecular hydrogen bonds in the starch chains, which may increase the accessibility of the pupuru granules to water [19]. starches with higher swelling power would be expected to release the active pharmaceutical ingredient from its compacts at a faster rate, where starch acts as a disintegrant [20].

The water solubility of the native pupuru ($28.10\pm 0.04\%$) was increased ($34.50\pm 0.41\%$) after acetylation and decreased ($12.40\pm 0.02\%$) after phthalation. Acetylated pupuru showed the highest water solubility while phthalated pupuru showed the least water solubility. The increase in water solubility of acetylated pupuru may be due to the structural reorganization that weakens the granules and enhances the amylase leaching. A similar increase in the water solubility upon acetylation have been reported for African yambean [21]. Many easy- to -cook processed foods that have high content of starch ,such as soups, puddings and creams are based on high solubility.

The gelatinization temperatures of the native, acetylated and phthalated starches, are shown in Table 1 Acetylated and phthalated pupuru has a lower gelatinization temperature compare to the corresponding native pupuru. Native pupuru has the highest gelation temperature (80°C) while acetylated pupuru has the least gelation temperature (75°C).

Lowering of gelatinization temperatures could be adduced to the addition of phthalated and acetyl group on the polymer backbone, which allows higher flexibility [22], resulting in the disruption of hydrogen bonds between the chains in the amorphous regions [23].

The pH values for acetylated and phthalated starches were found to be slightly lower than their corresponding native pupuru, but still fall within the pH range of 3-9 obtained for most materials used in pharmaceutical, domestic and food industries. The reduction in pH of native pupuru after acetylation and phthalation can be attributed to the introduction of acetyl phthalic groups on the pupuru molecules thereby increasing the acidity of the molecules.

The bulk density of the native (0.51 ±0.04) pupuru was reduced after acetylation(0.42 ±0.01) and phthalation (0.36±0.03) . Native pupuru showed the highest bulk density while phthalated pupuru showed the least bulk density. Acetylation and phthalation reduced the bulk density of the pupuru. The higher the bulk density the greater the quantity of material that can be packaged within a specified packaging space [24]. Decrease bulk density could be that modification has reduced the particle sizes of the pupuru. Samples with high bulk densities are considered heavy. Hence, phthalated pupuru in this study is light and could be used in the food processing industry.

Table 1. Functional properties of native and modified starches

Sample	WAC(%)	OAC(%)	SWP (%)	Solubility (g/g)	Gelation temp. (°C)	pH	Bulk density
Native pupuru	60.70±0.12 ^b	42.30±0.17 ^c	29.20±0.10 ^d	28.10±0.04 ^b	80	6.55	0.51±0.04 ^c
Acetylated pupuru	93.00±0.14 ^c	44.30±0.15 ^b	38.50±0.06 ^c	34.50±0.41 ^c	75	3.92	0.42±0.01 ^b
Phthalated pupuru	53.30±0.11 ^a	36.70±0.23 ^a	21.80±0.11 ^a	12.40±0.02 ^a	78	5.62	0.36±0.03 ^a

Values are means of three replicate (determined on dry weight basis) ± standard deviation, significantly different at p < 0.05. WAC- water absorption capacity; OAC- oil absorption capacity; SWP- swelling power

The results of least gelation of native and modified starches are presented in Table 2. . The lowest gelation concentration for native pupuru was 8%. However, none of the pupuru showed positive results at the concentrations of 2, 4 and 6%. At 10% concentration, all the native and modified pupuru formed gel . All other higher concentrations showed positive results. It was observed that increase in

concentration leads to gel formation. A similar increase in the least gelation concentration upon acetylation were obtained in acetylated starches of African yambean starch [21] and sweet potato starch [25]. Thus, the results suggested that the native starches are better gelating food additives than acetylated and phthalated starches.

Table 2. Least gelation concentration of native and modified starches

Sample	2 %	4 %	6 %	8 %	10 %	12 %	14 %	16 %
Native pupuru	-Viscous	-Viscous	-Viscous	+ Gel	+ Gel	+ Gel	+ Gel	+ Gel
Acetylated pupuru	-Viscous	-Viscous	-Viscous	-Viscous	+ Gel	+ Gel	+ Gel	+ Gel
Phthalated pupuru	-Viscous	-Viscous	-Viscous	-Viscous	+ Gel	+ Gel	+ Gel	+ Gel

Determination were carried out in triplicates

(-) No gelation (+) gelation

The pasting characteristic of native pupuru and their derivatives are presented in Table 3. The result of pasting property showed that peak viscosity ranged between 2132.00 – 3646.00 BU with phthalated pupuru having least value and native pupuru having highest value. The peak viscosity of native (3646.00 BU) pupuru was reduced after acetylation (3419.00 BU) and phthalation (2132.00 BU). This trend is in agreement with the results reported by [26] for modified mucuna bean starch. The decrease in peak viscosity could be as a result of partial cleavage of the glycosidic linkage due to acetylation and phthalation. This gave rise to a partially degraded network with weak shear resistance

and failure to maintain the integrity of the starch granule, thereby resulting in a lower viscosity [27]. The breakdown viscosity of native pupuru was reduced after acetylation and phthalation. Phthalation greatly reduced the breakdown viscosity of the pupuru. The reduction in breakdown viscosity has been adduced to the introduction of new substituent groups into the modified pupuru.[26]. The break down viscosity ranged between 410.00 – 1495.00 BU with phthalated pupuru having least value and native pupuru having highest value. Phthalated pupuru (410.00BU) has the least break down viscosity, indicating that it has more ability to withstand breakdown during heating.

Setback viscosity is a measure of the degree of retrogradation of starch, mainly amylose [28], implying that high setback viscosity value means a high tendency of starch to retrograde. The set back value, an index of retrogradation tendency of the native pupuru (987.00BU) was increased (999.00BU) after phthalation and reduced (684.00BU) after acetylation. The set back viscosity ranged between 684.00-994.00. phthalated pupuru has the highest value while acetylated pupuru has the least value. Phthalation increased the set back viscosity of the native pupuru and acetylation reduced the set back viscosity of the native pupuru. This reduction in the set back value of acetylated pupuru indicates that new substituent groups have been introduced into the modified derivatives and this restricted the tendency of the starch molecules to realign after cooling, thereby encouraging a lower setback value for the modified pupuru [29]. This reduction in setback values of acetylated pupuru were in agreement with the finding of [21] on reduction of setback values of native African yam beans after oxidation and acetylation. according to [30], staling of bread is a function of retrogradation (setback), that is association of the linear amylose

molecules. Acetylated pupuru could be a better stabilizing agent. [31]. High set back values of starch results to retrogradation.

The peak time ranged between 5.00 - 6.00 mins. Phthalated pupuru has the highest peak time value while phthalated pupuru has the least peak time value. The differences in peak time of the starches could be attributed to difference in intrinsic behaviours and responses of the pupuru to paste formation and botanical variations. The least value of acetylated pupuru (5.00mins) suggested that deformation occurs easily in acetylated pupuru.

The pasting temperature of the native pupuru (76.65°C) was reduced after acetylation (72.10°C) and phthalation (74.40°C). The decrease in pasting temperature of the pupuru after modification could be as a result of the weakening of the glucosidic bonds during the modification process. Weakening means disruption of the inter- and intra- molecular bonding such as hydrogen bond existing within the macromolecules and structural reorganisation of the amorphous region of the pupuru.

Table 3. Pasting Properties of Native Pupuru and their Derivatives

Samples	Peak viscosity (BU)	Trough (BU)	Breakdown (BU)	Final visc (BU)	Setback (BU)	Peak time (mins)	Pasting temp (oC)
Native pupuru	3646.0±0.03	2151.0±0.25	1495.0±1.25	3138.0±0.06	987.0±0.02	5.2±0.01	76.7±0.01
Acetylated pupuru	3419.0±0.04	2323.0±1.22	1096.0±1.20	3007.0±0.08	684.0±0.01	5.0±0.03	72.1±0.03
Phthalated pupuru	2132.0±0.12	1722.0±0.15	410.0±0.03	2084.0±0.16	999.0±1.25	6.0±0.01	74.4±0.06

Values are means of three replicate (determined on dry weight basis) ± standard deviation, significantly different at p< 0.05

CONCLUSION

Pupuru produced from fermented cassava was subjected to chemical modification by acetylation and phthalation. Functional and pasting properties of the native and modified pupuru were determined. Acetylation increased the water absorption capacity, oil absorption capacity, swelling properties and water solubility. Phthalation reduced the water absorption capacity, oil absorption capacity, swelling properties and water solubility. Modification reduced gelation and bulk density of the pupuru. Modification reduced the peak viscosity, break down viscosity, and pasting temperature. Acetylation reduced setback and peak time values while phthalation increased setback and peak time value. Increase in water absorption capacity of acetylated pupuru gave it advantage of being used as a thickener in liquid and semi liquids foods, it could be used in the development of confectionary products such as hard candies. Increase in swelling power results into high digestibility and ability to improved dietary properties of the acetylated pupuru. Increase in water solubility enhances easy to cook processed food. Decrease in gelatinization

temperature is beneficial to industry, as a thickening agent, the starch must form a gel at a lower temperature so as to reduce energy costs during the manufacture of the product.

The high peak viscosity of native pupuru corresponds to high thickening power which could be used as a tablet binder in pharmaceuticals.

Tendency for retrogradation reduced after acetylation in setback values. This is an advantage in food products which undergoes staling easily like bread and in sauces and soups which undergo loss of viscosity and precipitation as a result of retrogradation.

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