



Isolation and Characterization of Starch from the Purple Yam (*Dioscorea alata*) Anthocyanin Extraction Residue Obtained by Ultrasound-Assisted Extraction

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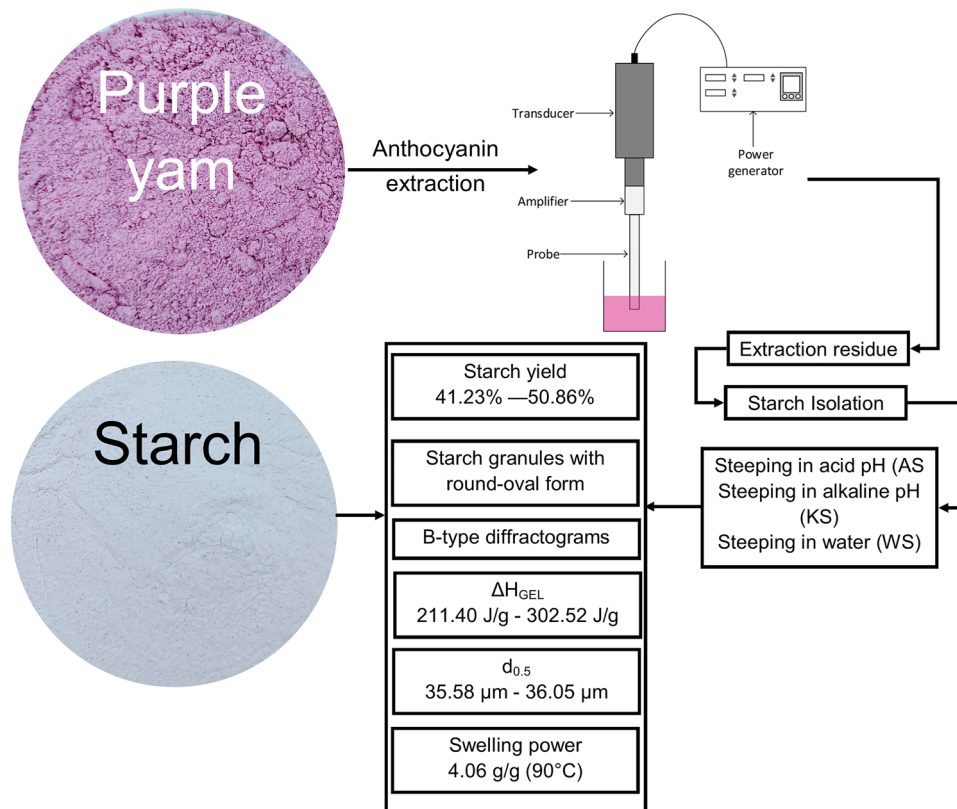
Abstract

Purple yam (*Dioscorea alata*) is a tuber crop found in tropical countries that could be a valuable source of anthocyanins and starch. After anthocyanin extraction, the starch from the extraction residue was isolated using steeping in acid pH (AS), steeping in alkaline pH (KS), and steeping in water (WS). The starch yield was higher in AS ($50.86 \pm 1.23\%$), followed by KS ($45.90 \pm 0.68\%$) and WS ($41.23 \pm 4.92\%$). WS starches presented softer round-oval forms, and AS and KS presented some layers in the spherical structures. Diffractograms showed a characteristic of a B type of starch with crystallinity between 43 and 48% for all isolation methods. Enthalpy of gelatinization was higher in the AS ($302.52 \pm 29.26 \text{ J g}^{-1}$) than KS ($211.40 \pm 35.09 \text{ J g}^{-1}$) and WS ($265.03 \pm 26.58 \text{ J g}^{-1}$). Monomodal distributions with mean volume diameter ranging between $35.58 \text{ }\mu\text{m}$ and $36.05 \text{ }\mu\text{m}$ were obtained for all isolation methods. As expected, the swelling power (SP) and water solubility index (WSI) increases as temperature increases for starches isolated using AS, KS, and WS. The isolation of starch from the residue of the extraction of anthocyanins from purple yam is suitable using AS, KS, and WS. Although no significant differences among isolation methods were observed in almost physicochemical parameters, AS isolation method produces starches with higher extraction yield and purity.

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Graphical Abstract



Keywords Purple yam extraction residue · Yam starch · Physicochemical properties

Statement of Novelty

Our study demonstrates the novel use of three different isolation methods, namely steeping in acid pH (AS), steeping in alkaline pH (KS), and steeping in water (WS), for the extraction of starch from the residue of anthocyanin extraction from purple yam (*Dioscorea alata*). We found that the AS method resulted in the highest starch yield ($50.86 \pm 1.23\%$), followed by KS ($45.90 \pm 0.68\%$) and WS ($41.23 \pm 4.92\%$). Additionally, the AS method produced starches with higher extraction yield and purity. This research provides valuable insights into the isolation of starch from a previously underutilized source and highlights the potential of purple yam as a viable and abundant source of both anthocyanins and starch.

Introduction

Starch-rich products such as potatoes, cassava, and yam are widely consumed worldwide. Among these crops, yam (*Dioscorea* spp.) is one of the most common tubers

consumed in tropical countries of Africa, Asia, South America, and the Caribbean due to its characteristics. Yam is an important crop for small-scale farmers in many countries, it provides a source of income and can help to improve food security in these regions. For example, West African countries cultivate around 90% of worldwide yam production, and more than 60 million people consume yam [1]. Among the most commonly cultivated types of yam are white yam (*Dioscorea rotundata*), yellow yam (*Dioscorea cayenensis*), purple yam (*Dioscorea alata*), air potato (*Dioscorea bulbifera*), and bitter yam (*Dioscorea dumetorum*) [2]. Yam is an excellent source of nutrients, including vitamin C, vitamin A, and potassium. For example, yam contains mainly starch (16–24%) and proteins (1.3–6.9%), as well as cellulose (1–7.5%), sugar (0.5–1.2%), fats (0.05–0.2%) and water (65–78%). Moreover, humans have consumed yam from ancient times due to its health benefits related to diabetes or anemia prevention and treatment [1].

Among the different kinds of yams, purple yam contains a group of phenolic compounds known as anthocyanins, which are responsible for their red-to-purple color. These compounds own bioactive properties. For example, they can

prevent cellular damage caused by stress conditions due to their antioxidant power [3]. As these compounds could be used as natural water-soluble colorants [4], anthocyanins can be extracted and used as natural food additives. However, after the extraction of anthocyanins from the purple yam, solid wastes rich in starch or other valuable compounds are generated. Starch represents around 20% of the total biomass of yam, and thus, the extraction residue could still retain almost all starch. Therefore, the residue of the anthocyanin extraction could be an excellent source of starch, and further recovery of the starch that remained in the extraction waste could be studied. Starch is a biodegradable hydrophilic polymer isolated from roots, legumes, cereals, and tubers such as purple yams. Amylose and amylopectin are the main components, and their ratio depends on the vegetal source. Moreover, starch is a low-cost ingredient used widely in the food and pharmaceutical industries [5]. For example, in the food industry, starch provides texture and serves as a thickener, emulsifier, gelling, or binding agent, among other specific functions. Moreover, yam starch can be used as a thickener in sauces and soups, a moisturizing agent in baked goods, a binder in meat products, and a viscosifier in beverages. [6]. On the other hand, non-food applications of starch in papermaking, textiles, and bioplastics are getting more attention, mainly through obtaining modified starches with better solubility and thermal properties [7]. Thus, purple yam and its anthocyanin extraction residues could be a valuable starch source. Moreover, corn, cassava, and potato are the primary starch sources. Therefore, non-conventional sources of starch as the residue of anthocyanin extraction could be an opportunity to valorize the purple yam crop. For example, the starches isolated from the anthocyanin extraction residue could have distinct properties of gelatinization, retrogradation, solubility, swelling power as well as water-binding capacity. In that context, the starches obtained from the residue could have specific functional properties that allow for improving their performance in products in the food and non-food industries. For example, Maniglia et al. [8] and Santana et al. [9] investigated the recovery of starch from turmeric wastes resulting from the extraction of dyes from turmeric using conventional (Soxhlet) and alternative extraction processes (supercritical fluid extraction and pressurized liquid extraction), respectively. In both works, starches with particular properties were obtained. In that context, starch isolation from a residue of the extraction of anthocyanins could contribute to the circular economy. This approach designs processes that reduce waste generation through re-utilization and recycling [10]. Moreover, a circular economy aims the development of processes that use raw materials intensively, for example, the residues of extraction processes. This work corresponds to the second

step of a project that aims to promote the use of purple yam in Colombia. In the first step, Ochoa et al. [11] extracted the anthocyanin using ultrasound-assisted extraction (UAE). In the second step (this work), starch from the residue of the anthocyanin extraction was isolated using steeping in acid pH (AS), steeping in alkaline pH (KS), and steeping in water (WS). Therefore, the objective of this work was to recover and characterize the starch from the anthocyanin extraction residue.

Material and Methods

Starch-Rich Residue

UAE was carried out using the procedure described by Ochoa et al. [11]. Anthocyanins were extracted from freeze-dried purple yam using a 750-W ultrasonic homogenizer (Cole-Parmer, Vernon Hills, IL, USA) under the following experimental conditions: 10 min of extraction time, 60% of amplitude, 60 °C, and an ethanol-to-water ratio, of 80:20. The extracts were filtered through a grade 3 qualitative-technical filter (65 g m⁻², pore size of 7–10 µm), and the starch-rich residue was dry in an oven at 60 °C for 48 h. Then, the starch-rich residue was milled and stored in hermetically sealed bags (Alico S.A., Colombia) at – 22 °C before use.

Starch Isolation

Three methods (AS, KS, and WS) were tested for starch isolation from purple yam anthocyanin extraction residue. The anthocyanin extraction residue was suspended in 1% (W/V) ascorbic acid solution (acid pH – 5.0) in AS, while in KS, a 2.5% (W/V) sodium hydroxide solution (acid pH – 10.0) was used. Deionized water (neutral pH – 7.0) was used for solid suspensions in WS. Then, the suspensions were stirred using a domestic blender (Waring Commercial, China) operating at maximum power for 1 min. The stirred suspensions were screened through 80-, 200-, and 270-mesh stainless-steel sieves with an aperture diameter of 180, 75, and 53 µm, respectively. The solids pie retained in the sieves were stirred and sieved again, four times for each method. Then, the slurry that passed through the sieves was centrifuged at 1500 × g for 10 min (Hermle, Z326K, Germany). The starches were re-suspended in the water while the supernatants were discarded. Finally, the starches were dried in an oven at 60 °C for 24 h (Memmert, model UN110, Schwabach, Germany). Then, the starches were milled and sieved through an 80-mesh sieve. The starches were stored in sealed dark flasks until further characterization. Experiments were carried out in triplicate.

Chemical Composition

The chemical composition of isolated starches was carried out using the methodology described by Estrada-León et al. [12]. Starches were analyzed in triplicate using AOAC methods to determine moisture (925.10), ash (923.03), proteins (920.87), and lipids (920.39) [13]. The amylose content, which is the linear fraction of starch that absorbs iodine and gives a blue complex, was determined by measuring the optic density at 600 nm. The amylose content was determined using a standard curve and expressed as a percentage. The sum of amylose and amylopectin corresponds to 100% of starch.

Starch Content

Total starch content was determined using an enzymatic kit purchased from Megazyme® (Bray Business Park, Bray, Co. Wick- low, low, Ireland), method number 996.11.

Scanning Electron Microscopy (SEM)

Scanning electron microscopy (SEM) was used to analyze the structure of starch granules. The samples were applied to circular aluminum stubs with double carbon sticky tape and coated with 200 Å of gold using a sputter coater (DENTON VACUUM, NJ, United States). The micrographs with a magnification of 300 were obtained using a scanning electron microscope (JEOL, JSM 6490 LV, Peabody, United States) at an accelerating potential of 15 kV and a current of 50 pA.

X-ray Diffraction (XRD) Analysis

The crystalline structure of starches was analyzed with an X-ray diffractometer (Empyeon 2012, Malvern-PANalytical, Netherlands) operating with sensor Pixel 3D, current of 40 mA, and a voltage of 40 kV. Samples were scanned over the range of 5°–50° with a step size of 0.05 and a step time of 50 s.

Differential Scanning Calorimetry

The thermal properties of the starches were determined by differential scanning calorimetry (DSC 2050; TA Instruments, New Castle, DE, USA). Approximately 1 mg was weighed directly into aluminum pans. Then, an aqueous

suspension containing 10% starch (w/w) was prepared with deionized water using a micro syringe. The heating was performed from 15 to 150 °C using a rate of 10 °C min^{−1}. The gelatinization temperature (T_O), the peak temperature (T_P), the end set temperature (T_E), and gelatinization enthalpy (ΔH_{GEL}) were calculated.

Particle Size Analysis

The particle size distribution was determined using a laser diffraction instrument (Mastersizer 2000, Malvern, England).

Swelling Power and Water Solubility Index

Swelling power (SP) and water solubility index (WSI) were measured according to Kusumayanti et al. (2015) with some modifications. 1 g of the sample was mixed with 10 mL of distilled water. Then, the solutions were heated in a water bath at 60 °C, 80 °C, and 95 °C for 30 min. Then, the samples were centrifuged at 315 g for 15 min. The SP and WSI were calculated using Eqs. 1 and 2, respectively.

$$SP (\%) = \frac{\text{weight of the sedimental paste (g)}}{\text{weight of the sample (g)}} \quad (1)$$

$$WSI(\%) = \frac{\text{weight of soluble solids (g)}}{\text{weight of the sample (g)}} \times 100 \quad (2)$$

Statistical Analysis

To compare the properties of the starches extracted from purple yam anthocyanin extraction residue, an analysis of variance (ANOVA) and Tukey test at a 5% significance level were performed. All analyses were performed using Minitab® version 16.

Results and Discussion

Chemical Composition

Table 1 shows the proximal composition of the starches isolated from the residue of the extraction of anthocyanins. The

Table 1 Chemical characteristics of starches isolated from the residue of the extraction of anthocyanins

	Moisture (%)	Proteins (%)	Lipids (%)	Ash (%)	Amylose (%)	Amylopectin (%)
KS	9.93 ± 0.74 ^a	0.15 ± 0.09 ^a	0.11 ± 0.05 ^b	0.11 ± 0.04 ^a	35.43 ± 0.71 ^a	64.57 ± 0.71 ^a
WS	10.07 ± 0.78 ^a	0.13 ± 0.04 ^a	0.12 ± 0.07 ^a	0.11 ± 0.03 ^a	35.55 ± 0.70 ^a	64.45 ± 0.70 ^a
AS	9.83 ± 0.31 ^a	0.15 ± 0.07 ^a	0.14 ± 0.04 ^a	0.10 ± 0.03 ^b	36.01 ± 0.32 ^a	63.99 ± 0.32 ^a

Means followed by different letters are significantly different ($p \leq 0.05$)

isolation method did not influence almost any parameters. Only lipid content and ashes showed significant differences ($p < 0.05$) among isolation methods. The moisture content of the three samples (KS, WS, and AS) ranged from 9.83% to 10.07%. These values are similar to other alternative or conventional starches such as parota [12] and corn [15]. The protein content in the three isolation methods was similar (0.15%), with no significant differences among the samples ($p > 0.05$). Therefore, the alkaline or acid medium did not solubilize the proteins in the starch. Similar behavior was observed in starch isolation from babassu mesocarp by AS and WS [16]. Moreover, protein values found in this work are lower than the protein values obtained in the starch isolation from annatto residue [17] and similar to elephant foot yam and green banana [18]. Lipid content ranged from 0.21% to 0.24%, with AS and WS having a higher lipid content than KS. Ash content around 0.11 was observed. These values agree with the values reported in the literature on starch isolation. However, KS and WS produced starch with higher ash content than AS. Similar behavior was observed for the starch isolation from annatto residue when the entry of Na ions into the starch granules resulted in an increase in the ash content.

Although no significant differences were observed among isolation methods, and starches isolated showed a low concentration of protein, lipids, and ash, further research is necessary to improve the purity of the starch extracted from the residue of the extraction of anthocyanins. On the other hand, no differences in the amylose content were observed in the starches isolated using the isolation methods. Starches with an amylose content of around 35% were obtained. This value is higher than the amylose content of other varieties of yam species. For example, they reported amylose content ranged between 26 and 35% in starches isolated from withe,

purple and yellow yam [19]. The amylose content of starches obtained is higher than commercial starches such as cassava (22%) and corn (28%) [20]. In this context, starch isolated from the residue of the extraction of anthocyanins could be used as a potential ingredient in functional foods and dietary supplements because starch with more amylose could have more resistance to digestion. Moreover, this starch could be used to produce biodegradable films and coatings for food packaging, texturizers in foods such as soups, sauces, and gravies, and an adhesive in various applications such as paper, textiles, and construction materials [21].

Starch Content

Figure 1 shows the starch yield (SY) and starch recovery (SR) of starches obtained from the residue of the extraction of anthocyanins. In this work, SY ranged between 41.23% and 50.86% was obtained. Moreover, SRs between 48.38% and 59.82% were observed. The literature reported different values of SY for some starch sources. For example, the SYs obtained in this work are higher than the values reported for Estrada-León et al. [12] and Vithu et al. [22] in the isolation of starch from parota (*Enterolobium cyclocarpum*) ($32.00 \pm 0.50\%$) and sweet potato ($16.38 \pm 1.47\%$), respectively. On the contrary, starch sources such as babassu mesocarp or pearled red sorghum have higher values of SY and SR. For example, Maniglia and Tapia-Blácido [16] obtained SY up to 85.00% in the starch isolation from babassu mesocarp using the AS isolation method. Moreover, an SY of 58.00% was reported by Micaela and Drago [23] in starch isolation from red sorghum using a sulfite/lactic acid solution. On the other hand, the results obtained in this work are similar to those found in the starch isolation from yam or other extraction residues. For example, Daiuto et al. [24] obtained SY of 43.64% and 42.73% using WS and KS isolation methods in the extraction of starch from Yam (*Dioscorea alata*). Regarding the starch isolation from anthocyanin extraction residues, the results obtained in this work are similar to those reported in starch isolation from curcuminoids and annatto pigment extraction residues. Santana et al. [9] isolated the starch after the extraction of volatile oil and curcuminoids from turmeric using supercritical fluid extraction and pressurized liquid extraction, respectively. In that work, after WS isolation, a YS of $36.00 \pm 3.00\%$ was obtained. In a similar approach, after the extraction of bixin by direct extraction with soybean oil from Annatto, the starch was isolated from the residue using AS, KS and WS. In that work, the SY was higher when WS ($32.00 \pm 1.00\%$) and AS ($31.00 \pm 1.50\%$) were used [17].

After performing a Tukey test, the AS isolation method was the method with the higher SY and SR, followed by KS and WS. Thus, the use of the ascorbic acid solution increases the extraction yield. Moreover, the sodium hydroxide water

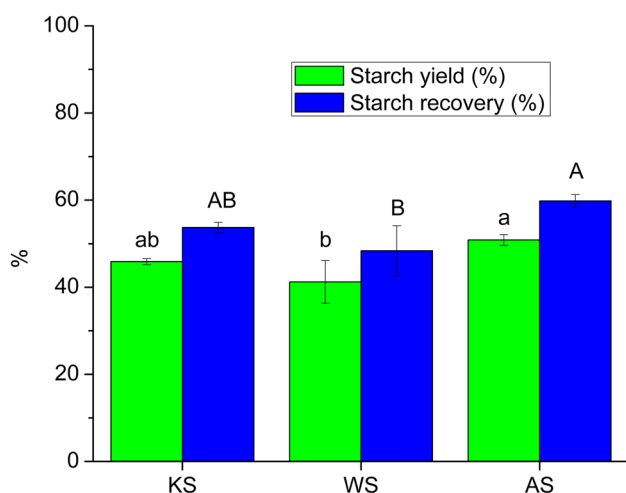
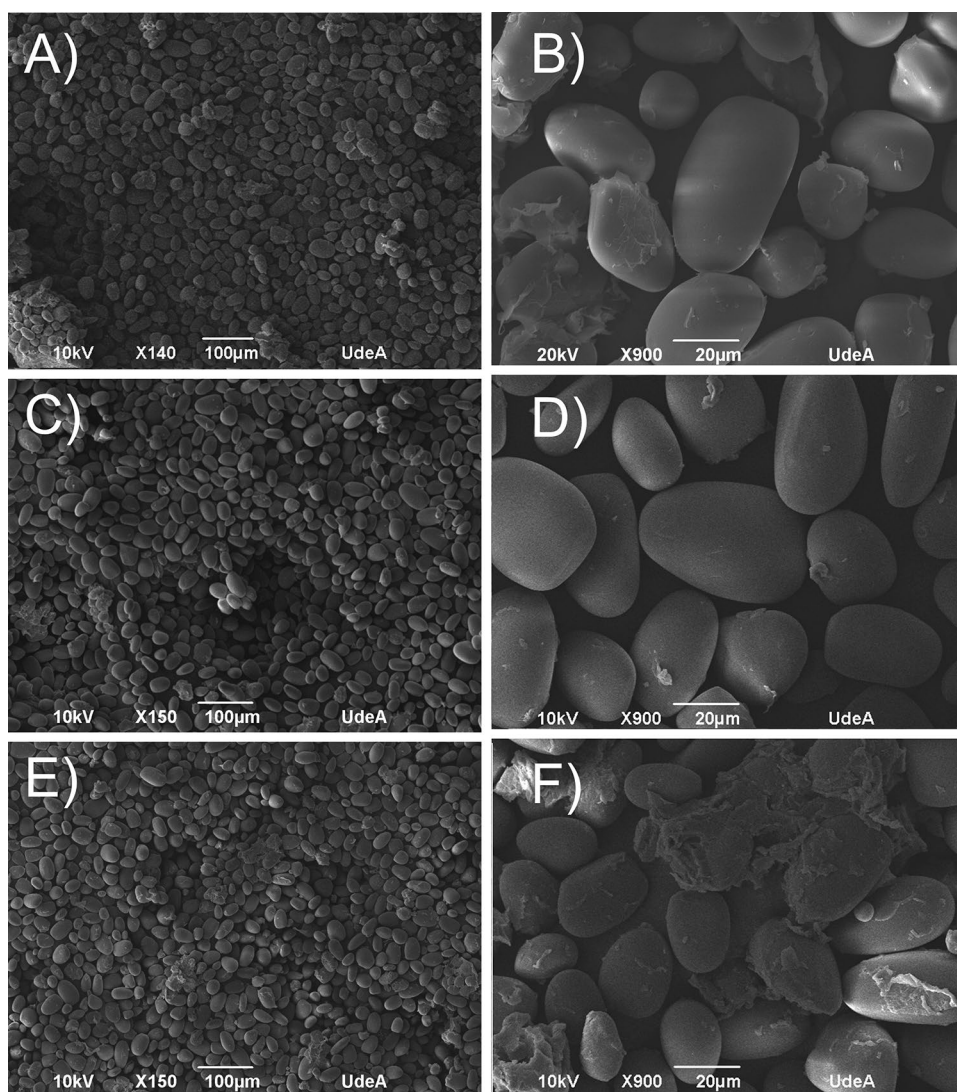


Fig. 1 Starch yield (SY) and starch recovery (SR) of starches from purple yam anthocyanin extraction residue

Fig. 2 Images from Scanning Electron Microscopy (SEM) of the starch granules obtained by methods KS (A and B), WS (C and D), and AS (E and F)



solution produces similar results. The use of an acid or alkali solution increases the SY and SR. This behavior has been observed by Garcia Silveira and Tapia-Blácido [17] and Estrada-León et al. [12], in the starch isolation from the residue of annatto pigment extraction and parota. Additionally, all starches presented a purity superior to 99.00%. This value indicates that all isolation methods are suitable for starch isolation. Moreover, the purity is similar to those obtained by Lopes Santos et al. [25] in starch isolation from purple yam (*Dioscorea trifida*), with purity of 99.70 and 99.61% for KS and WS, respectively. Thus, the residue of the extraction of anthocyanins from purple yam is an excellent source to obtain starches with high purity.

Scanning Electron Microscopy (SEM)

Figure 2 presents the SEM images of the starches obtained with the three isolation methods. For all isolation methods,

the starch granules possess a round-oval form. The spherical structure is very similar to those found in the isolation starch from different types of yam. For example, Lopes Santos et al. [25] studied the isolation of starch from purple yam (*Dioscorea trifida*) using KS and WS. In that study, the granule structure observed was round-oval and elongated. In another study, after starch isolation from yam (*Dioscorea alata* L.) using WS, Oliveira et al. [26] obtained irregular elliptical and round-oval starch granules. In this work, WS samples (Fig. 1 C and D) showed softer structures, while some layers were observed in KS (Fig. 2 A and B) and AS (Fig. 2 E and F) samples. This behavior was more intense in AS starch. For example, as shown in Fig. 2F, almost granules have some layers, which are more abundant in some granules. These layers are associated with fibers or proteins not removed during the isolation [9]. For example, mucilage is a yam compound composed mainly of mannan protein which could affect starch recovery and viscosity increase

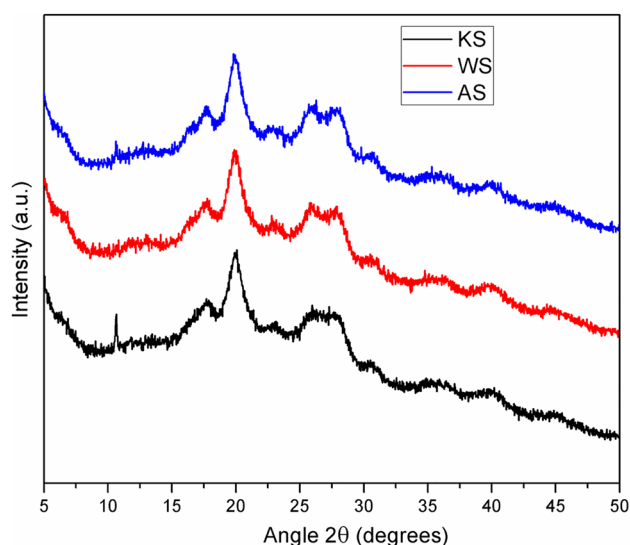


Fig. 3 X-ray diffractograms of starch from purple yam anthocyanin extraction residue

[25]. Perhaps the acid or alkaline conditions caused the precipitation over the granules of fibers or proteins and the further formation of these layers. Moreover, as the raw material was previously subjected to ultrasound-assisted extraction before the starch isolation, we expected some changes in the morphology. However, fractures or grooves caused by the ultrasound waves over the starch granules were not observed.

X-Ray Diffraction (XRD) Analysis

Figure 3 shows the diffractograms of starches from anthocyanin extraction residue. As can be observed, peaks in the 2θ diffraction angles at 17.80° , 19.94° , 26.31° , and 27.32° were identified. In this work, for all isolation methods, a strong diffraction peak was observed at approximately 20° . Similar diffractograms were found by Oliveira et al. [20] and Garcia Silveira and Tapia-Blácido [17] in the isolation of starch from yam. As expected, the diffractogram is characteristic of a B-type, found in tubers such as purple yam and other amylose-rich starches [27]. Moreover, a peak was observed at the 2θ diffraction angle of 11.64° for KS and AS, but with lower intensity in AS. It could be related to changes in the starch structure caused by the isolation method. Moreover, the crystallinity of KS, AS, and WS isolation methods was 49%, 48%, and 43%, respectively. Thus, the increase in the intensity of this peak could be related to an increase in crystallinity. Crystallinity values obtained in this work are higher than the values reported in yam by Lopes Santos et al. [25] (12%) and Oliveira et al. [20] (23%). However, these values are similar to the starch crystallinity of potatoes (45%) and different yam starches (24%–53.4%) reported by Lopez-Rubio et al. [28] and Zhu [29], respectively. Crystallinity is

Table 2 Thermal properties of starches from purple yam anthocyanin extraction residue

	ΔH_{GEL} (J/g)	T_p ($^\circ\text{C}$)	T_o ($^\circ\text{C}$)	T_E ($^\circ\text{C}$)
KS	211.40 ± 35.09^b	101.85 ± 4.08^a	47.73 ± 3.27^a	149.53 ± 3.29^a
WS	265.03 ± 26.58^{ab}	102.02 ± 1.66^a	52.43 ± 1.85^a	147.80 ± 0.30^a
AS	302.52 ± 29.26^a	100.20 ± 1.47^a	52.27 ± 0.97^a	147.87 ± 0.40^a

Means followed by different letters are significantly different ($p \leq 0.05$)

related to the amylopectin side chains, and its degree varies among starch sources. Crystallinity can be influenced by the yam type or the climatic conditions in the crop. According to Maniglia and Tapia-Blácido [16], the starch structure is related to the amylopectin side chains in the crystalline region. Therefore, the higher crystallinity may be caused by a high amylopectin content.

Differential Scanning Calorimetry

The thermal properties T_o , T_p , T_E , and ΔH_{GEL} are shown in Table 2. Values of ΔH_{GEL} between 211.40 ± 35.09 J/g and 302.52 ± 29.26 J g $^{-1}$ were obtained. According to the Tukey test, differences among isolation methods were observed, where starch obtained by AS is significantly higher than the KS starch. The values of the ΔH_{GEL} are higher than in other works. For example, ΔH_{GEL} of 12.15 J g $^{-1}$, 9.47 J g $^{-1}$, and 19.4 J g $^{-1}$ were obtained in starches from yam [20], banana [30], and parota [12], respectively. Higher values of ΔH_{GEL} (158.8 J g $^{-1}$) were reported by Noora et al. [31] in the isolation of starch from queen sago (*Cycas circinalis*) seed by AS. Higher ΔH_{GEL} is related to higher relative crystallinity. In this work, the crystallinity and ΔH_{GEL} obtained were high. Therefore, the granules are more resistant to gelatinization, and more energy is required to break the starch crystals. As can be observed in Table 2, the values for T_o , T_p , and T_E were around 50, 100, and 148°C . After performing Tukey's test, no significant differences were observed, and the behavior was similar for all isolation methods. These values are higher than ΔH_{GEL} observed in other starch sources. For example, after the starch isolation from other non-conventional sources such as babassu mesocarp flour [16], pitomba endocarp [6], and pearl millet [32], T_p of 66.90°C , 72.84°C , and 71.6°C were found, respectively. These results agree with those reported by Zhu [29], who stated that yam starches seem to have higher gelatinization temperatures than other tuber and root starches. Moreover, this behavior is similar to the gelatinization temperatures obtained by Lopes Santos et al. [25] in the isolation starch from purple yam (*Dioscorea trifida*). In that work, a T_p around 90°C was obtained after the isolation using KS and WS. The starch obtained from the residue of the extraction

Table 3 Mean particle size of starches from purple yam anthocyanin extraction residue

	Granule size ^a		
	d _{0,5} (μm)	D _{3,2} (μm)	D _{4,3} (μm)
KS	35.89 ± 0.65 ^a	16.75 ± 0.76 ^a	41.65 ± 5.11 ^a
WS	36.05 ± 1.45 ^a	16.71 ± 1.20 ^a	38.75 ± 4.81 ^a
AS	35.58 ± 0.89 ^a	16.85 ± 1.15 ^a	38.70 ± 5.28 ^a

Means followed by different letters are significantly different ($p \leq 0.05$)

^aThe d_{0,5} is the granule size at which 50% of all the granules by volume are smaller. The D_{3,2} and D_{4,3} are the surface-weighted and volume-weighted mean diameter, respectively

of anthocyanins has a high thermal resistance to gelatinization and stability [9]. Moreover, the high values of ΔH_{GEL} for all isolation methods suggest that granule microstructure is more stable and more energy is required to gelatinize. Moreover, as the ratio of long-chain to short-chain amylopectin is higher in B-type starch, the dissociation of amylopectin double helices requires more gelatinization starting energy, which could cause the higher values of ΔH_{GEL} [33, 34]. In addition, as the starch obtained has a higher amylose content than regular starches, it could have a higher number of hydrogen bonds and a more ordered structure, which requires more energy to break down and results in a higher enthalpy of gelatinization.

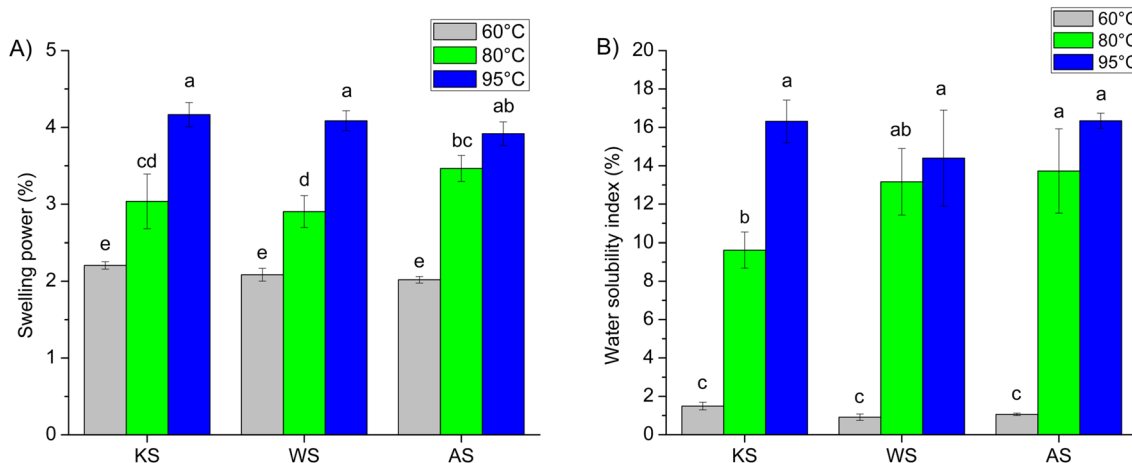
Particle Size Analysis

Table 3 shows the mean particle size of the starch obtained by KS, WS, and AS. Mean volume diameters (d_{0,5}) ranged between 35.58 μm and 36.05 μm were obtained with a monomodal distribution (Fig. not shown). The Tukey test showed

no significant differences in particle size among the isolation methods. The values obtained are slightly lower than the mean particle sizes of purple yam starch (40 μm) [35]. In this work, the granule size distribution was similar for all isolation methods. As was presented in the SEM images, the starches obtained from the purple yam anthocyanin extraction residue have a narrow particle size distribution and a low tendency to agglomerate. Values around 16.77 μm and 39.7 μm were obtained for D_{3,2} and D_{4,3}, respectively. The values found in this work are similar to those reported by Zou et al. [36] in the starch isolation from Chinese yam (*Dioscorea opposita* Thunb.). In that work, D_{3,2} and D_{4,3} values of 19.79 μm and 32.25 μm were obtained after soaked the yam flour in ethanol/sodium hydroxide (pH 8.5). Again, no significant differences among the isolation methods were found. Moreover, when the difference values (D_{4,3}-D_{3,2}) are analyzed, D_{3,2}-D_{4,3} values of around 23 μm are obtained. Thus, KS, AS, and WS produce starches with a similar particle size distribution, as mentioned before.

Swelling Power and Water Solubility Index

The swelling power (SP) and water solubility index (WSI) are shown in Fig. 4. As can be observed in Fig. 4A, similar SP values were obtained for all treatments when the test was performed at each temperature. For example, when the SP was determined at 60 °C, values around 2.1 g g⁻¹ were obtained for WS, KS, and AS. In the same way, for all starch isolation methods, SPs values around 3.1 g g⁻¹ and 4.06 g g⁻¹ were obtained at 80 °C and 95 °C, respectively. These values are lower than the values reported in other studies. For example, SP of 6.5 g g⁻¹ at 95 °C was obtained from yam (*D. alata* L.) starch from Brazil [20]. However, parameters such as SP are influenced by the yam starch source. For example, SP values from 2.74 g g⁻¹ to 16.8 g g⁻¹

**Fig. 4** Swelling power (A) and water solubility index (B) of starch from purple yam anthocyanin extraction residue

at 60 °C have been reported by them in *D. Opposita* and *D. Alata*, respectively [29]. Thus, the results obtained in this work agree with the literature. On the other hand, when a Tukey test was performed, no differences among AS, KS, and WS were observed in each temperature. However, significant differences were observed in the temperature. For example, when the temperature increases from 60 °C to 95 °C, the SP of the KS starch increases from 2.21 g g⁻¹ to 4.17 g g⁻¹, which represent an SP 1.8 time higher. As expected, the temperature promotes the formation of bonds with water molecules and, thus, an increase in the SP.

Regarding solubility, WSI ranged from 0.91% to 16.34% were obtained. The values of WSI in each temperature were similar, but when the temperature increased, the WSI increased too. This behavior was observed by [15] in starches from cereals, roots, tubers, and peas. On the other hand, significant differences among the isolation methods and temperatures were observed. As expected, the lower WSI was obtained at a lower temperature (60 °C). Moreover, when the temperature increased from 60 °C to 80 °C and 90 °C, as expected, the WSI increased significantly for all isolation methods. The solubility values obtained by KS are higher than those reported in the starches from sweet potatoes [14, 22] and Chinese yam [37], among others. Moreover, when the temperature increased from 60 °C to 80 °C and 90 °C, as expected, the WSI increased significantly for all isolation methods. In that context, some yam starches own higher WSI values than other starch sources. Even higher values of the WSI obtained in this work have been reported. For example, WSI values of 21.55% and 18.55% were reported in starches from *D. nipponica* Makino [38] and *D. bulbifera* Linn [39].

Conclusions

Starches from the purple yam anthocyanin extraction residue showed similar results in almost physicochemical properties. SY and SR varied from 41.23% to 50.86% and from 48.38% to 53.71%, respectively. Although all isolation methods allow the obtaining of starches with a purity higher than 99%, AS showed the highest yield. The structure of all starches was spherical, with softer shapes in WS samples. Some layers around the granules were observed in AS and KS. The diffractograms showed a strong diffraction peak in the 2θ diffraction angle typical of B-type starches. This behavior is commonly observed in tubers such as purple yams. High crystallinity and ΔH_{GEL}s were observed in all starches, where AS produced the starch with the highest ΔH_{GEL}. The starches isolated from the extraction residue of purple yam presented a narrow particle size distribution ranging around 36 μm with a low tendency to agglomerate.

Although similar values of SP among the starches, the solubility obtained is lower than the values reported in other studies. Thus, further research regarding the modification of the starches is necessary to improve the solubility. For example, starch isolated for anthocyanin extraction from purple yam could be used as raw material for its chemical or physical modification and further use in food or textile industries (e.g., viscosifier or textile Finishing). However, the results obtained in this work showed that the residue of the extraction of anthocyanins is a valuable source of starch. Specifically, the starch obtained in this work will be used as raw material for starch hydrolysis. The isolation of starch from the extraction residues contributes to the circular economy and allows the intensive utilization of purple yam.

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Data Availability Data included in article have been published on Mendeley Data, V1: <https://data.mendeley.com/datasets/f8rfr5ddvg/1>

Declarations

Conflict of interest The authors confirm that there are no conflicts of interest regarding this paper.

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