Structural, physicochemical, and pasting properties of native cassava (Manihot esculenta) and yam (Dioscorea alata) starch blends

Propiedades estructurales, fisicoquímicas y de empastamiento de mezclas de almidones nativos de yuca (*Manihot esculenta*) y ñame (*Dioscorea alata*)

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RESUMEN

ABSTRACT

Starch blends are a technological alternative aimed at the development of starchy matrices that exhibit improvements in some physicochemical properties from interactions between their individual components. Native cassava and yam starches were mixed in different proportions and the effect of the blend on the structural, physicochemical, and pasting properties was evaluated. The viscosity behavior as a function of temperature revealed a significant non-additive effect on the pasting parameters of all the blends with respect to the individual native starches. Similarly, non-additive variations were evident in the crystallinity index of some mix ratios (NSB-2: 40.11%). Likewise, the difference in the amylose content of each native starch (20.88-25.66%) possibly exerted an effect on the resulting semicrystalline characteristics of the blends and the gelatinization behavior. Hence, the botanical origin and the proportion of starch blends play an important role in the behavior of the resulting physicochemical properties and in vitro digestibility. Polymeric blends were obtained with a low tendency to retrogradation and lower crystallinity index values compared to their native counterparts and a regulated water absorption capacity, all potentially desirable characteristics in the food industry.

Key words: tuber starch, crystallinity index, non-additive effect, digestibility.

Las mezclas de almidones son una alternativa tecnológica dirigida al desarrollo de matrices amiláceas que presenten mejoras en algunas propiedades fisicoquímicas a partir de interacciones entre sus componentes individuales. Por lo tanto, se mezclaron almidones nativos de yuca y ñame en diferentes proporciones y se evaluó el efecto de la mezcla sobre las propiedades estructurales, fisicoquímicas y de empastamiento. El comportamiento de la viscosidad en función de la temperatura reveló un efecto no aditivo significativo sobre los parámetros de pasta de todas las mezclas con respecto a los almidones nativos individuales. De manera similar, las variaciones no aditivas fueron evidentes en el índice de cristalinidad de algunas proporciones de mezcla (NSB-2: 40.11%). Asimismo, la diferencia en el contenido de amilosa de cada almidón nativo (20.88-25.66%) posiblemente ejerció un efecto sobre las características semicristalinas resultantes de las mezclas y el comportamiento de gelatinización. Por lo tanto, el origen botánico y la proporción de mezcla de almidón juegan un papel importante en el comportamiento de las propiedades fisicoquímicas resultantes y la digestibilidad in vitro. Se obtuvieron mezclas poliméricas con una baja tendencia a la retrogradación y valores de índice de cristalinidad más bajos en comparación con sus homólogos nativos y una capacidad de absorción de agua regulada, todas ellas características potencialmente deseables en la industria alimentaria.

Palabras clave: almidón de tubérculos, índice de cristalinidad, efecto no aditivo, digestibilidad.

Introduction

Starches from different botanical sources exhibit unique physicochemical properties because of their specific granular size, amylose content, and chain length distribution of their molecular constituents (Waterschoot *et al.*, 2015). The specific characteristics related to the structure and granular composition govern the macroscopic behavior of each starchy material. In Colombia, starch-rich raw materials such as cassava and yam are cultivated; and their starchy constituents show differences in size and granular shape, chemical composition, and viscosity behavior under an excess of water (Karam *et al.*, 2006). For instance, cassava starch displays an amylose content of approximately 19%; and in yam starches it is around 28% (Karam *et al.*, 2006). Thus, the paste clarity and strength characteristics of native

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cassava starch gels, coupled with the remarkable hot viscosity stability of yam starch pastes, make these polysaccharide materials highly suitable ingredients that are attractive in the formulation of different products related to the food industry (Novelo-Cen & Betancur-Ancona, 2005; Karam *et al.*, 2006).

At the molecular level, starches are reserve macromolecules synthesized in the amyloplasts of different plants as granular particles with variable shapes and sizes, depending on their botanical origin (Ai & Jane, 2015). At the structural level, they have two main polysaccharide constituents: amylose and amylopectin. The first is essentially linear, made up of glucose units linked by α -(1,4) bonds. The second is present in a greater proportion in the starch granule and has abundant branches anchored to the polymer chains of glucose by α -(1,6) bonds. The latter exerts a significant effect on the semi-crystalline properties, swelling power in the granules, insolubility in water and viscosity (Pérez & Bertoft, 2010; Ee et al., 2020). In their native state, starches are used as stabilizing, binding, or thickening materials (Majzoobi & Farahnaky, 2021). However, their industrial application is limited (Maniglia et al., 2021), since they exhibit significant sensitivity to environments with low pH, high temperatures or high shear forces (Dupuis & Liu, 2019).

To develop new functionalities and inhibit some undesirable properties of native starches, modifications of their structural characteristics are made (Dolas et al., 2020) using physical, chemical, or enzymatic methods (Zia-ud-Din et al., 2017). In recent years, the growing consumer demand for clean-label products has generated an increase in research aimed at improving the functionalities of native starches through low-cost methods that use simple and powerful industrial technologies. Zhu et al. (2020) suggest a blend of native starches as an alternative process that makes possible the synthesis of polymeric matrices with desirable properties for the food industry (Waterschoot et al., 2015; Hornung et al., 2017) that guarantees the conservation of the category of the resulting starchy material as "food ingredient" instead of "food additive" (Park & Kim, 2021), thus allowing the development of chemical-free starch-based products. Obanni and Bemiller (1997) observe the formation of a continuous phase (native granules) and a discontinuous phase (swollen granules, fragments of granules or retrogradation products) in blends made up of granules of different botanical origin, showing a greater effect of interaction between starches compared to the responses observed in individual starches. Thus, the selection of starches that exhibit significant differences in

their intrinsic properties could cause unpredictable behavior for the resulting blends compared to the properties of their starchy counterparts.

The starch blends derived from various botanical sources are regarded as a mechanism capable of imparting novel functionalities to the industry through the interactions among the individual properties of each starchy component (Oliveira et al., 2018; Wang et al., 2020). In starch blending, additive effects are noticed when the properties of the blend can be predicted from the individual starches. Nevertheless, non-additive effects are associated with interaction phenomena among starches; consequently, predictions do not align consistently with the actual characteristics of the blend (Park & Kim, 2021). Waterschoot et al. (2015) establish that significant differences in properties such as gelatinization temperature, granular size, and amylose content of each component in the starch blend could lead to specific interactions, depending on the blend proportions evaluated by Waterschoot et al. (2016). Novelo-Cen and Betancur-Ancona (2005) report that the interactions among starch molecules and granular structures in starch blends result in behaviors that resemble the properties developed through chemical modifications. Hence, it is crucial to comprehend the potential types of interactions among starches in blends to enhance their functionalities for industrial applications (Zhu et al., 2020).

Different blend proportions were formulated for native cassava and yam starches in this research. In this case, native cassava and yam starches exhibit significant differences in chemical composition, granular and molecular structure, variations in terms of thermal stability, tendency to retrogradation, as well as limited commercial and industrial exploitation of yam starch (Karam et al., 2006; Salcedo Mendoza et al., 2016). The present study aims to assess the effect of different proportions of cassava and yam starches on the physicochemical, pasting, structural and in vitro digestibility properties of the blends, to regulate the tendency towards retrogradation and achieve improvements in stability of the viscosity of the resulting starchy materials. All were evaluated from the analysis of the interactions present in each property. The foregoing is intended to stimulate the investigation of processes that develop polymeric starch matrices that exhibit desirable properties for the food industry. These processes include starchy sources with limited technological use in Colombia, through a mechanism of functional improvement that avoids the use of chemical agents, ultimately facilitating the formulation of clean-label products.

Materials and methods

Materials

The native cassava starch (*Manihot esculenta* cv. M-TAI) was supplied by the company Almidones de Sucre S.A.S. (Induyuca[®], Sincelejo, Colombia). The tubers of Creole yam (*Dioscorea alata*) were purchased from the local market in the city of Sincelejo, Sucre, Colombia. Commercial enzymes such as bacterial α-amylase (Lyquozyme Supra-2.2X[®]) and fungal amyloglucosidase (Dextrozyme[®]) were purchased from Novozymes (Bagsvaerd, Denmark). Likewise, pancreatic α-amylase with biocatalytic activity \geq 5 U/mg (Sigma-Aldrich, Type VI-B, USA) was used.

Yam starch extraction

Yam starch was extracted following the methodology described by Salcedo Mendoza *et al.* (2016) with some modifications. At first, the yams were washed, dehusked and cut into cubes and processed in an Oster[®] domestic blender for 1 min. The resulting slurry was processed using a pilot air disperser bubbling equipment (pump 0.25 hp, air compressor 100 pounds) that promotes the separation between starch and bagasse due to density differences. This facilitates the natural decanting of starch. Later, filtrations were carried out with the intention of separating residual solid impurities, as well as successive washings to finally dry the starches at 35°C for 24 h. The samples were macerated and sieved (\leq 74 µm), and stored in hermetically sealed bags.

Starch blends

All mix ratios between cassava and Creole yam starches were prepared following the methodology described by Hornung *et al.* (2017) with some modifications. Thus, each proportion of starch blend was developed in a suspension of 30% w/v in distilled water, magnetically stirred (250 rpm) for 15 min to ensure homogeneity (Tab. 1). Afterward, each starchy mixture was centrifuged at 3500 rpm for 7 min, discarding the aqueous supernatant and drying the starches for 24 h at 35°C. Finally, the samples were ground, sieved (200 mesh, which is equivalent to 74 mm of mesh opening) and stored at room temperature in airtight plastic bags for further analysis.

Apparent amylose content

The apparent amylose content for all samples was determined by the conventional iodine staining spectrophotometric method described by Khoomtong and Noomhorm (2015), with minor modifications. One hundred mg of dry basis sample were dissolved in 1 ml of absolute ethanol plus 9 ml of 1M NaOH, heated to 100°C for 30 min. A sample of the diluted solution was used for estimating the apparent amylose content, mixing it with 200 μ l of 1M acetic acid, 400 μ l of lugol solution (2.0% KI and 0.2% I₂), and distilled water. A UV-VIS spectrophotometer Pharo 300 (Spectroquant[®], Darmstadt, Germany) was employed to measure the coloration of the samples at 620 nm. The amylose content was determined using a calibration curve with pure potato amylose as standard (Sigma Aldrich[®], USA).

TABLE 1. Percentage of starch used in the binary blends.

Somaloo	Starch (%)		
Samples	Cassava	Yam	
NCS	100	0	
NYS	0	100	
NSB-1	30	70	
NSB-2	50	50	
NSB-3	60	40	
NSB-4	45	55	
NSB-5	70	30	

NCS: native cassava starch; NYS: native yam starch; NSB-1: blend of cassava 30%-yam 70%; NSB-2: blend of cassava 50%-yam 50%; NSB-3: blend 60%-yam 40%; NSB-4: blend of cassava 45%-yam 55%; NSB-5: blend cassava 70%-yam 30%.

Morphology and birefringence

An aliquot of 10 μ l (0.1% w/v starch in deionized water) was poured onto a slide to examine the birefringent characteristics of the granules using a binocular microscope (DM1000 LED, Leica, Japan). Micrographs were obtained from polarized light fields at 40X magnification using a digital camera (Leica, ICC50W, Japan).

Diffraction patterns and crystallinity index

X-ray diffraction patterns of starch samples were acquired using a diffractometer (X'Pert Pro-MPD, Panalytical, Italy), employing CuK radiation (λ =1.55 pm) and a secondary beam graphite monochromator at 30 kV and 30 mA (Colussi *et al.*, 2020). Spectra were obtained over a 2 θ Bragg angle range of 4-30°, at an inspection speed of 2° min⁻¹ with a measurement interval of 0.02° for each sample. The crystallinity index was obtained from the ratio of the areas corresponding to the crystalline regions and the total area, obtained by numerical integration methods, using the MATLAB software (MathWorks, R2019a, USA).

Pasting properties

Viscosity profiles were determined following the method proposed by Fonseca-Florido *et al.* (2017) with slight modifications, using a rheometer (Anton Paar, MCR 302, Austria), a cell for analysis of starch suspensions (C-ETD160/ST) and a starch cell geometry (Anton Paar, ST24-2D/2V /2V-30, Austria). Thus, 2.0 g (dry basis) of each sample were suspended in 25 ml of distilled water. The suspensions were subjected to controlled heating and cooling cycles with a stirring speed of 250 rpm. Initially, each sample was subjected to a temperature sweep at 50°C for 1.0 min, then heated to 95°C in 7.5 min, held at 95°C for 5.0 min, cooled to 50°C in 7.5 min, and finally kept at 50°C for 2.0 min. The rates of ascent and descent were 7.5°C min⁻¹ for each stage. The following parameters were obtained: pasting temperature, peak viscosity, breakdown viscosity, and setback viscosity. Viscosities were recorded in centipoise (cP).

Water absorption capacity (WAC)

WAC values were estimated following the methodologies developed by Yadav *et al.* (2016) with some modifications. Briefly, a starch sample of 1.0 g on a dry basis was suspended in 10 ml of distilled water at room temperature, gently shaken to homogenize, and centrifuged at 3500 rpm for 15 min. The supernatant liquid was discarded, and the precipitated starch was weighed to estimate the percentage of water absorption capacity (Eq. 1).

WAC (%) =
$$\frac{W - W_0}{W_0} \times 100$$
 (1)

where, $W_0(g)$ is the weight of dry starch, and W(g) is the weight of the sediment after centrifugation.

In vitro digestibility

In vitro starch digestibility was determined as described by Englyst et al. (1992) with modifications. Initially, enzyme solutions were prepared by mixing 9 g of pancreatic a-amylase in 60 ml of citrate buffer (Solution I) and a solution of amyloglucosidase with biocatalytic activity of 140 AGU ml⁻¹ (Solution II). Then, 200 mg (dry basis) of starch was dispersed in 25 ml of citrate buffer (pH 5.2) and subjected to gelatinization for 20 min at 90°C. Afterward, 1 ml of Solution III (composed of 54 ml of Solution I and 6 ml of Solution II) was added to the gelatinized suspension and kept at 37°C with stirring at 250 rpm for 120 min. Aliquots were taken at 20 and 120 min to determine the content of glucose released, using the DNS method (3,5-dinitrosalicylic acid). The fractions of rapidly digestible starch (RDS), slowly digestible (SDS), and resistant starch (RS) in the blends were calculated according to Englyst et al. (1992).

Thermal properties

Samples of starch (3.0 mg, dry basis) were mixed with 11 μ l of distilled water and evaluated by differential scanning calorimetry (DSC) (Reyes-Atrizco *et al.*, 2019) with slight modifications. In this way, the aluminum capsules

were sealed and stored for 24 h prior to analysis to balance the system. Then, the samples were subjected to thermal treatments with heating from 20 to 120°C at a rate of 10°C min⁻¹ followed by cooling to 20°C with a ramp of 25°C min⁻¹ in a nitrogen atmosphere. The thermal parameters onset temperature, peak temperature, final temperature, and enthalpy of gelatinization (Δ H) were determined from the analysis of the thermogram using the TA Universal Analysis software (TA 2000, TA Instruments Inc., USA).

Statistical analysis

All determinations were expressed as the mean of three replicates \pm standard deviation. Means and significance of differences between samples were established using analysis of variance (ANOVA) and Tukey's test (*P*<0.05), using the statistical software Statgraphics Centurion (Statgraphics Inc., version XVI, USA).

Results and discussion

Amylose content

The amylose content of native starches and their blends is presented in Table 2. At first, native yam starch (NYS) had a higher amylose content compared to native cassava starch (NCS); these findings corresponding to those reported by Monroy *et al.* (2018) and Duan *et al.* (2020) for NCS and NYS. This could be related to the nature of the crystalline polymorphism of NYS and NCS, which were B-type and Atype. Hornung *et al.* (2017) report that starches with B-type diffraction pattern generally have a high retrogradation, so the greater tendency towards retrogradation shown by NYS pastes on cooling may be associated with its higher amylose content compared to NCS (Fig. 3). The amylose content exerts an important effect on the semi-crystalline characteristics, the gelatinization temperatures, and the paste behavior of starches (Hornung *et al.*, 2017).

The evaluated starch blends presented a possible additive behavior in the amylose content, so that the values obtained were in a range delimited by the amylose content of the individual native starches, guaranteeing a proportional relationship for the amylose content of starch blends as a function of the individual values of each starchy counterpart (Tab. 2). It is possible to predict the behavior of the amylose content of the starch blends from the values exhibited by NCS and NYS, while each mixture presented an additive variation in the content of amylose, corresponding to each mixing ratio between NCS and NYS. Likewise, a similar behavior was reported by Ma *et al.* (2020) for amylose leaching from binary blends between potato starch and high amylose rice starch, where amylose leaching for each mixing ratio of these starches was between the values exhibited by their individual counterparts, indicating an additive effect. Related to the above, Cruz-Benitez *et al.* (2019) state that the amylose content of each starch affects the physicochemical properties such as water absorption. Consequently, the possible proportional interaction observed in the binary blends of NCS and NYS in the amylose content could contribute to the display of non-additive behaviors in other physicochemical properties, and the absorption capacity of water in the blends.

TABLE 2. Water absorption capacity, amylose content, and crystallinity index.

Samples	WAC (%)	AC (%)	CI (%)
NCS	$65.72 \pm 1.11^{\text{a}}$	$20.88\pm0.07^{\text{a}}$	48.05 ± 0.70^a
NYS	$93.09\pm1.14^{\text{b}}$	$25.66\pm0.08^{\text{b}}$	$45.07\pm0.42^{\text{b}}$
NSB-1	$94.19\pm2.89^{\circ}$	$24.30\pm0.09^{\circ}$	$43.77\pm0.53^{\text{b}}$
NSB-2	78.84 ± 0.55^{d}	$23.34\pm0.04^{\text{d}}$	$40.11\pm0.16^\circ$
NSB-3	$81.57 \pm 1.12^{\circ}$	$22.88\pm0.06^{\text{e}}$	$44.87\pm0.46^{\text{b}}$
NSB-4	78.73 ± 0.65^{d}	$23.73\pm0.11^{\rm f}$	$44.67\pm0.66^{\text{b}}$
NSB-5	77.56 ± 0.92^{d}	$22.03\pm0.04^{\text{g}}$	$45.03\pm0.43^{\text{b}}$

WAC: water absorption capacity; AC: amylose content; CI: crystallinity index; NCS: native cassava starch; NYS: native yam starch; NSB-1: blend of cassava 30%-yam 70%; NSB-2: blend of cassava 50%-yam 50%; NSB-3: blend 60%-yam 40%; NSB-4: blend of cassava 45%-yam 55%; NSB-5: blend cassava 70%-yam 30%. Means followed by same letters within a column do not differ statistically according to Tukey's test (P<0.05).

Morphology and birefringence

Polarized light microphotographs of native starches and their blends are illustrated in Figure 1. At first, Figure 1-NYS presents the morphology of the NYS granules, which exhibit an ellipsoidal structure with a "Maltese cross" orientation directed towards the periphery of the granule (He & Wei, 2017), indicating that the position of the "hilum" is decentralized, a typical characteristic for starch granules from rhizomes and yams (Chakraborty *et al.*, 2020).

In contrast, the NCS granules presented a spherical shape, with the "Maltese cross" located in the granular center (Fig. 1, NCS), suggesting the presence of concentrically ordered semi-crystalline structures within the granules (Lin *et al.*, 2020). On the other hand, the micrographs of all blends between NCS and NYS exhibited a granular polydispersity dependent on each mixing ratio evaluated, where the conservation of the birefringent characteristics of each individual starch was appreciable, as well as the typical morphology of the granules in its native state after the mixing process (Fig. 1, NSB-1).

Hornung *et al.* (2017) find that the morphological structure of yam starch granules from different varieties remains unchanged after the mixing process. Wu *et al.* (2016)



FIGURE 1. Microphotographs under polarized light of native starches and their blends. NCS: native cassava starch; NYS: native yam starch; NCS-1: blend of cassava 30%-yam 70%; NCS-2: blend of cassava 50%-yam 50%; NCS-4: blend of cassava 45%-yam 55%; NCS-5: blend cassava 70%-yam 30%. Magnification 40X.

Arroyo-Dagobeth, Figueroa-Flórez, Cadena-Chamorro, Rodríguez-Sandoval, Salcedo-Mendoza, and Cervera-Ricardo: Structural, physicochemical, and pasting properties of native cassava (*Manihot esculenta*) and yam (*Dioscorea alata*) starch blends

highlight that the size and shape of the granules have a determining impact on the non-additive behavior resulting in the pasting properties of the blends between native sweet potato and mung bean starches, showing that significant differences in the morphological characteristics of the starches could trigger unpredictable behaviors in some physicochemical properties of the resulting blends. Hence, the differences in size and granular shape manifested by NCS and NYS could be associated with the non-additive behaviors observed in the pasting profiles (Fig. 3), crystal-linity index, and *in vitro* digestibility of the starch blends evaluated in this study.

X-ray diffraction (XRD) and crystallinity index (CI)

The semicrystalline order of native starches and their blends can be studied from the diffractograms shown in Figure 2. Firstly, NCS showed crystalline peaks characteristic of an A-type starch, exhibiting high magnitude intensities at the 20 Bragg angles: 15°, 17°, 18°, and 23°, similar to Sangian *et al.* (2018). In contrast, NYS showed crystallinity peaks at angles 5.7°, 15.1°, 17.1°, and 24.1°, associated with a B-type diffraction pattern, similar to Oliveira *et al.* (2021).

The behavior of the X-ray diffraction patterns of the starch blends suggested the occurrence of variations in the semicrystalline characteristics compared to their native starchy counterparts (Tab. 2; Fig. 2). All blends between NCS and NYS presented crystalline peaks at Bragg angles 15°, 17°, 18°, and 23°, characteristic of an A-type diffraction pattern similar to that exhibited by NCS; the NSB-1, NSB-3, and NSB-4 blends presented unresolved crystalline peaks



FIGURE 2. X-ray diffraction patterns of native starches and their blends. NCS: native cassava starch; NYS: native yam starch; NSB-1: blend of cassava 30%-yam 70%; NSB-2: blend of cassava 50%-yam 50%; NSB-3: blend 60%-yam 40%; NSB-4: blend of cassava 45%-yam 55%; NSB-5: blend cassava 70%-yam 30%.

at angles 5°, 10°, 12°, and 20°, so that, in these cases, the existence of a C-type pattern can be considered. Oliveira *et al.* (2018) similarly observe an A-type pattern for a binary blend between native potato and sweet potato starches, where the sweet potato A-type diffraction pattern predominates rather than the B-type exhibited by individual potato starch.

However, the differences observed in the intensities of the crystalline peaks present in the diffraction patterns of each blend caused variations in CI compared to the native starches (Tab. 2). At first, NCS and NYS exhibited a crystallinity index of 48.24% and 44.56%, estimates corresponding to Tester et al. (2004) and Figueroa-Flórez et al. (2019). In this context, the behavior of the crystallinity index presents a correlation with the amylose content of NCS and NYS, where NYS showed a lower CI due to its higher content of amylose compared to NCS. However, the evaluated starch blends showed a non-additive behavior in the CI compared to their native counterparts. Thus, except that NSB-2, blends did not present differences (P>0.05) in CI compared to NYS despite being evaluated in different proportions of their starchy constituents, suggested the occurrence of a possible effect of interaction between the starches of each blend. However, NSB-2 showed an unpredictable behavior in CI due to a reduction (P<0.05) compared to the rest of the samples evaluated. Similar to the above, Gomes et al. (2018) demonstrate the occurrence of a non-additive behavior in the crystallinity index of binary blends between native pea and rice starches, Oliveira et al. (2018) in sweet potato-potato blends, and Hornung et al. (2017) for a binary blend between starches from yams Dioscorea piperifolia and Dioscorea trifida. These last authors establish that the developed mixture process alters the semi-crystalline characteristics of the starches that make up the starchy system. Likewise, the previous behaviors in the crystallinity index could be correlated with the non-additive effects observed in the enthalpy of gelatinization of the samples (Tab. 4), since the thermal requirements to trigger the gelatinization of the starch blends would be associated with the semicrystalline behavior exhibited by each blend.

Water absorption capacity (WAC)

The water absorption capacity of native starches and their blends can be seen in Table 2. At first, NCS and NYS showed differences in WAC with values of 65.72 and 93.09%. Waterschoot *et al.* (2015) report that starches with B-type crystallinity pattern have a greater space between the double helices of the amylopectin chains compared to those starches with A-type diffraction patterns. The space allows a greater capacity for water absorption. The WAC was within the ranges reported by Ikegwu *et al.* (2009) and Ayetigbo et al. (2018) for native cassava starch and by Donaldben et al. (2020) for native yam starch. The blends of starches exhibited a non-additive interaction behavior between the starches associated with the in WAC, because the starchy blends exhibit a non-proportional trend in said parameter compared to the individual values of NCS and NYS for each mix ratio. In other words, the WAC of the starch blends cannot be predicted from the mix ratio and the individual values manifested by NCS and NYS. These facts revealed complex phenomena of interaction between the granular populations of each evaluated proportion, possibly associated with the characteristics of the granular surface and the amylose content of each starch present in the blend (Hagenimana & Ding, 2005). The starch blends are a mechanism that allows the development of polymeric matrices with novel and different physicochemical characteristics from the individual starches involved.

Pasting properties

The pasting behavior of native starches and their blends is presented in Figure 3. At first, NCS presented the highest peak viscosity value (2110 cP), indicating a marked tendency for the suspension to reach higher viscosity in the heating section compared to the rest of the starchy samples evaluated, while NYS reached a peak viscosity value (1658 cP) lower than NCS (P<0.05). In contrast, high-temperature viscosity was more stable in NYS compared to NCS, exhibiting a lower breakdown viscosity value (P<0.05), corresponding to 20 cP and 1348 cP. However, the tendency for retrogradation, related to the setback viscosity, was more evident in NYS (2043 cP) than in NCS (795 cP). Thereby, Hornung et al. (2017) report that starches with a B-type diffraction pattern retrograde much more than those with an A-type, as in the case of NYS. Similar pasting profiles have been reported for NCS (Novelo-Cen & Betancur-Ancona, 2005; Lin et al., 2013) and NYS (Huang et al., 2006; Salcedo Mendoza et al., 2016). The behavior of the starch blends viscosities exhibited a non-additive effect on all the pasting parameters compared to their individual counterparts (Fig. 3). All the blend ratios evaluated presented a double peak viscosity, possibly associated with the disparity in granular size and the differentiated water absorption of the two amylaceous species in the blend. In this context, Karam et al. (2006) refer to blends of starches in which the swelling of yam starch granules could be restricted due to the presence of other starchy constituents. Waterschoot et al. (2015) review that small granules in binary starch blends would fill the voids between the large granules, producing a packed system with reduced swelling power that would be positively correlated with the behavior on peak viscosity parameter in starch blends (Wu et al., 2016). Similarly, NCS and NYS present significant differences in granular size, where NYS presents larger granules than NCS, so that it can also be inferred that there was a possible interaction phenomenon between NCS and NYS swelling granules in the heating section for all starch blends, leading to a generalized reduction in the peak viscosity parameter of the evaluated ratios. This was presumably associated with the possible formation of a packed system between small and large granules. However, Obanni and Bemiller (1997) speculate that the double viscosity peak observed in the viscosity profiles of starch blends was a consequence of the significant differences between the breakdown viscosities of the individual starch constituents, a hypothesis that could also be supported by the results obtained in this work. Likewise, similar double peak viscosity behaviors have been reported for binary blends of potato-maize (Ai & Jane, 2015), cassava-sweet potato (Li et al., 2019), and potato-waxy maize starches (Waterschoot et al., 2014). Moreover, the non-additive behavior of the blends between NCS and NYS was more evident in the setback and breakdown viscosity parameters. For example, NSB-2 presented values for breakdown and setback viscosities of 429.9 cP and 655.9 cP, respectively, instead of 684 cP and 1419 cP, where the latter will be the expected values for a 50:50 binary blend for the case that there is an additive behavior of their individual counterparts. The foregoing suggests that it is not possible to establish predictions in the pasting parameters for the blends of NCS and NYS because there are interaction phenomena in the behavior of the viscosities of the starches when they are blended. Thus, non-additive reductions were evident in the pasting parameters of the evaluated starch blends, depending on the proportion of the individual counterparts. Thus, similar results were reported by Zhu et al. (2020), where a binary blend of potato:quinoa (0.67:0.33) presented no additive behavior in the viscosities of the suspension at 95°C and 50°C, showing higher values than those exhibited by the individual potato and quinoa starches.

Interaction phenomenon was observed in the behavior of setback and breakdown viscosities of the starch blends. The blends evaluated exhibited similarity to NCS in the viscosity profile on cooling so that the values of setback viscosity of the blends showed a marked proximity to NCS. This suggested the occurrence of synergistic effects in said parameter (P<0.05), and in practical terms they refer to the fact that the blends of starches showed a response with a low tendency to retrogradation compared to NYS. Likewise, the breakdown viscosities of the starch blends showed greater proximity in said parameter to NYS. Thus,

the starch blends presented greater stability of the hot viscosity compared to NCS (*P*<0.05).



FIGURE 3. Viscosity profiles of native starches and their blends. NCS: native cassava starch; NYS: native yam starch; NSB-1: blend of cassava 30%-yam 70%; NSB-2: blend of cassava 50%-yam 50%; NSB-3: blend 60%-yam 40%; NSB-4: blend of cassava 45%-yam 55%; NSB-5: blend cassava 70%-yam 30%.

The blends exhibited greater viscosity stability against thermal stress. This was possibly due to the presence of NYS, as well as a reduced tendency to retrograde compared to NYS, perhaps associated with the proportion of NCS in each case. There was non-additive behavior in the pasting parameters of the starch blends that is not likely to be predicted from the viscosities of the individual counterparts. Likewise, Zhu *et al.* (2020) considered that the non-additive behavior present in the pasting parameters of the potato, sweet potato and quinoa starch blends could be attributed to the differences in the chemical, structural and granular composition of the starchy components, which applies to the blends of NCS and NYS, where the individual starches exhibited significant differences in morphology, and amylose content among themselves. In

TABLE 3. Gelatinization parameters of native starches and their blends.

contrast, Zhang *et al.* (2011) report that no variations were observed in breakdown and setback viscosities of blends between potato and corn starches. This could be the result of interactions between granular components leached during the gelatinization of the blend.

Thermal properties

The gelatinization parameters of NCS, NYS and their blends are shown in Table 3. At first, the transition temperatures in the NCS gelatinization were lower than those observed in NYS. This could be related to the crystalline polymorphism of NYS that was B-type, different from NCS, which was A-type. Consequently, the presence of abundant amylopectin chains with a high degree of polymerization in B-type starches could cause a lower thermal energy requirement to trigger gelatinization in NYS compared to NCS (Karam et al., 2006). The NYS gelatinization enthalpy was lower than that observed in NCS (Tab. 4). Similar results were reported for the transition temperatures of native cassava and yam starches (Chen et al., 2011; Duan et al., 2020). Likewise, the gelatinization enthalpies for NCS and NYS were 9.39 and 5.77 J g⁻¹. These results are consistent with Jyothi et al. (2005) and Duan et al. (2020).

All the starch blends exhibit the presence of two peak temperature values (Tab. 4), probably due to the independent gelatinization of the starches in each blend. Waterschoot *et al.* (2015) report that the DSC tests for binary blends of starches with intermediate moisture contents (35-65%), show two endotherms in the DSC profiles. In the present study, the first peak is related to the onset gelatinization temperature of NCS and the second corresponds to NYS.

The onset temperatures T_0 for each blend showed values like NCS, whereas the final temperatures T_f were close to NYS. Therefore, the evident independence in the gelatinization of each starch in the mixture suggests additive

Samples	T ₀ (°C)	Т _{р1} (°С)	Τ _{p2} (°C)	Т _f (°С)	∆H (J g⁻¹)
NCS	$67.88\pm0.03^{\text{a}}$	$74.18\pm0.05^{\text{a}}$	-	82.84 ± 0.49^{a}	$9.39\pm0.06^{\text{a}}$
NYS	79.45 ± 0.01^{b}	83.37 ± 0.02^{b}	-	$89.48\pm0.30^{\text{b}}$	5.77 ± 0.05^{b}
NSB-1	$67.60 \pm 0.06^{\text{b}}$	$72.18 \pm 0.01^{\circ}$	82.56 ± 0.01^{a}	$86.65\pm0.56^{\circ}$	$5.73\pm0.02^{\text{b}}$
NSB-2	67.76 ± 0.01^{b}	$72.13 \pm 0.01^{\circ}$	$83.38 \pm 0.01^{\text{b}}$	$88.20\pm0.48^{\text{b}}$	$6.82\pm0.06^{\circ}$
NSB-3	$67.45 \pm 0.01^{\circ}$	$72.32\pm0.01^{\rm d}$	$83.25\pm0.43^{\text{b}}$	$88.29\pm0.41^{\text{b}}$	$7.59\pm0.38^{\rm d}$
NSB-4	$67.40 \pm 0.32^{\circ}$	$72.51 \pm 0.01^{\circ}$	$83.37 \pm 0.13^{\text{b}}$	$88.07 \pm 0.04^{\text{bc}}$	$6.81 \pm 0.02^{\circ}$
NSB-5	$67.45\pm0.03^{\circ}$	72.24 ± 0.01^{f}	$82.56\pm0.24^{\text{ab}}$	$88.89 \pm 1.00^{\text{b}}$	7.31 ± 0.02^{e}

T₀: onset temperature, T₁: peak temperature; T₁: final temperature; ∆H: enthalpy of gelatinization; NCS: native cassava starch; NYS: native yam starch; NSB-1: blend of cassava 30%-yam 70%; NSB-2: blend of cassava 30%-yam 40%; NSB-4: blend of cassava 45%-yam 55%; NSB-5: blend cassava 70%-yam 30%. Means followed by same letters within a column do not differ statistically according to Tukey's test (*P*<0.05), average ±standard deviation.

behavior in the transition temperatures. However, a nonadditive effect was observed in gelatinization enthalpies of the blends between NCS and NYS, so that the thermal energy required to trigger the gelatinization of the mixtures could not be predicted from the individual values of each counterpart because of certain phenomena of interaction with each other. Obanni and Bemiller (1997) report nonadditive behavior in the gelatinization enthalpy of binary blends of cassava-wheat and potato-rice starches, where the differences in granular size in each case could cause the stochastic behavior for ΔH . The non-additive variations observed in the crystallinity index of starch blends may be related to the unpredictable behavior observed in ΔH , while the thermal requirements to trigger gelatinization respond to the semi-crystalline characteristics of starchy materials. Furthermore, the concentration of starch present in the DSC analyzes of the present study (33%) could also play an important effect on the resulting non-additive behavior of ΔH .

In vitro digestibility

The fractions of rapidly digestible starch (RDS), slowly digestible (SDS), and resistant starch (RS) in gelatinized samples of NCS, NYS and their blends are represented in Table 3. At first, NCS and NYS showed differences between them (P<0.05) in terms of the proportion of RDS and RS, where NYS presented a higher content of RDS compared to NCS. It is relevant to show that the behavior of the starches *in vitro* digestibility indicates significant variations when the samples are gelatinized, demonstrating an increase compared to the native samples without gelatinizing. Similar results have been reported for RDS in native cassava (Jyothi *et al.*, 2005) and yam starches (Zhou & Kang, 2018).

RDS, SDS, and RS for the starch blends showed stochastic differences compared to their individual components. The foregoing is related to non-additive behavior of the

blends in some parameters related to RDS, SDS, and RS. For example, NSB-2 and NSB-3 presented RDS values significantly higher than the individual values of NCS and NYS, suggesting the interactions between the starchy constituents of the blend that cause unpredictable behaviors in the digestion of starch fractions in the first 20 min of the process. Similarly, the SDS fractions of NSB-3 and NSB-4 showed non-linear reductions relative to the individual counterparts. Ma et al. (2020) also report nonadditive variations on in vitro digestibility parameters for blends of rice starches with different amylose content, where stochastic increases and reductions in RDS, SDS, and RS of some mix ratios were appreciable. Thus, the variations in the amylose content of NCS and NYS, together with the differences in granular size and crystalline pattern, could have influenced the resulting nonadditive behavior of the mix ratios of starches evaluated in the present study.

Conclusions

There were changes in the physicochemical and structural properties of the cassava and yam starch blends compared to their native counterparts, as well as a general conservation of the granular morphological characteristics after the mixing process. The significant differences in the intrinsic properties of each starch exhibited a prevalent additive-type effect on amylose content and gelatinization temperatures, in contrast to the effects observed on the pasting parameters and crystallinity index of all the mix ratios evaluated that were non-additive. Specifically, the significant differences in amylose content and granular morphology of each individual starch would be responsible for the various resulting effects exhibited by mixing ratios. Thereby, the process of mixing native starches could be considered as an alternative mechanism in the polymeric matrix design with improved physicochemical properties from interaction

Samples	RDS (%)	SDS (%)	RS (%)
NCS	77.91 ± 0.63^{a}	10.52 ± 1.13^{a}	11.56 ± 0.74^{a}
NYS	$84.02\pm1.22^{\text{b}}$	11.25 ± 1.63^{ab}	$4.72\pm0.40^{\rm b}$
NSB-1	86.17 ± 1.03^{b}	11.50 ± 0.21^{ab}	$2.31\pm0.86^{\rm b}$
NSB-2	$88.48\pm0.39^{\circ}$	$7.45\pm1.50^{\text{ab}}$	4.06 ± 1.71^{b}
NSB-3	$89.23\pm0.91^{\circ}$	$6.29\pm0.30^{\text{b}}$	$4.47\pm1.06^{\rm b}$
NSB-4	$85.40 \pm 0.67^{\text{b}}$	9.87 ± 1.62^{a}	$4.72\pm0.94^{\text{b}}$
NSB-5	$86.19\pm0.42^{\text{b}}$	9.56 ± 0.30^a	$4.24\pm0.19^{\text{b}}$

RDS: rapidly digestible starch; SDS: slowly digestible starch; RS: resistant starch. NCS: native cassava starch; NYS: native yam starch; NSB-1: blend of cassava 30%-yam 70%; NSB-2: blend of cassava 50%-yam 50%; NSB-3: blend 60%-yam 40%; NSB-4: blend of cassava 45%-yam 55%; NSB-5: blend cassava 70%-yam 30%. Means followed by same letters within a column do not differ statistically according to Tukey's test (*P*<0.05).

Arroyo-Dagobeth, Figueroa-Flórez, Cadena-Chamorro, Rodríguez-Sandoval, Salcedo-Mendoza, and Cervera-Ricardo: Structural, physicochemical, and pasting properties of native cassava (*Manihot esculenta*) and yam (*Dioscorea alata*) starch blends

phenomena between individual starches where the resulting blends showed in the present case a low tendency for retrogradation, lower crystallinity index values compared to their native counterparts, and a regulated water absorption capacity, all potentially desirable characteristics in the food industry. However, the proportionate choice of each starch in the mixture is considered a determining factor of the resulting behaviors in the physicochemical and structural properties, therefore, the selection criteria of a particular mix ratio must be evaluated depending on the application or further processes of modification.

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Conflict of interest statement

The authors declare that there is no conflict of interests regarding the publication of this article.

Author's contributions

EDAD, JAFF, ECC designed the experiments; EDAD and JAFF carried out the experiments and data collection in laboratory experiments. EDAD, ERS, MACR and ECC contributed to the data analysis. EDAD, JAFF, JGSM, and ERS wrote the article. All authors reviewed the final version of the manuscript.

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