



Original research

Variability of properties of starch isolated from yam-soybean composite flour formulation intended for 'amala' (yam-based doughmeal) preparation

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ABSTRACT

This study investigated variability in starch properties within a yam-soybean flour blend intended for making Amala. Soybean flour was added at 10-50% levels. Starch was isolated using standard procedures and evaluated for yield, pH, bulk density, swelling power, water and oil absorption, pasting properties, and Fourier Transform Infrared spectroscopy. The result showed that substituting yam flour with soy flour caused significant yield changes in isolated starch from the blends. Higher soybean flour concentration in the composite formulation resulted in decreased starch yield. The pH values also moved towards more acidity with an increase in soybean flour level. The bulk density ($0.61 - 0.79 \text{ g/cm}^3$), WAC (246.98 – 265.76%), OAC (120.4 – 181.17%) of the isolated starch samples were all found to decrease as a result of soybean flour inclusion. However, the swelling capacity of the isolated starch samples ($2.90 - 3.69 \text{ g/g}$) was found to increase with soybean flour inclusion. The pasting properties of the isolated starch samples including peak viscosity (2745 – 5743 cP), breakdown viscosity (318 – 1006 cP), final viscosity (3845 – 8120 cP), and setback viscosity (1178 – 3434 cP) were all found to decrease with the inclusion of soybean flour; while peak time (5.27 – 5.32 min) and peak temperature ($82.4 - 85.1^\circ\text{C}$) increased in values as a result of soybean flour inclusion. The FTIR spectroscopy of the starch samples exhibited differences in the spectra pattern in terms of shifting in the position of the wavenumber and the appearance of new functional groups.

Keywords: Starchy food; Composite blend; Dough characteristics; FTIR

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1. Introduction

Amala is a popular starchy meal widely consumed by the Yoruba people of South-Western Nigeria (Ojokoh & Adeleke, 2020; Eyinla et al., 2022; Tanimola et al., 2022) as well as non-Yoruba communities in Nigeria and other countries in West Africa such as Benin Republic and Ghana, where it is known as "kokonte" among the Ashante people (Tanimola et al., 2022). It is exclusively made from yam or cassava with yam flour being preferred due to its traditional significance and unique texture (Jimoh et al., 2017; Jeannelle et al., 2020). The preparation of *amala* essentially involves reconstituting yam flour in boiling water until it forms a dark brown smooth paste. *Amala* is primarily composed of carbohydrates, lacking adequate other nutrients and is popular particularly among rural dwellers.

Efforts have been made to enhance the nutritional value of this staple food by incorporating legumes and other protein-rich plant foods. Studies have shown that supplementing yam flour with 35% distillers spent grain can increase the protein content of the yam-based doughmeal by over 100%, while fortifying yam flour with 30% soybean flour can raise the protein content from 3.16 to 18.21% (Karim et al., 2017; Salome et al., 2021). Soybean, known for its nutritional value, serves as an ideal grain for meeting protein and energy requirements for both humans and animals. Soybean flour is commonly used in food fortification programmes and has been proven to improve the nutritional quality of various food products (Malomo et al., 2012). It is often combined with other flours to create composite flour. Studies on the fortification of yam, cassava, and plantain flours with soybean have demonstrated enhanced nutritional quality in resulting meals, including *Amala*. However, it is important to consider that fortification may influence

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the functional and pasting characteristics of flour-based foods (Malomo et al., 2012).

While soybean flour fortification has shown promise in enhancing the nutritional value of *amala*, the effect of soybean flour fortification on the starch properties of the resultant flour blends has not been extensively studied. This study intended to find out the influence which the flour compositing would have on such starch properties as pH, bulk density, water/oil absorption capacity, pasting properties and structural changes as depicted by Fourier transform infrared (FTIR) spectroscopy. Therefore, the study investigated the changes in the properties of starch isolated from the composited flour samples particularly the one involving yam flour fortification with soybean flour intended for the production of 'amala'.

2. Material and Methods

2.1. Materials

The dried yam chips (*Gbodo*) and soybean used for the study were procured from Oja-Oba market in Akure, Ondo State, Nigeria. All the reagents used in the study were of analytical grade including potassium hydroxide (KOH) and potassium bromide (KBr) pellets.

2.2. Production of soybean flour

Soybean flour was prepared by the method as described by Sanful and Darko (2010). Soybean was sorted to remove particles, defective seeds and stones before cleaning thoroughly were done in clean tap water. The seeds were boiled for 30 mins and drained so as to inactivate the trypsin inhibitors followed by dehulling using manual method (i.e., hand rubbing within two palms). After dehulling, the soybean seeds were dried in a hot air oven at 60°C for 18 h. After drying the soybean hulls were removed by winnowing, the dried samples were milled to fine powder and sieved through a standard sieve of 400 µm particle size. The flour was packaged in a Ziploc bag and stored in dry environment before subsequent usage.

2.3. Production of yam flour

Yam flour was produced following the method as described by Fiuro (2005). The yam chips were sorted to remove sand, dirt and other adhering materials. The yam chips were milled by using a hammer mill machine and the yam flour was sieved through a standard sieve of 400 µm particle size, packaged in a Ziploc bag and stored in dry environment before subsequent usage.

Table 1. Blending ratio of yam and soybean flour.

S/N	Blend Codes	Percentage of components (%)	
		Yam flour	Soybean flour
1	LF01	100	0
2	LF02	90	10
3	LF03	80	20
4	LF04	70	30
5	LF05	60	40
6	LF06	50	50

2.4. Flour blend formulation

The yam flour and soybean flour was blended together using a warring blender as described by Malomo et al. (2012) at different ratios of 100:0, 90:10, 80:20, 70:30, 60:40 and 50:50% (yam flour: soybean flour; w/w). The flour blends were labelled as LF01, LF02, LF03, LF04, LF05 and LF06, respectively. Table 1 shows the details of the flour blend formulation.

2.5. Isolation of starch from flour samples

The procedure of Izuagie et al. (2012) was used for the isolation of starch from the flour samples. About 2 kg of the blended batches of yam and soybean flour were respectively weighed and soaked into 14 litres of potable water and solubilized in a big plastic bucket. The suspension was thereafter sieved in a 250-µm sieve bowl and the starch milk allowed to settle by refrigerating at 8±2°C for 8 h. This was followed by decantation of the supernatant and dewatering of the starch sediment using a muslin cloth. The dewatered starch cakes from each of the sample batches were spread on drying trays, and put into the hot air oven at 55°C to dry for 24 h. After drying, the samples were cooled and pulverized. The starch powder was then put into a Ziploc bag for storage.

2.6. Starch yield from the flour samples

The starch yield was evaluated as described by Awolu and Olofinlae (2016). It is the percentage ratio of starch recovered after extraction to the initial flour sample. The starch yield was estimated using the equation below (Eq. 1).

$$\text{Starch yield (\%)} = \frac{\text{Weight of starch (g)}}{\text{Weight of flour sample (g)}} \times 100 \quad (1)$$

2.7. pH determination of starch samples

The pH was determined according to the method described by Ashogbon and Akintayo (2014). The pH meter (model WPA CD70, India) was calibrated with KOH buffer solutions of pH 7.0 and 4.0 before the measurements. The starch sample (5 g) was weighed in triplicate into a beaker, mixed with 20 mL of distilled water. The resulting suspension was stirred for 5 min and left to settle for 10 min. The pH of the starch solution was determined using the calibrated pH meter.

2.8. Determination of bulk density of starch samples

The bulk density of each of the samples was determined according to Nwosu et al. (2014). A weighed sample (10 g) was put in a calibrated 50 mL measuring cylinder. Then the bottom of the cylinder was tapped repeatedly unto a firm pad on a laboratory bench until a constant volume was observed. The packed volume was recorded. The bulk density was calculated (Eq. 2) as the ratio of the sample weight to the volume occupied by the sample after tapping.

$$\text{Bulk density (g/cm}^3\text{)} = \frac{\text{Weight of sample (g)}}{\text{Volume of sample (cm}^3\text{)}} \quad (2)$$

2.9. Determination of water and oil absorption capacities

The water absorption capacity (WAC) and oil absorption capacity (OAC) were determined as described by [Omowaye-Taiwo et al. \(2015\)](#). One gramme (1 g) of each sample was transferred into 10 mL distilled water ($\rho = 1 \text{ g/mL}$) and Power[®] vegetable oil ($\rho = 0.92 \text{ g/mL}$) separately for water and oil absorption capacities, respectively. The resulting suspension was stirred with a magnetic stirrer for 5 min. The content was centrifuged at $2,500 \times g$ for 30 min and the supernatant decanted and the volume obtained was measured. The water absorbed by the flour was calculated as the difference between the initial water used and the volume of the supernatant obtained after centrifuging while that of the oil absorbed was also measured but the density of the oil was used to obtain the weight absorbed. The water/oil absorbed by the flour was expressed as a percentage.

$$\text{Water absorption capacity (\%)} = \frac{W_w - W_i}{W_i} \times 100 \quad (3)$$

where, W_w = Final weight after water absorption and W_i = Initial weight of the flour.

$$\text{Oil absorption capacity (\%)} = \frac{W_o - W_i}{W_i} \times 100 \quad (4)$$

where, W_o = Final weight after oil absorption and W_i = Initial weight of the flour.

2.10. Determination of swelling power of starch samples

The swelling power of the starch samples was determined using the method of [Leach et al. \(1959\)](#). One gram of sample was separately weighed into centrifuge tubes and 50 mL distilled water added respectively. These tubes were immersed in a water bath at temperature 60°C for 30 mins and thoroughly stirred using a stirrer. The tubes were removed, cooled to room temperature and centrifuged at 3500 rpm for 20 min. The supernatant was carefully transferred into a conical flask; 5 mL out of it was transferred into petri-dish, dried in oven at 110°C for 4 h. The weight of the pastes was determined and used to calculate the swelling power as per gram of sediment paste per gram of starch sample.

$$\text{Swelling power (g/g)} = \frac{\text{Weight of wet sediment (g)}}{\text{Weight of dry matter in the gel (g)}} \quad (5)$$

2.11. Evaluation of structural changes in the starch samples using Fourier transform infrared (FTIR) spectroscopy

The influence of yam-soybean flour blending on the structural changes in the isolated starch samples was studied using fourier transform infrared (FTIR) spectroscopy (Shimadzu IR affinity-I 8000 FT-IR spectrometer, Japan). The study was carried out under dry air at room temperature ($29 \pm 2^\circ\text{C}$) using potassium bromide (KBr) pellets. One milligram (1 mg) sample was mixed with 300 mg of KBr supplied with FTIR unit. The samples were pressed directly onto attenuated reflectance KBr crystal into the sampling unit. Spectra were scanned in the frequency range of 4000 and 400

cm^{-1} at the resolution of 4 cm^{-1} and a maximum source aperture ([Bhat & binti-Yahya, 2014](#); [Zhao et al., 2014](#)).

2.12. Statistical analysis

Determinations were made in triplicates and data generated were subjected to One-way Analysis of Variance (ANOVA) using Statistical Package for Social Sciences (SPSS) version 23.0. The means were separated using New Duncan Multiple Range Test (NDMRT) at 95% confidence level ($p \leq 0.05$).

Table 2. Starch yield and pH of the isolated starch samples.

Samples	Starch yield (%)	pH
LF01	68.5	5.4
LF02	67.9	5.4
LF03	60.4	4.5
LF04	52.5	4.7
LF05	43.8	4.7
LF06	38.5	4.6

LF01 = 100% yam flour (Control), LF02 = 90% yam flour and 10% soy flour, LF03 = 80% yam flour and 20% soy flour, LF04 = 70% yam flour and 30% soy flour, LF05 = 60% yam flour and 40% soy flour, LF06 = 50% yam flour and 50% soy flour.

3. Results and Discussion

3.1. Percentage yield and pH values of isolated starch from the flour samples

The percentage starch yield from the isolation of starch samples is presented in [Table 2](#). The starch yield ranged from 38.5 – 68.5%. Sample LF01 (100% yam flour) had the highest yield at 68.5% while sample LF06 (blend of 50% yam flour and 50% soybean flour) had the least percentage starch yield. The result showed that the starch yield was decreasing as the concentration of soybean flour was increasing in the formulation. Starch yield is an important parameter in determining the efficiency of the starch extraction process as it affects the overall quantity and quality of the extracted starch. The implication of this observation is that increasing the ratio of soybean flour in the mixture would lead to the lowering of overall starch yield.

The pH of the isolated starch samples is also presented in [Table 2](#). The pH values of the starch samples ranged from 4.6 to 5.4. Sample LF06 (50% yam flour and 50% soybean flour) had the lowest pH (4.6) while LF01 (100% yam flour) had the highest pH value (5.4). These values showed that the starch samples were moderately acidic in nature. Again, it was observed that the pH values of the isolated starch samples became more acidic with an increase in the concentration of soybean flour in the formulation. The pH of any starch is a significant asset when it comes to industrial applications because pH is being used generally to indicate the acidic or alkaline properties of liquid media ([Ashogbon & Akintayo, 2012](#)). The observed decrease in pH with increasing proportion of soybean flour in the mixture could be attributed to the acidic nature of soybeans as soybeans are known to have a lower pH compared to yam flour ([Barine & Frankline, 2019](#)).

Table 3. Functional properties of starch samples.

Samples	Bulk Density (g/cm ³)	Swelling Power (g/g)	Water Absorption Capacity (%)	Oil Absorption Capacity (%)
LF01	0.84 ^d ±0.01	2.70 ^a ±0.01	267.76 ^d ±2.01	190.27 ^d ±6.86
LF02	0.79 ^e ±0.01	2.90 ^b ±0.01	265.39 ^e ±4.01	181.17 ^e ±2.15
LF03	0.77 ^d ±0.01	3.13 ^c ±0.01	263.00 ^d ±3.01	162.73 ^d ±3.06
LF04	0.71 ^e ±0.01	3.33 ^d ±0.01	260.61 ^e ±2.02	154.43 ^e ±2.21
LF05	0.66 ^b ±0.01	3.51 ^e ±0.01	259.58 ^b ±1.01	130.43 ^b ±2.06
LF06	0.61 ^a ±0.01	3.69 ^f ±0.01	246.98 ^a ±2.01	120.40 ^a ±1.11

Values are means ± standard deviation. Means with different alphabetical superscripts in the same column are significantly different ($p \leq 0.05$). LF01 = 100% yam flour (Control), LF02 = 90% yam flour and 10% soy flour, LF03 = 80% yam flour and 20% soy flour, LF04 = 70% yam flour and 30% soy flour, LF05 = 60% yam flour and 40% soy flour, LF06 = 50% yam flour and 50% soy flour.

Table 4. Pasting properties of starch samples.

Samples	Peak viscosity (cP)	Trough (cP)	Breakdown viscosity (cP)	Final Viscosity (cP)	Setback (cP)	Peak time (min)	Pasting temperature (°C)
LF01	7716	5890	1826	9516	3626	5.13	82.2
LF02	2745	2380	365	3962	1482	5.27	82.4
LF03	5561	4598	963	8032	3434	5.28	83.1
LF04	2963	2667	318	3845	1178	5.29	83.1
LF05	5743	4737	1006	8120	3383	5.31	84.1
LF06	3188	2797	391	4075	1278	5.45	85.1

cP = centipoise. LF01 = 100% yam flour (Control), LF02 = 90% yam flour and 10% soy flour, LF03 = 80% yam flour and 20% soy flour, LF04 = 70% yam flour and 30% soy flour, LF05 = 60% yam flour and 40% soy flour, LF06 = 50% yam flour and 50% soy flour.

3.2. Functional properties of the isolated starch from the flour samples

The functional properties of the isolated starch samples are presented in Table 3. The bulk density of starch samples ranged from 0.61-0.84 g/cm³ with sample LF01 (100% yam flour) having the highest value (0.84 g/cm³) and sample LF06 (50% yam flour and 50% soybean flour) having the lowest value (0.61 g/cm³). The result of the bulk density showed that there was no significant difference between sample LF01, LF02 and LF03 but there was significant difference ($p \leq 0.05$) between LF03 and LF04. The general observation here was that the bulk density of the starch samples tended to decrease with an increase in the concentration of soybean in the formulation. Bulk density plays a role in flour packaging as less weight would be packaged in a specific volume of container with flour of lower bulk density (Bolade & Adeyemi, 2012). The implication of this observation is that the incorporation of soy flour into yam flour has a tendency of lowering the overall bulk density (Bolade & Adeyemi, 2010).

The swelling power of the isolated starch samples ranged from 2.70 – 3.69. Sample LF01 (2.70 g/g) had the lowest swelling power followed by LF02 (2.90 g/g) and both samples were significantly different at $p < 0.05$. However, sample LF06 had the highest swelling power at 3.69 g/g. The swelling power of the starch samples increased significantly ($p < 0.05$) as the proportion of soy flour increased. The swelling power is an indication of presence of amylose in the starch. The variation in the swelling power indicates the degree of exposure of the internal structure of the starch to the action of water. The variability of the swelling power amongst these starch samples is likely due to extraction

processes which influence the internal bond strength present in the starch granules, thus, influencing swelling power (Paraginski et al., 2014).

The water and oil absorption capacities of the isolated starch samples are also shown in Table 3. The water absorption capacity (WAC) of the starch samples ranged from 246.08 to 268.06%, with sample LF06 having the lowest value while sample LF01 had the highest value.

The WAC was generally observed to decrease with an increase in the concentration of soybean flour in the flour formulation. Water absorption capacity is the ability of flour to absorb water and swell for improved consistency in food. It is desirable in food systems to improve yield and consistency and give body to the food (Osundahunsi et al., 2003). The observation from the result implies that the incorporation of soy flour into yam flour has a tendency to lowering the overall water holding capacity. This effect was probably due to loosen association of amylose and amylopectin in the native granules of starch and weaker associative forces involved in maintaining the granular structure (Lorenz et al., 2020).

The oil absorption capacity (OAC) of the isolated starch samples also revealed a gradual decrease in the values as the concentration of soybean flour inclusion was increasing. Sample LF01 (100% yam flour) had the highest value of OAC (190.27%) while sample LF06 (50% yam flour and 50% soybean flour) had the lowest OAC value (120.4%). The oil absorption capacity (OAC) is important as oil acts as a flavour retainer and improves the mouthfeel of foods (Kinsella, 1976). It is also an indication of the rate at which protein binds to fat in food formulations (Omimawo & Akubor, 2012; Thomas et al., 2014).

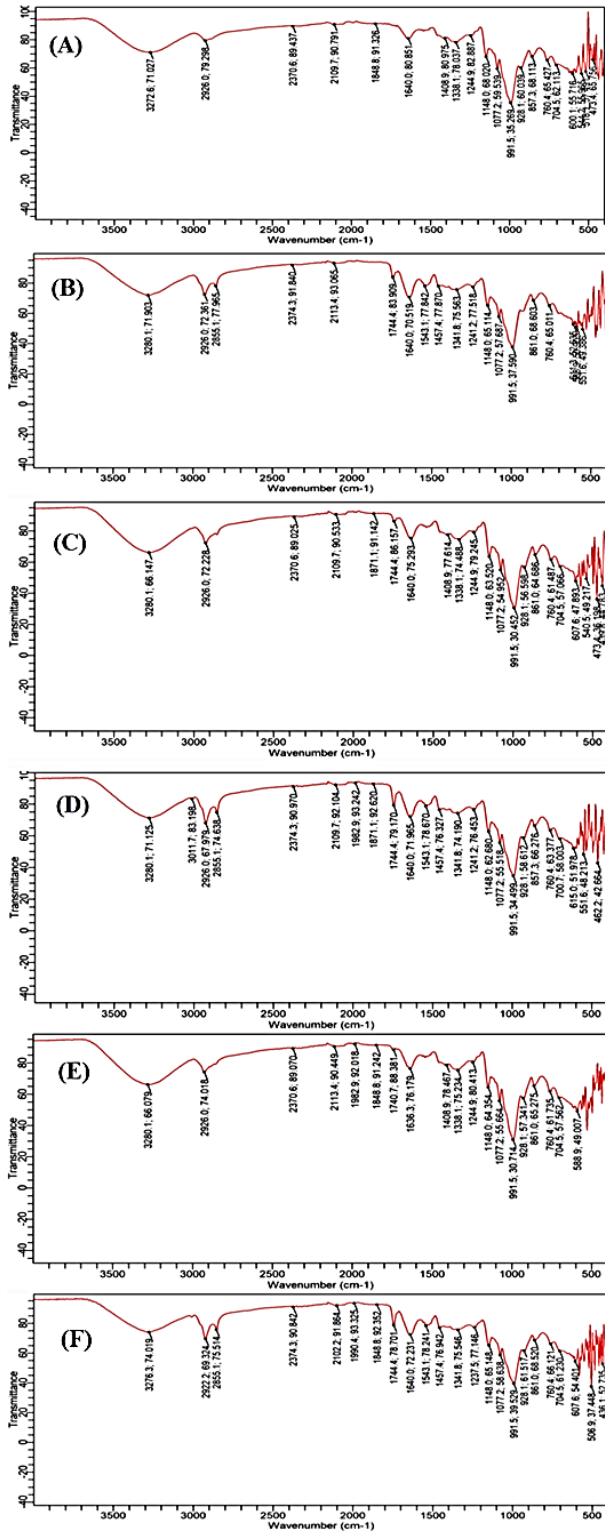


Fig. 1. FTIR spectra of starch samples. [A] = 100% yam flour (LF01), [B] = 90% yam flour & 10% soy flour (LF02), [C] = 80% yam flour & 20% soy flour (LF03), [D] = 70% yam flour & 30% soy flour (LF04), [E] = 60% yam flour & 40% soy flour (LF05) and [F] = 50% yam flour & 50% soy flour (LF06).

3.3. Pasting properties of the isolated starch from the flour samples

Table 4 shows the result for the pasting properties of isolated starch samples. The peak viscosities of different starch samples ranged between 2745 and 7716 cP. Sample LF01 had the highest peak viscosity (7716 cP) while sample LF02 had the lowest peak viscosity (2745 cP). The peak viscosities of the samples were generally observed to decrease with the presence of soybean flour in the formulation. Peak viscosity essentially demonstrates the ability of starch to swell freely before their physical breakdown (Sanni et al., 2004). The higher peak viscosity in the control sample (LF01) is an indication that the sample formed a thick paste after cooking and had the ability to withstand heating and shear stress. The time taken to reach peak viscosity by the starch samples also ranged between 5.13 and 5.45 min, with sample LF01 giving the shortest time while LF06 gave the longest time. The time to reach peak viscosity was increasing with increasing quantity of soy flour in the formulation. The decrease in the peak viscosity of starch samples may be attributed to a decreased rate of water absorption and starch granule swelling during heating by virtue of yam flour dilution with flour from soybeans. The implication of this occurrence is that the presence of soy flour in the composite formulation during *amala* preparation could lead to a decrease in the peak viscosity with concomitant slower cooking due to increased time in the attainment of peak viscosity (Awoyale et al., 2015).

The breakdown viscosity of the isolated starch samples generally decreased due to the presence of soybean flour in the formulation. The breakdown viscosity is regarded as a measure of the degree of disintegration of starch granules or paste stability during heating (Dengate, 1984). The implication of this observation is that the starch samples with lower breakdown values were less resistant to heat and shear force during heating and that there was more starch granule rupture which could therefore lead to a less stable cooked paste (Farhat et al., 1999).

The final viscosity values of the isolated starch samples showed highest value (9516 cP) in sample LF01 (100% yam flour) while other samples exhibited lower values due to the presence of soybean flour in the formulation. The final viscosity as a parameter indicates the ability to form a firm, visco-elastic paste or gel after cooking and cooling owing to re-association of starch granules (Newport Scientific, 1998).

The setback viscosity revealed that sample LF01 exhibited the highest value (3626 cP) while the presence of soybean flour in other samples led to the lowering of setback viscosity ranging between 1178 and 3434 cP. The setback viscosity is essentially an index of the tendency of the cooked starch to harden on cooling due to amylose retrogradation (Adeyemi, 1989). It is a parameter being used as an index of retrogradation tendency of gel obtained from flour or starch cooking (Sandhu et al., 2007).

3.4. Structural changes in the isolated starch samples as depicted by Fourier transform infrared (FTIR) spectroscopy

The influence of compositing flour from yam and soybean on the structural changes in isolated starch samples is presented in Fig. 1(A-F). The spectral peaks exhibited by sample LF01 (100% yam flour) revealed that several functional groups were present in the sample at different wavenumber regions. These includes —OH group occurring at 3200-3600 cm^{-1} wavenumber region (Ismail et al., 1997); —C—H bonds of the —CH₂ or —CH₃ alkyl groups occurring at 2853-2962 cm^{-1} wavenumber region (Ikram et al., 2021); C=C bonds occurring at 2040-2250 cm^{-1} wavenumber region (Ismail et al., 1997); and C=O bonds occurring at 1700-1900 cm^{-1} wavenumber region (Thompson, 2018), among others. However, the inclusion of soybean flour on the spectral pattern of the isolated starch samples revealed the appearance of additional functional groups. The disappearance of some of the existing functional groups and the shifting of the wavenumber regions some spectral peaks were located or occurrence of identical spectral peaks.

The appearance of new functional groups occurred in samples LF02 (90% yam flour and 10% soybean flour), sample LF04 (70% yam flour and 30% soybean flour), sample LF05 (60% yam flour and 40% soybean flour), and sample LF06 (50% yam flour and 50% soybean). The newly introduced functional groups include the cumulated double bonds of the (>C=C=CH₂) unit of the conjugated aliphatic and aromatic compounds which has an assigned wavenumber regions of 1900-2000 cm^{-1} (Larkin, 2011). Another functional group that appeared in the isolated starch samples from the composited flour was the C—N stretching and —N—H bending vibration of the amide II origin which has an assigned wavenumber regions of 1530-1560 cm^{-1} (Stuart, 2004). The functional group that was observed to have disappeared among the spectral peaks was CH₂ bending vibration of the O=C=CH₂ and CH₂—C≡N units of the aliphatic and aromatic compounds which has an assigned wavenumber regions of 1405-1445 cm^{-1} (Larkin, 2011). This disappearance occurred in samples LF02, LF04 and LF06 respectively. The shifting of the wavenumber positions where some spectral peaks were located did occur in all the starch samples and this occurrence is, most probably, because of the weakening or strengthening of the chemical bonds in the starch molecules occasioned by flour blending. Previous observation had postulated that the positions of absorption of infrared radiation might be shifted due to factors such as resonance electron withdrawing and donating effects, steric interaction and/or hydrogen bonding (Thompson, 2018) within the functional groups of starch molecules. In the case of identical spectral peaks in the starch samples, it is a reflection of the functional groups being structurally similar (Thompson, 2018). The wavenumber region of 500-1000 cm^{-1} has been regarded as the 'fingerprint' region for all samples and is a region that shows the true identity of any given sample (Coates, 2000).

4. Conclusion

The fortification of yam flour with soybean flour had a significant effect on the properties of the resultant starch isolated from the blends. The results indicated that the inclusion of soybean flour in the formulation caused the resultant starch samples to exhibit reduction in the values of starch yield, pH, bulk density,

water absorption capacity, oil absorption capacity, peak viscosity, breakdown viscosity, final viscosity, and setback viscosity. However, some factors that exhibited increased values were swelling power, peak time, and pasting temperature. The Fourier transform infrared (FTIR) spectroscopy of the starch samples exhibited noticeable differences in the spectra pattern in terms of shifting in the position of the wavenumber as well as the appearance of new functional groups. Finally, the information provided in this study gave more knowledge on the influence of yam and soybean flour blend on the starch properties of yam-based doughmeal.

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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