






Transformation and Agroindustry

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# Evaluation of Proximate, Structural, Morphological, Physicochemical and *In Vitro* Digestibility Properties of Yam and Sweet Potato Flour Blends

Evaluación de las propiedades proximales, estructurales, morfológicas fisicoquímicas y de digestibilidad *in vitro* en mezclas de harinas de ñame y batata

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**Abstract:** Yam (*Dioscorea spp.*) and sweet potato (*Ipomeas batata*) flours must be treated through modification processes because they have limitations in their physicochemical and structural properties for use in agri-food matrices. This study aimed to evaluate the effect of different proportions of flours on the proximate, structural, morphological, physicochemical, pasting, and *in vitro* digestibility properties of yam and sweet potato flour blends. Flours were obtained by drying the tubers with hot air at 55 °C, and mixtures were prepared at 25:75, 50:50, and 75:25 w/w sweet potato and yam flour proportions, respectively. The yam starch granules were spherical, oval, irregular, and larger than those of sweet potatoes, which are ovoid and flared. Furthermore, yam and sweet potato granules presented with type-B and type-A diffraction patterns, respectively, which, together with the starch content, amylose, non-starch components, and their molecular interactions, influenced the decrease in the molecular order and degree of crystallinity of the flour blends, without altering the morphological characteristics of the simple flours. Additive and non-additive effects were observed on flour blends' swelling power and water absorption capacity. Likewise, breakdown and setback viscosity increased, which decreased thermal stability, increased the tendency to retrogradation, and elevated the fractions of rapidly digestible starch and resistant starch. These results suggest the high potential of flour blends in preparing baked products from the Caribbean Region, such as white bread, cookies, and cakes.

**Keywords:** tubers, hydrothermal treatments, resistant starch, solubility, gelatinization.

**Resumen:** Las harinas de ñame (*Dioscorea spp.*) y batata (*Ipomeas batata*) deben intervenir mediante procesos de modificación por las limitaciones en sus propiedades fisicoquímicas y estructurales para su utilización en matrices agroalimentarias. Se evaluó el efecto de diferentes proporciones de harinas en las propiedades proximales, estructurales, morfológicas, fisicoquímicas y de digestibilidad *in vitro* de mezclas de harinas de ñame y batata. Estas se obtuvieron mediante secado de los tubérculos con aire caliente a 55 °C y las mezclas se prepararon en proporciones de 25:75, 50:50 y 75:25 p/p de harinas de batata y ñame, respectivamente. Los gránulos de almidón de ñame eran esféricos, ovalados, irregulares y de mayor tamaño que los gránulos ovoides del almidón de batata. También, presentaron patrones de difracción tipo B y A, respectivamente, los cuales, junto con el contenido de almidón, la amilosa, los componentes no amiláceos y sus interacciones supramoleculares, disminuyeron el orden molecular y el grado de cristalinidad de las mezclas, sin alterar las características morfológicas de las harinas simples. Se observaron efectos aditivos y no aditivos en el poder de hinchamiento y la capacidad de absorción de agua en las mezclas. La viscosidad de rompimiento y asentamiento aumentó, lo cual disminuyó la estabilidad térmica y aumentó la tendencia a la retrogradación. No obstante, se obtuvo un material amiláceo con mayor contenido de almidón de rápida digestión y almidón resistente. Estos resultados sugieren un alto potencial de las mezclas como ingredientes en productos horneados de la Región Caribe como pan blanco, galletas y tortas.

**Palabras claves:** tubérculos, tratamiento hidrotérmico, almidón resistente, solubilidad, gelatinización



## Introduction

Tropical tubers, such as cassava, sweet potato, and yam, are food sources of great importance due to their high carbohydrate content that favors a high energy intake. They can play an important role in sustainable food development, implementing processing technologies that allow their optimal use for the benefit of the population (Hasmadi et al., 2020). However, the lack of knowledge of the physicochemical properties of yams and sweet potatoes has restricted their use for fresh consumption and traditional preparations (Arroyo-Dagobeth et al., 2023). In addition, the lack of technologies for using and industrializing these starchy raw materials favors post-harvest losses, estimated at 25 % of national production. Since these tubers can be used as raw material in transformation processes to produce flour, this approach could solve the problem of losses and waste (Araújo & Pena, 2020). Previous studies have found that in Asian countries, tuber flours can be used to prepare many food products, such as bread, noodles, and cakes (Zou et al., 2021b). In contrast, in Colombia, the production of tuber flours comes mainly from the cultivation of cassava, which is basically for animal consumption. There are also significant scientific gaps regarding the quality of root or tuber flours intended for human consumption.

Tuber flours, being materials with high starch content, are raw materials of great importance in the industry due to their properties as thickening agents, gelling agents, and stabilizers and their capacity to act as fat substitutes in agri-food applications such as the production of meat products, bakery products, and candies (De Oliveira et al., 2018). However, in the native state, these properties cannot be exploited as they are susceptible to extreme processing conditions, such as high temperatures and shear rates (Oyeyinka et al., 2021). These undesirable characteristics can be improved by modification methods involving chemical, physical, biological, or simultaneous processes. However, clean label technologies are the most widely used because they do not use or generate chemical substances but result in a product with characteristics for specific industrial applications, especially in food processing (Chen et al., 2020).

In this sense, flour blends and composite flours are a form of physical modification that elaborates mixtures between different species of tubers, cereals, and legumes with or without adding wheat flour. The structure of starch, which is the main component in these matrices and is responsible for defining the applications and functionalities of the flours, can be influenced. In addition, it has been demonstrated that the combination of these blends with other raw materials allows for obtaining products with physicochemical properties parallel to those of wheat flour, thus addressing the current challenges in the production of bakery and confectionery foods (Ugwuona et al., 2021; Waterschoot et al., 2015).

Kehinde et al. (2020) analyzed the nutritional composition, physicochemical, and pasting properties of yellow yam (YY) and white bean (WB) flour mixtures. The research results established an optimum substitution of 94.11 YY/5.89 WB, from which a high content of crude protein, starch, and amylose was achieved. However, there was a decrease in total carotenoid content, water absorption capacity, swelling power, and paste properties such as maximum viscosity and stability. However, it showed a high potential for producing food products such as extruded snacks and cooked pasta. Meanwhile, Utami et al. (2018) reported increased water and

oil absorption capacity, mineral content, carbohydrate, amylose, antioxidant activity, and total phenolic compounds when evaluating yam (*Dioscorea alata*) and fava bean flour mixtures. These results agree with those reported by Chandra et al. (2015), who obtained higher swelling capacity, water absorption, oil, emulsion activity and stability, foaming capacity and stability, and gelatinization temperature by replacing wheat flour with rice flour, green gram flour, and potato flour in cookie making.

According to research, flour blends and composite flours have a high potency as a substitute for wheat flour. However, the properties of flour blends are related to several factors, including the variety and proportion of each type of flour, amylose content and leaching, swelling power, and relative size of starch granules (Hornung et al., 2017). Based on the above, the processing of yam and sweet potato flour blends will additively and non-additively affect their physicochemical and *in vitro* digestibility properties. Still, research on flour blends from non-conventional sources reported in the literature is scarce. Therefore, the following study aims to evaluate the effect of different proportions of flours on the proximate, structural, morphological, physicochemical, pasting, and *in vitro* digestibility properties of native yam and sweet potato flour blends to enhance their application in the food industry.

## Materials and Methods

### Materials

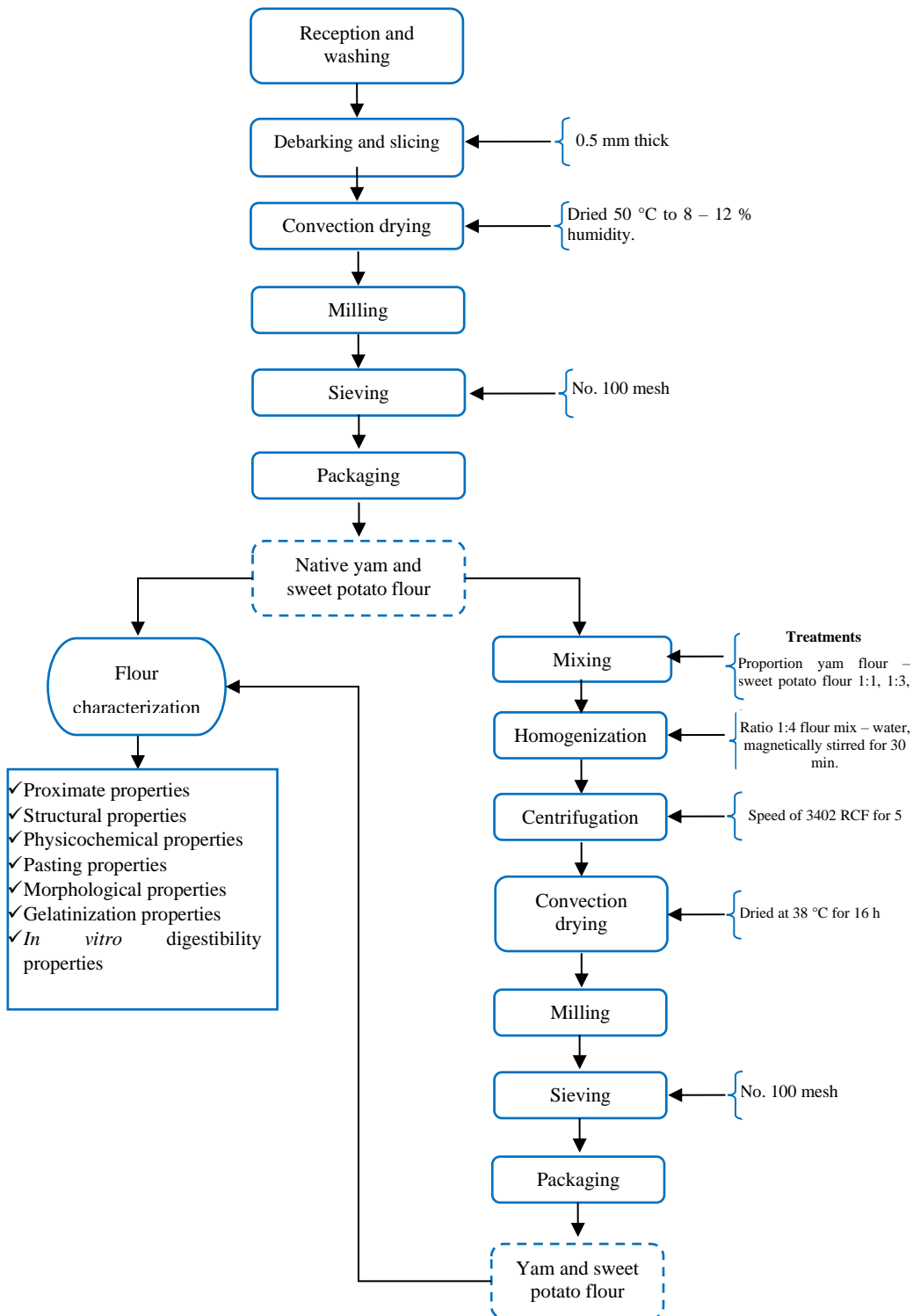
Yam (*Dioscorea alata* cv. Criollo) and sweet potato (*Ipomoea batatas* cv. Agrosavia Aurora) were supplied by ANPPY. Bacterial  $\alpha$ -amylase (Lyquozyme Supra 2,2 Xr, Denmark), fungal amyloglucosidase (DextrozimerGA, Denmark), and pancreatic  $\alpha$ -amylase were donated by Dupont® and Novozymes®. Potato amylose (A0512, USA) and corn amylopectin (10120, USA) standards were purchased from Sigma-Aldrich®.

### Methods

The process of obtaining native yam and sweet potato flours and preparing the mixtures was carried out according to the stages described in Figure 1.

#### Obtaining Native Flours

The washed, peeled, and sliced tubers (0.5 mm thick) were dried by convection in an oven (Binder, FD-115, Germany) at 50 °C until reaching a moisture content between 8–12 % (w/w; Mustapha et al., 2019). Subsequently, a grinding process was performed in a multipurpose mill (Tecnal, TE-631/4, Brazil), followed by a sieving operation using a No. 100 mesh (~150  $\mu$ m). The native flours obtained were packaged in hermetically closed laminated bags.



**Figure 1.** Process for obtaining native yam and sweet potato flours and preparing blends. Source: Prepared by the authors.

## Preparation of Flour Blends

The native flour mixtures were prepared using the wet method proposed by Hornung et al. (2017) with slight modifications. Ratios of 1:1, 1:3, and 3:1 w/w yam and sweet potato flour were established, respectively. Then, 100 g of the flour blends was suspended in distilled water (400 ml), magnetically stirred for 30 min, and centrifuged at a speed of 3,402 RCF for 5 min. The supernatant was removed, and the precipitate was dried at 38 °C for 16 h. Finally, the flour was milled in a multipurpose mill (Tecnal, TE-631/4, Brazil), sieved (No. 100 mesh), and packaged in laminated bags.

## Proximate Properties

The flour samples were analyzed for protein, ash, fat, fiber, carbohydrate, and moisture by the methods of the Association of Official Analytical Chemists (AOAC, 2005), as follows: protein by Kjeldahl method (AOAC 955.04/90), ash by gravimetric method (AOAC 923.03), fat by Soxhlet extraction method (AOAC 920.39/90), crude fiber by digestibility method (AOAC 985.29/90), and moisture by gravimetric method (AOAC 964.22/90). Total carbohydrate content was determined by difference.

## Starch Content and Amylose Content

Starch content (SC) was determined by enzymatic hydrolysis. A sample of 200 mg of flour was suspended in 25 ml of citrate buffer; the sample was dextrinized with 100  $\mu$ l of  $\alpha$ -amylase at a temperature of 90 °C for 15 min, and 200  $\mu$ l of amyloglucosidase was added for 30 min at 60 °C. Finally, the concentration of reducing sugars was determined using the method proposed by Miller (1959). The apparent amylose content (AAC) was established by the spectrophotometric method in iodine staining from an amylose curve (0–100 % w/w; Figueroa-Flórez et al., 2023). The curve was prepared using standard mixtures of pure potato amylose and fractionated corn amylopectin (Sigma Aldrich, USA).

## Degree of Molecular Order

FTIR-ATR spectra were acquired using an infrared spectrophotometer with an attenuated total reflectance module. Each spectrum was recorded with a resolution of 8  $\text{cm}^{-1}$  and 32 readout scans in the 450–4000  $\text{cm}^{-1}$  range. The band ratios 1047/1022  $\text{cm}^{-1}$  and 995/1022  $\text{cm}^{-1}$  were used to estimate the short-range molecular order (OM1 y OM2, respectively; Yu et al., 2021).

## Morphological Properties

The native and modified flours were observed by scanning electron microscopy (SEM) using a microscope (JEOL, JSU-5600 LV, Japan) under operating conditions of 15 KV, 30 mA, and a 2000X amplitude range. The granule size was analyzed under an amplitude range of 500X and a scale of 20  $\mu\text{m}$  using Image J software (JVM, National Institutes of Health, USA; Figueroa-Flórez et al., 2019). Birefringence capacity was observed through a binocular microscope (C-

B10+, Optika, Italy) under polarized light fields; images were obtained using a digital camera (OptikamB10, Italy; Figueroa-Flórez et al., 2023).

### **Diffraction Patterns and Degree of Crystallinity**

Diffraction patterns were determined using a diffractometer (Panalytical, X'Pert MPD, Switzerland) operated at 1.8 kW and 40 mA. Spectra were acquired in the 10–30 ° range at a scan rate of 2 °/min and a sampling interval of 0.02 ° (Arroyo-Dagobeth et al., 2023). The degree of crystallinity (DC) was estimated as the ratio of absorption area peaks (crystalline zone) over the total area of the diffractogram using numerical integration methods and MATLAB software (MathWorks, R2019, USA).

### **Physicochemical Properties**

To determine swelling power (SP), 0.5 g of sample dry basis (db) was suspended in 12.5 ml of distilled water preheated to 70 °C. The slurry was kept at 70 °C and stirred every 10 min for 30 minutes. Water was added at room temperature for cold water solubility (CWS). The dispersion was then centrifuged at 3,402 g for 15 min. The weight of the precipitate was recorded. The supernatant was separated and dried at 90 and 110 °C for SP and CWS, respectively (Anderson et al., 1970; Eastman & Moore, 1984). Water absorption capacity (WAC) and oil absorption capacity (OAC) were determined by centrifugation according to the method described by Dereje et al. (2020).

### **Pasting Properties**

A rheometer (Anton par, MCR 302, Austria), a cell for analysis of starch suspensions (C-ETD160/ST), and a blade spindle (Anton Paar, ST24-2D/2V/2V-30, Austria) were used according to the method adopted by Arroyo-Dagobeth et al. (2023) with slight modifications. Suspensions of 8 % w/v flours were heated to 50 °C for 1 min, then to 95 °C for 7.5 min and held steady at 95 °C for 5 min, then cooled to 50 °C for 7.5 min, and finally held at 50 °C for 2 min. Viscosities were recorded in centipoise (cP).

### **Gelatinization Properties**

Gelatinization properties were determined through temperature sweeps according to the methodology used by Roy and Kumar (2023). A rheometer (Anton par, MCR 302, Austria) with parallel plate geometry, a diameter of 25 mm, and a gap distance of 1 mm was employed. After the linear viscoelasticity region (LVR) was validated, 30 % w/v flour suspensions were subjected to a temperature sweep between 30 and 90 °C at a constant frequency of 1.0 Hz and a deformation of 0.5 %. The values of the elasticity ( $G'$  [Pa]) and viscous modulus ( $G''$  [Pa]) were obtained during the process. The results were processed using RheoCompass software (v1.12, Anton-Paar, Austria).

### ***In vitro* Digestibility Properties**

The fractions of rapidly digested starch (RDS), slowly digested starch (SDS), and resistant starch (RS) were estimated by the *in vitro* digestibility method proposed by Englyst et al. (1992) with slight modifications. A starch sample (1 mg) was suspended in 0.1 M sodium citrate buffer solution (pH = 5.2) and subjected to gelatinization for 20 min at 90 °C. The gelatinized samples were hydrolyzed by adding 1 mL of an enzyme solution prepared with 54 mL of pancreatic  $\alpha$ -amylase (0.15 % w/v) and 6 mL of fungal amyloglucosidase (140 AGU/mL). Biocatalytic activity was performed at 37 °C for 120 min and inactivated using 2 mL of 90 % ethyl alcohol. Aliquots were taken at 20 and 120 min and centrifuged at 3402 RCF for 6 min to separate the supernatant and quantify the glucose concentration through the method proposed by Miller (1959).

### **Experimental Design and Statistical Analysis**

A one-factor categorical design was established with five (5) levels corresponding to the mixing ratio, as described in Table 1. The results were expressed as the mean of three replicates  $\pm$  standard deviation, analyzed by analysis of variance (ANOVA) and Tuckey's test for the mean difference at a significance level of 5 %. The data were processed using Statgraphics statistical software (Centurion XVI, Statgraphics Inc., USA).

**Table 1.** Experimental design implemented for the preparation of flour mixes

<b>Treatment</b>	<b>Nomenclature</b>
Native sweet potato flour	NSF
Native yam flour	NYF
Blend 1: yam flour (75 %) and sweet potato flour (25 %)	FB1
Blend 2: yam flour (50 %) and sweet potato flour (50 %)	FB2
Blend 3: yam flour (25 %) and sweet potato flour (75 %)	FB3

Source: Prepared by the authors.

## **Results and Discussion**

### **Proximate Properties**

The proximate composition of yam and sweet potato flours and blends is listed in Table 2. Native yam flour (NYF) showed higher protein, ash, and moisture content than native sweet potato flour (NSF); however, NSF showed higher fat, fiber, and carbohydrate composition. These differences in the proximate content of the flours may be related to botanical origin, variety, tuber maturity during harvest, and processing methods for obtaining the flours (Aprianita et al., 2014; Dereje et al., 2020). In the flour blends, protein and ash content decreased as the proportion of NSF increased. However, there were no significant differences between them, which could indicate the existence of non-additive behavior since they exhibit a non-proportional trend in that parameter concerning the individual values of NSF and NYF (Utami et al., 2018). Fat, fiber, and carbohydrate contents increased with an increasing proportion of

NSF in the mixture due to the high presence of these macromolecules in the NSF treatment, which suggested an additive behavior (Edun et al., 2019; Karigidi & Olaiya, 2021). Regarding moisture content, the evaluated treatments showed values within limits allowed by the NTC 5986, which could ensure greater microbiological stability of the product and probably extend its shelf life during storage, favoring its use in the food industry (Araújo & Pena, 2020).

**Table 2.** Proximate composition of native flours and their blends

Treatment	Proteins (%)	Ash (%)	Fat (%)	Fiber (%)	CHO (%)	Moisture (%)
NYF	2.77 ± 0.25 <sup>a</sup>	2.88 ± 0.08 <sup>a</sup>	1.58 ± 0.01 <sup>a</sup>	4.34 ± 0.17 <sup>a</sup>	79.54 ± 1.39 <sup>a</sup>	8.89 ± 0.24 <sup>a</sup>
NSF	3.64 ± 0.25 <sup>b</sup>	2.94 ± 0.12 <sup>a</sup>	0.43 ± 0.02 <sup>b</sup>	1.35 ± 0.26 <sup>b</sup>	79.75 ± 0.82 <sup>a</sup>	11.89 ± 0.69 <sup>bc</sup>
FB1	2.18 ± 0.01 <sup>c</sup>	1.35 ± 0.04 <sup>b</sup>	1.11 ± 0.04 <sup>c</sup>	1.47 ± 0.12 <sup>b</sup>	82.90 ± 0.61 <sup>b</sup>	10.99 ± 0.62 <sup>cd</sup>
FB2	1.89 ± 0.25 <sup>c</sup>	1.61 ± 0.02 <sup>c</sup>	0.58 ± 0.06 <sup>d</sup>	1.79 ± 0.09 <sup>b</sup>	81.42 ± 0.19 <sup>c</sup>	12.71 ± 0.05 <sup>b</sup>
FB3	1.75 ± 0.01 <sup>c</sup>	1.60 ± 0.03 <sup>c</sup>	1.38 ± 0.03 <sup>e</sup>	3.13 ± 0.16 <sup>c</sup>	81.63 ± 0.29 <sup>c</sup>	10.54 ± 0.35 <sup>d</sup>

NYF: native yam flour; NSF: native sweet potato flour; FB1: (75 %) yam and (25 %) sweet potato flour blends; FB2: (50 %) yam and (50 %) sweet potato flour blends; FB3: (25 %) yam and (75 %) sweet potato flour blends; CHO: carbohydrates.

Source: Prepared by the authors.

### Starch and Amylose Content

The starch (SC) and amylose (AAC) contents of the native flours and their blends are presented in Table 3. The results indicated significant differences among treatments. NYF had higher SC and AAC than NSF. This behavior may be associated with differences in species, varieties, genotypes, soils, and climatic conditions; these factors can influence the properties and functionality of starchy materials (Hasan et al., 2023; Shao et al., 2020). These results for SC and AAC are similar to those reported in the literature for yam and sweet potato flours, respectively (Dereje et al., 2020; Mustapha et al., 2019).

As for the blended flour, no significant differences were found in SC for NSF and FB3 and between FB1 and FB2. The results indicated that as the percentage of NSF in the flour blends increased, the SC and AAC decreased for all treatments. These behaviors relate to the low SC and AAC in NSF. Therefore, additive effects could be considered for these properties. Similar results were reported by Praseptiangga et al. (2018) and Utami et al. (2018), who found decreases in SC and AAC with increasing concentrations of jack bean (*Canavalia ensiformis*) and lima bean (*Phaseolus lunatus*) flours in yam and canna (*Canna edulis*) based composite flours.

### Degree of Molecular Order

The FTIR-ATR spectra shown in Figure 2 allowed us to examine functional groups and possible changes at the molecular level structure (short-range order) in flours and chain conformation and double helix order in flour blends (Chen et al., 2017b). Changes in absorption intensities were identified in the diagnostic zone (4,000–1,500 cm<sup>-1</sup>), highlighting bands 3,600–3,200 cm<sup>-1</sup> associated with O-H groups stretching vibrations in the molecule; the low absorbance in NSF



and flour blends concerning NYF could reveal a lower hydrophilic affinity. Similarly, peaks between 3,000–2,800  $\text{cm}^{-1}$  indicated stretching in C-H groups, possibly due to carbohydrates and lipids in flours. Also, peaks near the band 1,656–1,640  $\text{cm}^{-1}$  were associated with bending vibrations of bound water molecules in starch granules (Trela et al., 2020). Variations in absorption intensities could be associated with strong water-starch bonds in the molecules, leading to a higher energy requirement to undergo vibrational excitation (Arroyo-Dagobeth et al., 2023; Kumar et al., 2018).

**Table 3.** Starch content, amylose content, molecular order, and degree of crystallinity of native flours and their blends

Treatment	SC (%)	AAC (%)	OM <sub>1</sub>	OM <sub>2</sub>	DC (%)
NYF	77.32 ± 0.82a	19.48 ± 0.27a	0.78 ± 0.01a	1.25 ± 0.01a	24.87 ± 0.03a
NSF	54.35 ± 0.81b	11.95 ± 0.29b	0.83 ± 0.01b	1.18 ± 0.01b	35.41 ± 0.21b
FB1	67.63 ± 0.44c	17.50 ± 1.05ac	0.75 ± 0.01c	1.46 ± 0.01c	24.80 ± 0.06a
FB2	66.12 ± 0.19c	17.04 ± 0.61c	0.74 ± 0.01c	1.22 ± 0.02a	22.53 ± 0.05c
FB3	52.84 ± 0.47b	14.79 ± 0.92c	0.75 ± 0.01c	0.96 ± 0.01d	21.62 ± 0.13d

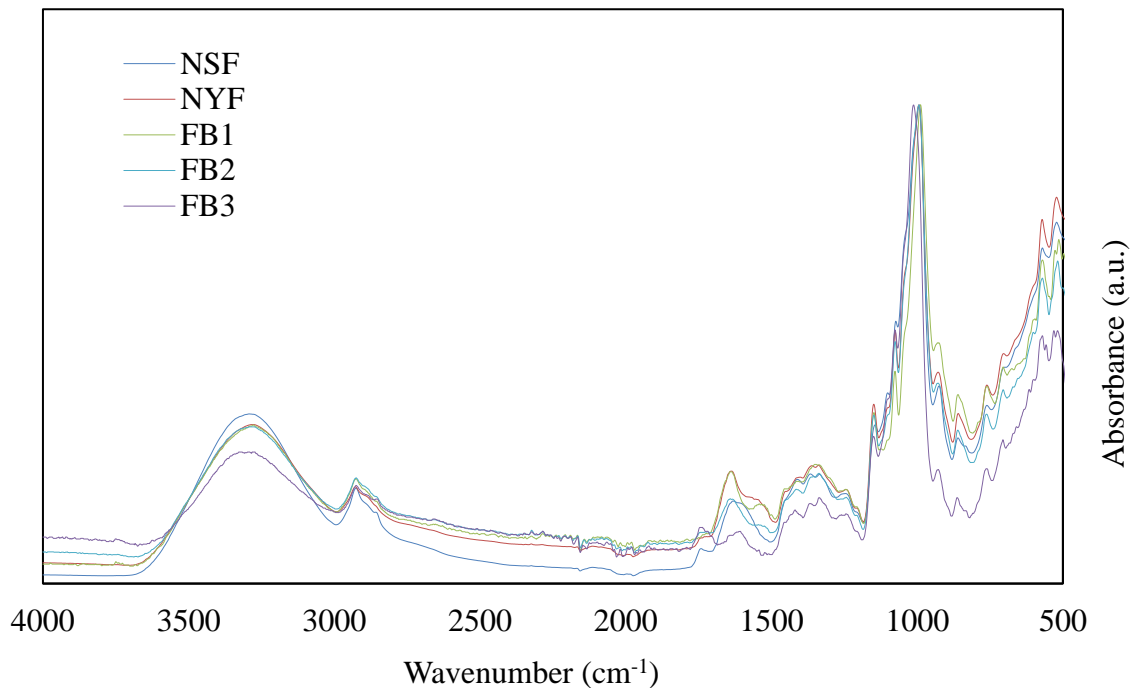
NYF: native yam flour; NSF: native sweet potato flour; FB1: (75 %) yam and (25 %) sweet potato flour blends; FB2: (50 %) yam and (50 %) sweet potato flour blends; FB3: (25 %) yam and (75 %) sweet potato flour blends; SC: starch content; AAC: apparent amylose content; OM<sub>1</sub>: ratio 1047/1022  $\text{cm}^{-1}$ ; OM<sub>2</sub>: ratio 995/1022  $\text{cm}^{-1}$ ; DC: degree of crystallinity.

Source: Prepared by the authors.

The fingerprint region showed peaks in the 1,047 and 1,022  $\text{cm}^{-1}$  bands, related to the ordered and amorphous crystalline regions of starch; the 995  $\text{cm}^{-1}$  band had C-OH bending vibrations associated with the interaction between water and the single helix crystal structure (Chen et al., 2017b). Table 2 presented a higher OM<sub>1</sub> for NSF concerning NYF, which could indicate a more ordered structure and, therefore, more crystalline structure; however, the OM<sub>2</sub> was lower in NSF concerning NYF, which could indicate a strong correlation between short-range hydrated and ordered structures (Yu et al., 2021). On the other hand, there was a decrease in OM<sub>1</sub> and OM<sub>2</sub> for flour blends as the proportion of NSF increased; however, there were no significant differences in OM<sub>1</sub>; this could suggest a lower short molecular order as a consequence of a non-additive behavior influenced by factors such as diffraction pattern, interactions between content and number of amylopectin and amylose branches, granular size, mixture proportion, and botanical origin (Arroyo-Dagobeth et al., 2023).

### Morphological Characteristics and Birefringence

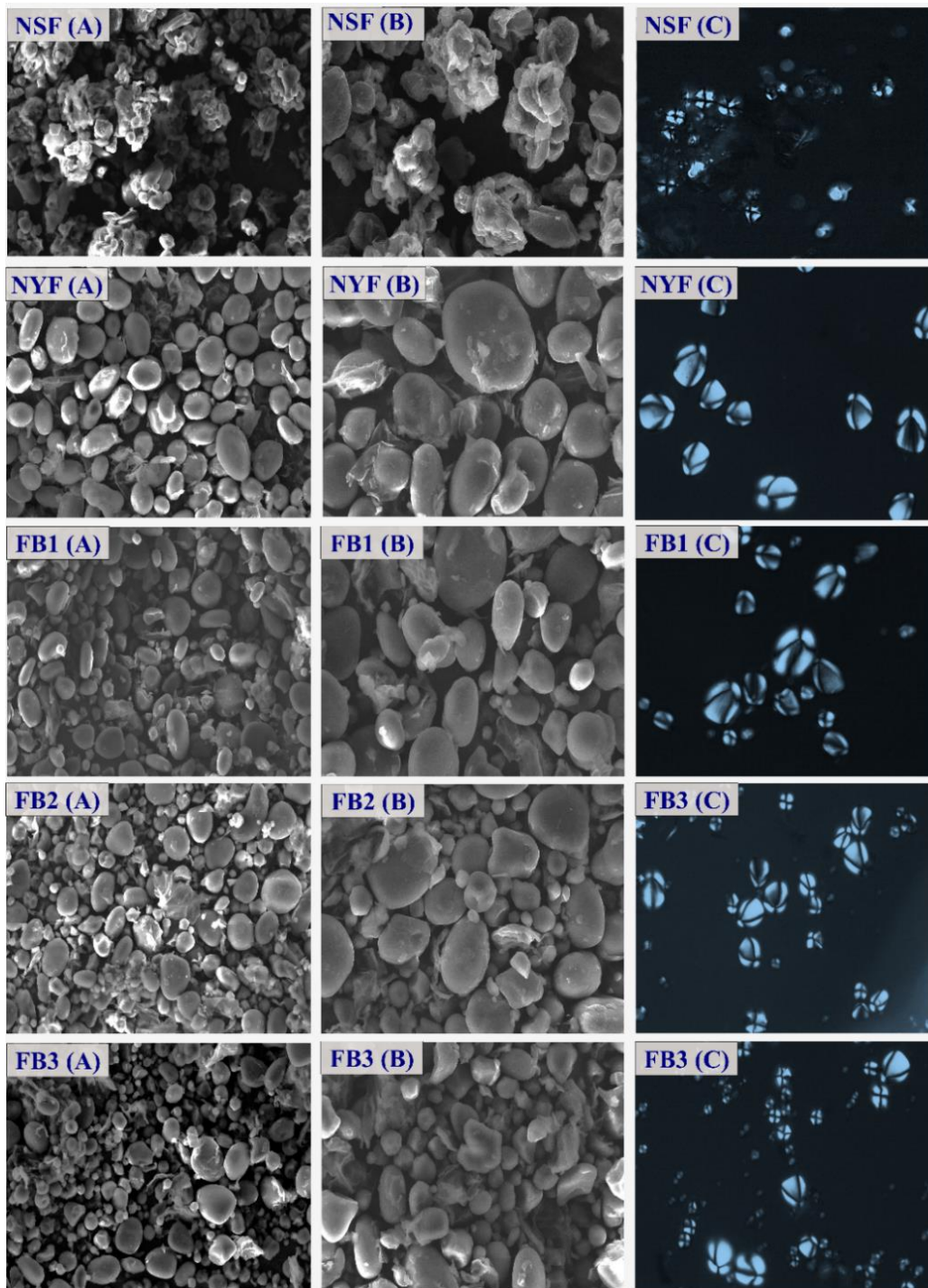
SEM and polarized light micrographs of native flours and their blends are shown in Figure 3. The starch granules of NSF were observed diffusely because of a layer that could correspond to fibers, ashes, and proteins. The granules presented spherical, ovoid, and polygonal morphologies with smooth surfaces and no evidence of cracks, with sizes between 9 and 25  $\mu\text{m}$  according to SEM. Granule birefringence exhibited typical clear cross-polarization with a crossover point near the starch granule core (Chen et al., 2021).



**Figure 2.** Infrared spectra in native yam, sweet potato, and flour blend flours. NYF: native yam flour; NSF: native sweet potato flour; FB1: (75 %) yam and (25 %) sweet potato flour blends; FB2: (50 %) yam and (50 %) sweet potato flour blends; FB3: (25 %) yam and (75 %) sweet potato flour blends.

Source: Prepared by the authors.

On the contrary, the NYF sample revealed larger granules (15 and 40  $\mu\text{m}$ ), with spherical, oval, and some irregular shapes and smooth surfaces, and the presence of non-starchy materials was observed as in NSF (Chen et al., 2017b). Under polarized light, the granules were observed bright against the dark field and showed an eccentric and non-symmetrical Maltese Cross towards the periphery of the granule; this behavior is typical in starches of *Dioscorea* species (Pérez et al., 2016). Similarities were found in orange-fleshed sweet potato starches and yam flours of different varieties (Chen et al., 2017b; Liang et al., 2023). Concerning the flour blends, the proportion in which NSF and NYF were present influenced the granular population. It was found that as the NSF content increased in the flour blends, the presence of smaller granules increased, which occupied the granular spaces of NYF, forming a more compact structure; likewise, the morphological and birefringence characteristics of each species did not show significant changes.



**Figure 3.** Photomicrographs of native flours and flour blends evaluated by scanning electron microscopy at 1000X (A) scale 50  $\mu\text{m}$ ; 2000X; (B) scale 20  $\mu\text{m}$  and polarized light at 100X; (C) scale 10  $\mu\text{m}$ . NYF: native yam flour; NSF: native sweet potato flour; FB1: (75 %) yam and (25 %) sweet potato flour blends; FB2: (50 %) yam and (50 %) sweet potato flour blends; FB3: (25 %) yam and (75 %) sweet potato flour blends.

Source: Prepared by the authors.

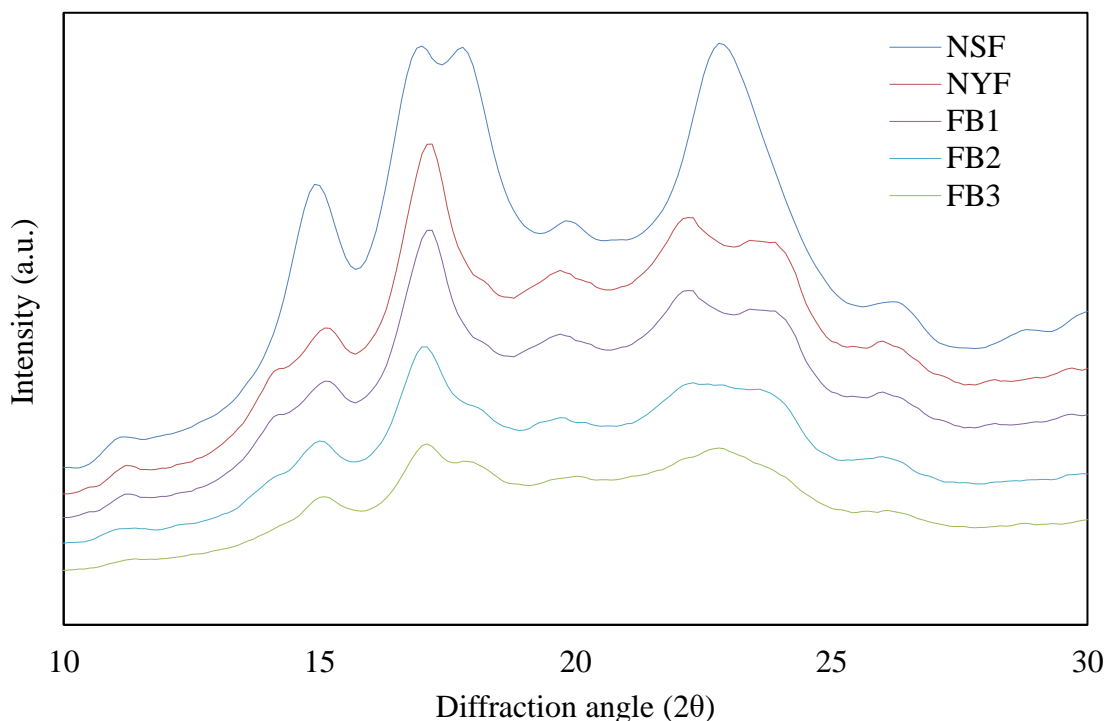
## X-ray Diffraction and Degree of Crystallinity

The X-ray diffraction patterns of native flours and flour blends are observed in Figure 4. The NSF sample had an A-type pattern with characteristic peaks at  $2\theta$  diffraction angles around 15, 20, and 23.1 °, a double peak between 17.1 and 18 °, and weak peaks at 20 and 26.3°; similar results were reported by Wang et al. (2020b). Moreover, NYF exhibited a type-B polymorphism, with three strong diffraction peaks at 17.1, 21.8, and 23.8 °, a double peak between 14.4 and 15.2 °, and a weak peak at 20 °, corresponding to that in purple yam starch (*Dioscorea alata* L.; Lu et al., 2022). In flour blends, the ratio of NYF and NSF could influence the diffraction patterns. In FB1 and FB2, crystalline peaks at  $2\theta$  were observed as follows: inconclusive peaks at 14.4, 20, and 23.8 ° and intense peaks at 15.2, 17.1, and 23.8 ° suggesting a type-B polymorphism consistent with the higher yam flour content of the mixture. In contrast, FB3 showed greater dominance of the type-A pattern with peaks similar to NSF. In that sense, De Oliveira et al. (2018) and Arroyo-Dagobeth et al. (2023) reported equivalent behaviors in binary mixtures of potato and sweet potato starches and cassava and yam, respectively. On the contrary, Babu et al. (2015) found a combination of type-A and type-B crystalline structures in sweet potato starches, so they considered that it corresponded to a type-C pattern.

The DC of the NYF and NSF samples had values of 24.87 and 35.41 %, respectively; similar behaviors were reported in the literature (Yu et al., 2021; Zhang et al., 2023). In addition, there is a correlation between amylose content and degree of crystallinity; therefore, the higher amylose content could have influenced the low crystallinity index in yam flour (Arroyo-Dagobeth et al., 2023; Hornung et al., 2017). Also, relative crystallinity could be affected by the differences in the structure and positioning of the double helices, average chain extension, and the proportion of short amylopectin chains (Li et al., 2019). Conversely, a decrease in the GC was observed in flour blends as the NYF concentration increased (Table 2). FB1 did not show significant differences concerning NYF. In contrast, FB2 and FB3 showed a lower GC than NYF, so it could be considered a non-additive behavior due to a possible interaction effect between the starches of each mixture (Arroyo-Dagobeth et al., 2023). Furthermore, Hornung et al. (2017) reported that the blending process alters the semi-crystalline characteristics of the starches composing the starchy system. Similar behaviors of non-additive effects on GC were found in binary mixtures of potato and sweet potato starches (De Oliveira et al., 2018).

## Physicochemical Properties

Table 4 shows the physicochemical properties of native flours and their blends. SP was higher in NYF than NSF; however, the values obtained are lower than those reported in the literature (Tortoe et al., 2017). This could be related to proteins, fibers, fats, minerals, and other non-starch components in flours, which can adhere on the surface of starch granules and favor the formation of complexes with amylose, forming a rigid matrix that limits the access of water to the interior of the granule (Aprianita et al., 2014; Suriya et al., 2016). SP in flour blends decreased with the addition of NSF, possibly due to reduced starchy components during the wet mixing process. A similar behavior was reported by Praseptianga et al. (2018), who found that the substitution of canna flour (*Canna edulis*) for jack bean flour (*Canavalia ensiformis*) reduced the SP of composite flours.



**Figure 4.** X-ray diffraction patterns of native flours and flour blends. NYF: native yam flour; NSF: native sweet potato flour; FB1: (75 %) yam and (25 %) sweet potato flour blends; FB2: (50 %) yam and (50 %) sweet potato flour blends; FB3: (25 %) yam and (75 %) sweet potato flour blends.

Source: Prepared by the authors.

The WAC of flours and their blends are listed in Table 4. The NYF sample showed a higher AAC compared to NSF; these differences could be associated with (1) agronomic factors, tuber species, and variety; (2) flour production process; (3) starch granule shape and size; (4) diffraction patterns (Arroyo-Dagobeth et al., 2023; Belkacemi, 2022). The results obtained are lower than those found in the literature (Effah-Manu et al., 2022). Mixtures showed a significant increase in AAC concerning native flours, which could be related to a possible increase in leaching and solubility of amylose during mixing; in addition, it could have occurred that NSF provided non-starch constituents that favor the interaction with water (Singthong, 2018). These results are similar to those Chandra et al. (2015) reported on wheat, potato, rice, and green gram composite flours.

The native flours and blends showed a higher affinity to water than oil. However, the NSF sample had the highest OAC value (Table 4). These results are higher than those of Dereje et al. (2020), who reported an OAC between 9 and 12 % for red and white sweet potato flours and between 94.90 and 106.7 % for orange-fleshed sweet potatoes. On the other hand, the values achieved for yam flour differ from those reported by some authors who reached an OAC between 97 and 200 % in creole yam flour (Egbedike et al., 2016). Regarding flour blends, a

significant increase in OAC occurred when the proportion of NSF increased, which could be considered an additive behavior. Similar results were reported in composite flours by increasing the substitution of wheat and yam flour for orange-fleshed sweet potato flour and rice bran flour, respectively (Edun et al., 2019; Egbedike et al., 2016).

**Table 4.** Physicochemical properties of native yam and sweet potato flours and their blends

Treatment	SP (g/g)	WAC (%)	OAC (%)	CWS (%)
NYF	2.87 ± 0.01 <sup>a</sup>	154.17 ± 0.35 <sup>a</sup>	87.94 ± 0.78 <sup>a</sup>	14.27 ± 0.15 <sup>a</sup>
NSF	1.68 ± 0.01 <sup>b</sup>	115.24 ± 0.54 <sup>b</sup>	124.61 ± 0.17 <sup>b</sup>	44.59 ± 0.14 <sup>b</sup>
FB1	2.47 ± 0.01 <sup>c</sup>	145.50 ± 0.13 <sup>c</sup>	75.38 ± 0.68 <sup>c</sup>	9.70 ± 0.22 <sup>c</sup>
FB2	2.47 ± 0.01 <sup>c</sup>	165.45 ± 0.25 <sup>d</sup>	89.73 ± 0.30 <sup>d</sup>	12.55 ± 0.41 <sup>d</sup>
FB3	2.44 ± 0.01 <sup>d</sup>	168.66 ± 0.22 <sup>e</sup>	114.35 ± 0.22 <sup>e</sup>	20.46 ± 0.19 <sup>e</sup>

NYF: native yam flour; NSF: native sweet potato flour; FB1: (7 %) yam and (2 %) sweet potato flour blends; FB2: (5 %) yam and (5 %) sweet potato flour blends; FB3: (2 %) yam and (7 %) sweet potato flour blends; SP: swelling power; WAC: water absorption capacity; OAC: oil absorption capacity; CWS: cold water solubility.

Source: Prepared by the authors.

As for CWS, the results indicated that NSF has approximately three times higher CWS than NYF. Variations in CWS could be associated with the molecular structure of the starchy material, as the ordered semi-crystalline arrangement and organization of the different levels that make up the granules of native starch can impede the penetration of cold water and hydrogen bonds (Akhila et al., 2022). In addition, the proximate composition of flours is another factor that may affect solubility, probably due to the large number of biomolecules that could form inclusion complexes with amylose, subsequently affecting the hydrophilic properties of the granule (Arazu et al., 2021; Kehinde et al., 2020). As the proportion of NSF increased in flour blends, CWS increased; this behavior could indicate an additive effect associated with its high CWS value. Similar behaviors were found in corn and soybean flours enriched with jackfruit (*Artocarpus heterophyllus* L.) powder and composite flours from rice and wheat (Jan et al., 2022; Nansereko et al., 2023).

### Pasting Properties

Viscosity profiles of native flours and their blends are presented in Figure 5. The NSF sample exhibited a significantly lower peak viscosity compared to NYF, which could be related to its lower starch content, amylose/amylopectin ratio, granule stiffness, and size, along with the presence of proteins, fibers, and lipids that may have restricted the swelling of starch granules during the heating phase (Effah-Manu et al., 2022; Zou et al., 2021a). Throughout the heating phase at high temperatures, a gradual decrease in viscosity under the action of shear force was observed; it could be considered that the NSF sample was more stable during heating because it exhibited the lowest breakthrough viscosity, in turn, the high value obtained for NYF could

indicate that it possesses low thermal and mechanical stability when exposed to shear forces and high temperatures (Effah-Manu et al., 2022; Li et al., 2022).

Subsequently, viscosity increased due to starch retrogradation with the decrease in temperature. In this aspect, the NYF sample exhibited a higher setback viscosity, with a greater tendency to retrogradation. This phenomenon is due to the reassociation of amylose and amylopectin chains, which combine with water molecules through hydrogen bonds, forming a three-dimensional network structure. It could also be associated with the diffraction pattern, as starches with type-B polymorphism have been found to possess a higher degree of retrogradation than those with type-A crystalline polymorphism (Arroyo-Dagobeth et al., 2023; Hornung et al., 2017). Overall, the results obtained for NYF and NSF differ from those reported in the literature (Effah-Manu et al., 2022; Singthong, 2018).

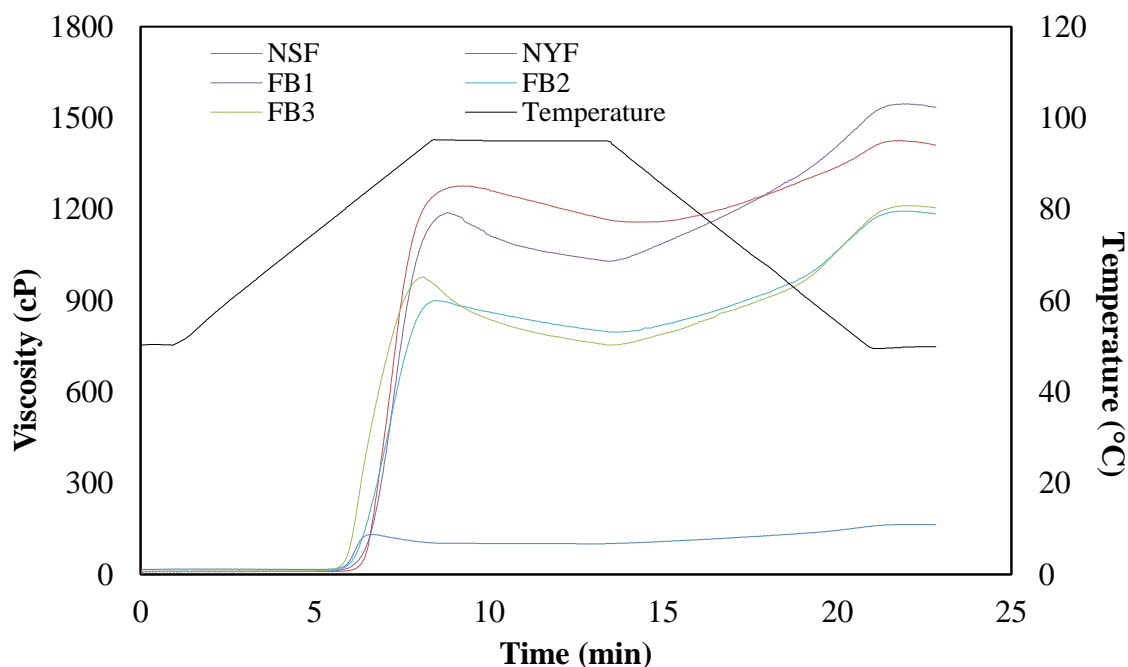
Flour blends showed a decrease in maximum viscosity as the proportion of NSF increased; there was an increase in the breakdown and setback viscosity, indicating a lower thermal stability and a greater tendency to retrogradation. In this context, Younge et al. (2022) reported that variations in peak viscosity could be related to the rate at which starch granules in flour blends absorb water and swell during heat treatment. Likewise, a significant decrease in the breakdown and setback viscosities of the mixtures was determined. This behavior may be associated with the nature of the materials, temperature, the proportion of the mixture, and the shear rate applied (Edun et al., 2019). Similar results have been reported by Giri et al. (2022) and Younge et al. (2022) regarding a decrease in viscosity profiles as the proportion of cream and orange-fleshed sweet potato flour in composite flours increased.

### **Gelatinization Properties**

Table 5 shows the gelatinization properties of yam and sweet potato flours and their blends. The NSF and NYF samples did not show significant differences in the onset ( $T_o$ ), peak ( $T_p$ ), and final ( $T_c$ ) gelatinization temperatures. High  $T_o$ ,  $T_p$ , and  $T_c$  values could be associated with the presence of non-starch components such as fibers, proteins, and lipids in the flours, which can compete for water with each other, leading to disruption of the gelatinization process (Hasmadi et al., 2020). Furthermore, interactions of these components with starch molecules could have strengthened molecular networks, requiring higher temperatures to alter starch crystallites (Mustapha et al., 2019). Likewise, the cell wall in the plant cells that make up the yam and sweet potato flours may act as a barrier capable of preventing water from entering the starch granule, which could decrease the amount of energy released during gelatinization (Wang et al., 2020a). The literature reported similarities through scanning calorimetry assays on yam and sweet potato flours (Li et al., 2019; Zou et al., 2021a).

This behavior could be related to a higher starch content in flour blends and to the decrease of non-starch components, which favors the availability and swelling of the granules during the gelatinization process (Lawal et al., 2021; Roy & Kumar, 2023). An increase in gelatinization temperature range ( $T_c-T_o$ ) was observed as well, which could indicate a higher degree of heterogeneity in the starch crystals within the granules (Wang et al., 2020a). Similarities have been found in ternary mixtures of germinated sorghum, cowpea, and tapioca flours (Marchini et al., 2022). Some authors have reported that thermal properties are related to the size of starch

particles in starchy material, morphology and structural composition (amylose and amylopectin), molecular weight, the chemical composition of starch (phosphate content), damage generated by mechanical processes (cuts and abrasions), and biochemical damage of starch granules caused by the degradation of microorganisms and enzymes inherent to the material and crystalline structure (Zou et al., 2021a).



**Figure 5.** Viscosity profiles of native yam and sweet potato flours and their blends. NYF: native yam flour; NSF: native sweet potato flour; FB1: (75 %) yam and (25 %) sweet potato flour blends; FB2: (50 %) yam and (50 %) sweet potato flour blends; FB3: (25 %) yam and (75 %) sweet potato flour blends.

Source: Prepared by the authors.

**Table 5.** Gelatinization properties of native yam and sweet potato flours and flour blends

Treatment	To (°C)	Tp (°C)	Tc (°C)	Tc-To(°C)
NSF	78.99 ± 1.01 <sup>ab</sup>	84.07 ± 1.01 <sup>a</sup>	88.15 ± 1.02 <sup>a</sup>	9.16 ± 0.01 <sup>a</sup>
NYF	77.97 ± 1.02 <sup>a</sup>	85.09 ± 1.02 <sup>a</sup>	89.17 ± 1.01 <sup>a</sup>	11.20 ± 0.01 <sup>b</sup>
FB1	72.88 ± 1.02 <sup>b</sup>	83.06 ± 1.01 <sup>a</sup>	87.13 ± 1.01 <sup>a</sup>	12.21 ± 0.01 <sup>c</sup>
FB2	74.92 ± 1.02 <sup>bc</sup>	83.06 ± 1.01 <sup>a</sup>	87.13 ± 1.01 <sup>a</sup>	13.23 ± 0.01 <sup>d</sup>
FB3	75.94 ± 1.02 <sup>bc</sup>	84.07 ± 1.01 <sup>a</sup>	89.17 ± 1.01 <sup>a</sup>	14.25 ± 0.01 <sup>e</sup>

NYF: native yam flour; NSF: native sweet potato flour; FB1: (75 %) yam and (25 %) sweet potato flour blends; FB2: (50 %) yam and (50 %) sweet potato flour blends; FB3: (25 %) yam and (75 %) sweet potato flour blends; To: onset temperature; Tp: peak temperature; Tc: final temperature, Tc-To: gelatinization temperature range.

Source: Prepared by the authors.



### ***In vitro* Digestibility Properties**

Fractions of RDS, SDS, and RS of native flours and their blends are shown in Table 6. NSF and NYF samples showed significant differences ( $p < 0.05$ ). NSF had a higher proportion of RDS than NYF but a lower proportion of SDS and RS. In this regard, starchy materials with low amylose content have higher digestibility than those with high amylose content (Mahajan et al., 2022). In addition, starches with type-B structures are less digestible than type-A and C patterns. Since the granule is partially accessible for enzymatic catalysis by amylase due to its crystalline structure, type-B polymorphisms have a higher RS content (Villarroel et al., 2018). Also, granule morphology and starch complexation with non-starch components could significantly impact this property (Onyango et al., 2020). The results obtained for NSF and NYF differ from the values reported for sweet potato starches and yam flours of different varieties (Chen et al., 2017a; Huang et al., 2016; Liu et al., 2019; Trung et al., 2017).

**Table 6.** *In vitro* digestibility properties of native flours and their blends

<b>Treatment</b>	<b>RDS</b>	<b>SDS</b>	<b>RS</b>
<b>NYF</b>	65.92 ± 0.24 <sup>a</sup>	24.98 ± 0.62 <sup>a</sup>	9.10 ± 0.40 <sup>a</sup>
<b>NSF</b>	73.62 ± 0.96 <sup>b</sup>	20.40 ± 0.83 <sup>b</sup>	5.98 ± 0.13 <sup>b</sup>
<b>FB1</b>	79.55 ± 0.25 <sup>c</sup>	10.74 ± 0.13 <sup>c</sup>	9.70 ± 0.12 <sup>a</sup>
<b>FB2</b>	84.07 ± 0.67 <sup>c</sup>	6.00 ± 0.20 <sup>d</sup>	9.92 ± 0.50 <sup>a</sup>
<b>FB3</b>	87.53 ± 0.70 <sup>d</sup>	5.44 ± 0.35 <sup>d</sup>	7.02 ± 0.69 <sup>b</sup>

NYF: native yam flour; NSF: native sweet potato flour; FB1: (75 %) yam and (25 %) sweet potato flour blends; FB2: (50 %) yam and (50 %) sweet potato flour blends; FB3: (25 %) yam and (75 %) sweet potato flour blends; SDS: slow digestion starch; RDS: rapid digestion starch; RS: resistant starch.

Source: Prepared by the authors.

Regarding flour blends, the proportion of RDS was significantly higher than the individual treatments and increased as the percentage of NSF increased; an inverse behavior was observed for the SDS fraction. Results suggest a non-additive effect in blends, probably due to the interaction of starchy and non-starch components in the flours. According to Ma et al. (2020), blends favor starch digestibility because a decrease in the spaces between granules occurs, as observed in SEM micrographs, which in turn increases the packing density of granular populations that could induce more available areas for amylase enzymatic hydrolysis. Similar results were reported by Arroyo-Dagobeth et al. (2023) on the non-additive effects on the *in vitro* digestibility properties of native cassava and yam starch mixtures.

The different proportions of flour blends presented with low SDS and RS fractions concerning native yam and sweet potato flours. Also, the mixing effect significantly decreased the cold water solubility capacity. Therefore, in the search for new functionalities of flour blends, it is necessary to evaluate hydrothermal or biocatalytic processes that increase their versatility in the food industry. It should be noted that hydrothermal and biocatalytic processes are safe, innocuous, and environmentally friendly. Additionally, they are characterized by improving the stability of starch pastes during heating and increasing the starch fractions of low digestibility. They can also

be combined to prepare flours with high water retention capacity and soluble in cold water (Arroyo-Dagobeth et al., 2023; Figueroa-Flórez et al., 2023).

## Conclusions

Sweet potato flours exhibited spherical, ovoid, bell, and polygonal-shaped starch granules, while yam starch granules showed spherical, oval, and irregular shapes. Granular sizes were also significantly different, with yam granules larger than sweet potato granules. In contrast, both starchy materials had smooth surfaces, free of porosities. Significant differences existed between the starch and amylose content of sweet potato and yam flours, with values of 54.35 and 77.32 % for starch content and 11.95 and 19.48 % for amylose content, respectively.

Development of blending processes from yam and sweet potato flours generated changes in proximate composition, starch, and amylose content concerning native flours that defined both additive and non-additive behavior of molecular order, degree of crystallinity, physicochemical and gelatinization properties, thermal stability, higher tendency to retrogradation during cooling, and increase in ADR and ARS fractions, without altering the granular morphological characteristics of native flours. Therefore, the development of blends can be considered a simple method capable of influencing the properties of the materials based on interaction phenomena between the starchy and non-starch components of the individual flours.

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## Authors' Contributions

All authors reviewed the final version of the manuscript. The contributions made by the authors were as follows: Karen Margarita Seña-Rambauth: Formal analysis, Investigation, Methodology, Writing – Original draft, review & editing; Jorge Emilio Hernandez-Ruydiaz: Resources, Supervision and validation; Jorge Antonio Figueroa-Flórez: Conceptualization, Supervision, validation and Writing – review & editing; Jairo Guadalupe Salcedo-Mendoza: Funding acquisition, Supervision and validation; Fabian Alberto Ortega-Quintana: Supervision and validation.

## Ethical Implications

In the development of this article, no ethical implications were identified or presented that required further consideration or review.

## Conflict of Interest

In the development of this article, no ethical implications were identified or presented that required further consideration or review.

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