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Physicochemical characterization of dehydrated starches extracted from tubers from La Paz

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Abstract

The extraction of starches from four species of tubers, namely potato (*Solanum tuberosum*), oca (*Oxalis tuberosa*), papalisa (*Ullucus tuberosus*), and Isaño (*Tropaeolum tuberosum*) and their dehydrated products, was carried out to characterize the physicochemical parameters, the microscopy of the starch granules, enzymatic hydrolysis, and gel properties, and to relate the behavior of the starch granule and study its variation with respect to fresh and dehydrated tubers. The results show a difference in the percentage of starch yield depending on the species and its fresh and dehydrated state. A relationship was observed between the content of amylose/amylopectin, morphology of the starch granule, size of the starch granule, and percentage of glucose obtained from the enzymatic hydrolysis process, where starches that have a lower amylose content have smaller, spherical or ellipsoidal granules and are more prone to enzymatic attack. The properties of the gel such as maximum viscosity, breakdown, and setback of the starches obtained were also studied. The sample starches were characterized using X-ray diffraction to identify their crystal structure.

Keywords: viscoamylography; x-ray diffraction; scanning electron microscope; enzymatic hydrolysis.

Practical Application: It was observed that the process to which fresh tubers are subjected to obtain their dehydrated starches influences their physical and chemical characteristics. A relationship was found between granule morphology and amylose/amylopectin content. X-ray diffraction patterns could corroborate the crystallinity of the starches studied. It was also observed that the amount of amylose/amylopectin is related to other physical properties such as gelation and crystallinity, for each group of samples studied.

1 INTRODUCTION

Andean crops show great genetic diversity, which leads to diversity in the chemical composition, depending on the part of the plant that is used as food for humans, that is, tubers, roots, and grains (Mejía Cabezas et al, 2021) . The tubers are thickened stems of a plant, generally underground, that store nutrients and contain shoots from which new stems grow (Green Facts, 2023). Andean tubers constitute an important regional resource, growing in lands with more than 3000 m of altitude in the Andean region, and these foods have been consumed for more than 3000 years by Andean peoples such as Incas, Quechuas, and Aymaras (Cruz et al., 2016; Segura Ortega et al., 2024). Among the most widespread crops is the potato (*Solanum tuberosum*) with approximately 80% water and 20% dry matter, with starch being the main component, which is important since it reaches 70% of the total solids. The oca (*Oxalis tuberosa*) is the most common tuber in the Andean region after potato, and finally, the papalisa (*Ullucus tuberosus*) which is grown at altitudes of 3000–3800 m in the central and southern region of the Andes (Bolivia, Peru, Ecuador, Colombia, Venezuela, and Chile), being unknown outside the Andean region (Iturriaga et al., 2022). Regarding Isaño, this tuber is native to the Andes; geographically, it is distributed from Colombia to Bolivia between 1500 and 4200 m above sea level. In addition to having a high nutritional value, it has medicinal properties, which is being traditionally used as anti-inflammatory and antibacterial agent; it is also used as an insecticide and nematicide. However, the production of this crop is marginal due to lack of knowledge of its nutritional and medicinal values and because there is no greater demand for consumption by the population (Aruquipa et al., 2016; Chuquilín Goicochea, et al., 2020).

To provide a longer shelf life to these foods, pre-Columbian inhabitants used dehydration (freezing and drying processes) as a preservation method, becoming one of the oldest processing techniques, and currently, it remains the most popular method for food preservation (Aruquipa et al., 2016). Following this method, products known as tunta and chuño (dehydrated potatoes), white caya and black caya (dehydrated OCA), and lingli or chullcce (dehydrated papalisa) were obtained, and they became foods of high consumption and with important commercial value throughout the Andean region.

Starch constitutes the main source of food reserve in plants, necessary for their germination and subsequent growth. Starch is a mixture of polysaccharides, which is present in the form of

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granules composed of amylose and amylopectin, which mainly influence the rheological and sensory properties of foods due to their hydration and gelatinization capacity (Marquez Mendoza, 2019). The behavior of starches depends on the physicochemical composition of the botanical source, the state of maturity of the plant, variety, growth zone, and fertilization (Marquez Marusic, 2022).

The physicochemical, functional, and structural properties of starches vary significantly between and within botanical species, even when the same plant cultivar is grown under different environmental conditions (Hancco Cayllahua, 2021). These variations are also influenced by the size of the starch granule, the content of phosphorus and amylose, the amylose–lipid complex, and the structure of amylopectin (Barraza-Jáuregui et al., 2020; Martínez, 2019). Martínez et al. (2019) pointed out that starches from different botanical sources differ considerably in their properties, even finding that each granule in the same population is different from the other granules, both in its fine structure and properties. Iturriaga et al. (2022) mentioned that the functional properties of starch, such as its ability to thicken and gel in food systems, depend mainly on the amylose: amylopectin ratio and the size and distribution of the granules, among others. Marquez Marusic (2022) indicated that both the size and the shape of the starch granule are characteristic of each botanical species. The difference in these characteristics is being used in the development of different microscopic methodologies to identify the source of different starches. Among other important characteristics of starches, it is necessary to note viscosity, which is related to the distribution of chains of amylopectin molecules. Furthermore, amylopectin with a higher molar mass generates more viscous pastes at high temperatures, that is, the higher the viscosity values in the extracted starch, the more viscous pastes generated will be (Ligarda Samanez et al., 2020).

In the present paper, the physicochemical properties of starches extracted from tubers from the Department of La Paz (Bolivia), the microscopic characteristics of the starch granules, the glucose content present in the hydrolysates, and the thermal properties of each starch sample were studied.

1.1 Relevance of the work

This work is based on the study of the physicochemical characteristics of starches extracted from various native tubers of the Department of La Paz-Bolivia, which was conducted from the microscopic morphology of the granule to X-ray diffraction spectra, demonstrating that all starches have general characteristics as well as particular characteristics. It was observed that the process to which fresh tubers are subjected to obtain their dehydrated starches influences their physicochemical characteristics.

2 MATERIALS AND METHODS

2.1 Reagents

The reagents used were anhydrous sodium sulfite (p.a., PANREAC QUIMICA S.A. Barcelona. España); amylose, Type III: from potato, essentially free of amylopectin (Sigma Aldrich, Chemie GmbH, Steinheim, Germany) and amylopectin, from potato starch (Sigma Aldrich, Chemie GmbH, Germany); enzyme BAN 480L (Novozymes A/S, Krogshoejvej 36, 2880-Bagsvaerd, Dinamarca) and enzyme Dextrozyme DX 1.5X (Novozymes A/S, Krogshoejvej 36, 2880-Bagsvaerd, Dinamarca); anhydrous glucose (p.a., Sigma Aldrich, Chemie GmbH, Steinheim, Germany); and DNS reagent (3,5-dinitrosalicílico acid, p.a., Sigma Aldrich, Chemie GmbH, Steinheim, Germany).

2.2 Materials

The analyzed samples correspond to starches extracted from tubers such as Black Imilla potato (*S. tuberosum*) from the Pacajes Province, oca (*O. tuberosa*), papalisa (*U. tuberosus*), Isaño (*Tropaeolum tuberosum*), and black Isaño (*T. tuberosum*), of which the latter four were obtained from the municipality of Sorata, and dehydrated tubers such as chuño were obtained from the municipality of Calamarca, tunta from the municipality of Collana, white caya and black caya from the municipality of Achacachi, and chuño from papalisa. All samples were obtained from the Department of La Paz.

2.3 Processing of the samples

Depending on the sample being processed, the sample was washed, dried, liquefied, or ground to homogenize, thus achieving a value that is as representative as possible for the analyses carried out.

2.4 Starch extraction

For starch extraction, the method reported by Guzmán Condarco (2014) was used. A 0.005 M solution of anhydrous sodium sulfite (p.a.) was prepared and added to the previously treated sample in order to avoid enzymatic browning. It was then allowed to rest for 24 h at room temperature, and the solution was filtered using a standard testing sieve: sieve no. 120 of 125 μm (VWR, USA). The solid residue was washed several times with distilled water to remove all the starches from the plant residue; subsequently, it was allowed to settle for 24 h, during which the water present and the starch obtained were separated. It was washed twice with distilled water, and the third wash was carried out with 96% alcohol, and finally, it was dried at room temperature.

2.5 Determination of amylose and amylopectin content

For the amylose–amylopectin analysis, a Type III amylose standard (from potato, essentially free of amylopectin) and a potato starch amylopectin standard were used. The amylose–amylopectin content was determined using the International Organization for Standardization (ISO) standard (1987) cited by Huanca Rengel (2013), which is based on the colorimetric measurement of the iodine–amylose complex, taking the readings at 620 nm using the Cytation 3 cell plate reader, an imaging multi-mode microplate reader (Biotek Instruments, Inc. Winooski, USA).

2.6 Morphological analysis of starch granules by optical microscopy

For the morphological characterization and photographic recording of the starch granules, an Optika Vision Pro optical microscope (M.A.D. Apparecchiature Scientifiche S.R.L., Ponteranica, Italy) with a built-in USB digital camera was used. The image capture of starch granules was performed at a magnification of 20 ×.

The characterization of the starches was complemented by the use of scanning electron microscope (SEM), Philips/FEI (XL30) SEM and Phillips XL 30 multipurpose SEM. This technique allowed for observation at higher magnifications and in three dimensions, corroborating certain details of their composition and surface.

2.7 Starch granule size distribution – Biotek

The measurement of starch granule sizes was carried out using the Cytation 3 cell imaging multi-mode microplate reader (Biotek Instruments, Inc. Winooski, USA), which has a built-in gray-scale CCD camera with a Sony chip for image collection. The CCD sensor has 1384 × 1036 pixels, with an image size of 1280 × 960 pixels. A built-in tube lens captures images directly through the selected lens. This way the statistical analysis was carried out on the captured images using one of the calculation tools provided by the software.

2.8 Enzymatic hydrolysis

The enzymatic hydrolysis process consists of two stages: liquefaction and saccharification.

Following the methodology described by Huanca López et al. (2015), a 3 0% starch solution was prepared, the pH was adjusted to 6, then calcium ions and the BAN 480L alpha-amylase enzyme were added and then incubated at 70°C for 90 min. For the second stage, "saccharification," the work was carried out at a pH between 4.02 and 4.05, the enzyme Dextrozyme DX 1.5X amyloglucosidase was added and incubated at 60°C for 72 h, and subsequently, the enzyme was deactivated.

2.9 Glucose quantification in hydrolyzed samples

The quantification of glucose in the hydrolyzed samples was carried out according to the method described by Huanca López (2014), based on the colorimetric measurement of 3-amino-5-nitrosalicylic acid, a compound that results from the reaction between glucose and the DNS reagent (3,5 dinitrosalicylic acid). Readings were taken at 540 nm using the µQuant plate reader with ultraviolet–visible (UV–Vis) spectrophotometer (Biotek Instruments, Winooski, USA).

2.10 Properties of the gel by viscoamylography

Following the method of Yañiquez Vedia (2019), a Micro Visco-Amylo-Graph Brabender (Model 803202, made in Germany) was used to study the properties of the gel. To prepare the sample, 10.0 g of starch was weighed into the equipment's container, 100 mL of distilled water was added, and the sample was homogenized. The homogenized sample was introduced into the Brabender equipment and the program was configured on a computer to measure the sample, recording the humidity value corresponding to the sample and the volume of water that was used for homogenization. The heating stage in the equipment and the program used for the analysis of the viscosity and gelatinization properties of starches began at 30°C and heated to 85°C for 7.33 min. This temperature was maintained for 5 min, then cooled to 50°C for 4.37 min, and finally it was maintained at 50°C for 3 min. The heating–cooling rate was 7.5°C/min.

2.11 X-ray diffraction

X-ray diffraction (XRD) measurements of the starches were carried out using the PanAnalytical Expert Plus Diffraction (XRD) equipment with a Cu (copper) X-ray generator tube and an Ni (nickel) filter, giving K alpha radiation of Cu with a wavelength of 1.54178 Å, under the following operating conditions: radiation at 40 kV and 40 mA, recording range from 3º to 60º, detection with a 125-channel XCELERATOR, and an automatic detection scale.

3 RESULTS AND DISCUSSION

The results obtained from the different analyses carried out on the studied samples were grouped as follows: Group 1: papa, tunta, and chuño; Group 2: oca and white caya and black caya; Group 3: papalisa and papalisachuño; and Group 4: Isaño and black Isaño.

3.1 Starch extraction yield in percentage

The yield of the starch extracted from both the tubers and the dehydrated samples analyzed in this work is shown in Table 1.

Within Group 1 (Table 1), tunta presents the highest percentage of starch extraction compared to the other two samples (potato and chuño). Regarding Group 2, the highest percentage of starch extraction corresponds to black caya. Between papalisa and papalisachuño (Group 3), the latter shows a higher starch extraction yield. Regarding Group 4, the Isaño has a higher starch content compared to the black Isaño. It can also be observed, based on the results obtained in Table 1, that in

Table 1. Percentage of starch yield in percentage.

	Sample	%RA
Group 1	Black potato Imilla (Pacajes)	23.1 ± 1.1
	Tunta (Collana)	56.1 ± 0.8
	Chuño (Calamarca)	39.9 ± 1.1
Group 2	Oca (Sorata)	11.8 ± 0.7
	White Caya (Achacachi)	22.8 ± 1.2
	Black Caya (Achacachi)	26.0 ± 0.7
Group 3	Papalisa (Sorata)	10.1 ± 0.5
	Chuño de papalisa	25.9 ± 0.7
Group 4	Isaño (Sorata)	8.6 ± 0.8
	Black Isaño (Sorata)	3.4 ± 1.1

%RA: starch extraction yield in percentage.

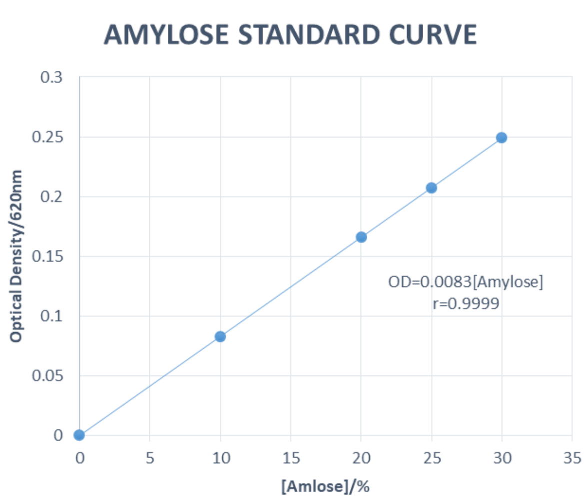
Groups 1, 2, and 3, the samples that present the highest starch yield correspond to the products obtained by dehydration, with or without exposure to solar radiation. This result was expected because the fresh samples have a higher percentage of water in their composition compared to the dehydrated samples, which is part of the initial weight taken to carry out the extraction of starch from fresh samples. This indicates that the composition of the initial weight taken for analysis in fresh samples is different from that of dehydrated samples due to the higher water content present in fresh samples. Group 4 is made up of fresh tubers (Isaño and Black Isaño), with Isaño being the sample that presents the highest percentage of starch extraction compared to Isaño negro, thus demonstrating that even in samples of the same genus and species, the content of starch varies.

3.2 Amylose – amylopectin quantification

The quantification of amylose and amylopectin of the different starches extracted from tubers and dehydrated samples was carried out based on the construction of the calibration curve or standard curve of the amylose standard (Graph 1).

Using the amylose standard curve, in Table 2, the results for the tuber and dehydrated groups can be studied. Within Group 1 and Group 2, chuño starch and black caya starch had the highest amylose content; in Group 3, it was observed that the amylose content in papalisa starch and papalisachuño starch was similar, showing a difference of only 0.2%. On the contrary, in Group 4, a difference of 21% in amylose content was observed between Isaño starch and black Isaño starch, with the latter being the one that has the highest amylose content compared to Isaño starch. Group 4 showed a clear example of tubers that belong to the same species and can have different chemical compositions.

In a global comparison of the results obtained in this analysis, it was observed that the tubers obtained by freezing, dehydration, and with exposure to solar radiation have a higher amylose content. This could probably be due to the breaking of the branches of the amylopectin chains by solar radiation. Since this method does not differentiate the degrees of polymerization of the amylose chains, the fractionated amylopectin would be added to the percentage of amylose. Valdivieso Molina and Mollinedo (2021) reported that treatment with UV radiation directly influences the composition and structural characteristics of potato starch, causing the photodegradation of starch, where the main photochemical reactions are chain excision, dehydroxylation, dehydrogenation, and the breaking of C–C bonds of the glucose structure, thus generating the shortening of the amylose chain and the debranching of the amylopectin chains. Furthermore, exposure of starch to UV radiation leads to physical changes, such as water loss, conformational transformation, and the formation of less ordered structures than innate polysaccharides (Valdivieso Molina & Mollinedo, 2021). Therefore, the formation of less ordered structures implies the presence of a higher content of amylose (the amorphous part of starch) and loss of the crystalline structure whose characteristic is given by amylopectin. For this reason, as shown in Table 2, the products obtained by freezing and dehydration with exposure to solar radiation have a higher amylose content compared to fresh tubers.



Graph 1. Amylose standard curve obtained at 620 nm.

On comparing only the fresh samples, it was observed that not all starches show the same amylose/amylopectin ratio. This is because the climate, cultivation site, age, starch source, species, and variety are among some parameters that play a very important role within the characteristics that the tuber presents. This difference in the amylose/amylopectin content allows starches to have variations in characteristics such as viscosity, gelatinization, texture, solubility, swelling power, water absorption index, gel stability, and retrogradation (Guevara Lastre, 2020).

3.3 Morphological properties

Using the Optica Vision Pro microscope with a magnification of 20 × and the SEM at 500 × (Figures 1 and 2), the morphological structures of the starch granules, corresponding to the tuber samples––both fresh and dehydrated––studied in the present work, were observed (Table 2). For the starch granule size distribution, the Cytation 3 cell imaging multi-mode microplate reader was used, using the calculation tool provided by the software based on the captured images. Observations of the photomicrographs obtained with both microscopes revealed differences in the shape of the starch granule, location of the hilum, and granule size. Group 1 starch granules exhibited spherical and ellipsoidal shapes, exhibiting a centric and eccentric punctual hilum with mixed sizes of starch granules. The starch granules of Groups 2 and 3 exhibited spherical, ellipsoidal, and irregular shapes, showing an eccentric punctual hilum. As for Group 4, the Isaño exhibited starch granules with a spherical shape and a centric hilum, with the granules mostly small in size, and the black Isaño exhibited spherical, ellipsoidal, and truncated morphology, exhibiting a centric and eccentric punctual hilum, with mixed granule size.

In general, the starches of the studied samples present mixed populations in terms of granule morphology; small granules exhibit a spherical or ellipsoidal shape, medium and large granules exhibit an ellipsoidal, irregular, or truncated shape. With respect to the size distributions based on the diameter of the starch granule (Table 2), it was observed that all the samples studied were made up of large granules (> 20 μm), medium granules (> 10 to 20 μm), and small granules (1 to 10 μm) with the

Table 2. Chemical composition, size and distribution of granules, total glucose, and gel properties of starches isolated from fresh and dehydrated tubers.

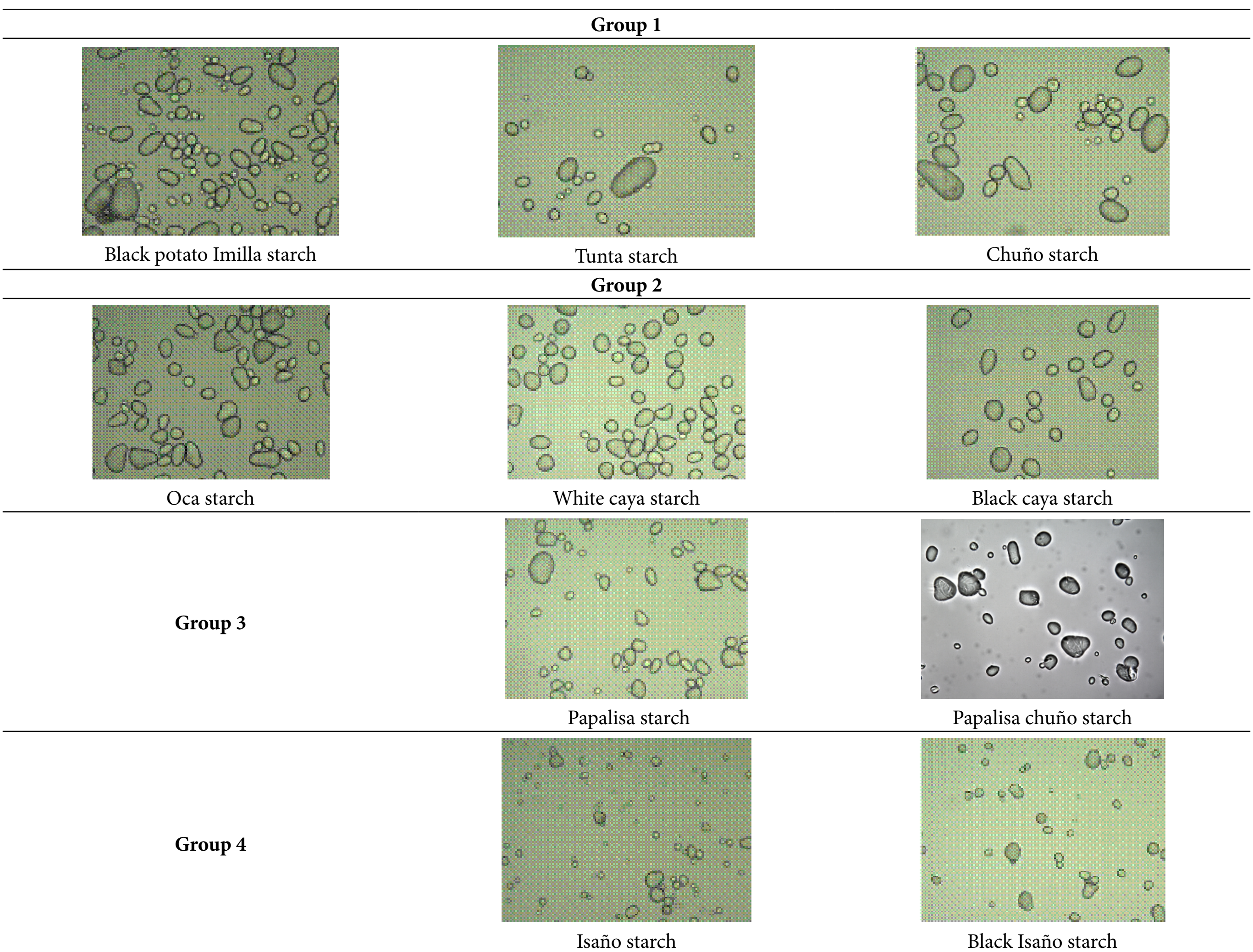
	Group 1			Group 2			Group 3		Group 4	
Sample starch	Black potato Imilla (Pacajes)	Tunta (Collana)	Chuño (Calamarca)	Oca (Sorata)	White Caya (Achacachi)	Black Caya (Achacachi)	Papalisa (Sorata)	Papalisa Chuño	Isaño (Sorata)	Black Isaño (Sorata)
Amylose %	22.7	19.2	26.4	19.2	20.1	20.9	15.8	16.0	7.6	28.6
Amylopectin %	77.3	80.8	73.6	80.8	79.9	79.1	84.2	84.0	92.4	71.4
Particle size										
Small (1 to 10 µm) %	43.3	49.8	30.4	10.1	1.8	0.4	34.6	51.7	89.1	50.3
Medium (> 10 to 20 µm) %	29.7	29.1	32.6	50	41.4	31.5	45.2	39.1	10.9	48.6
Big (> 20 µm) %	27.0	21.2	37.0	39.9	56.8	68.1	20.2	9.2	0	1.2
Microscopic image										
Granule shape	Spherical and elliptical	Spherical and elliptical	Spherical and elliptical	Spherical, elliptical, and irregular	Spherical, elliptical, and irregular	Spherical, elliptical, and irregular	Spherical, elliptical, and irregular	Spherical, elliptical, and irregular	Spherical	Spherical, elliptical, and truncated
Hilum localization	Centric and eccentric punctual	Centric and eccentric punctual	Centric and eccentric punctual	Eccentric punctual	Eccentric punctual	Eccentric punctual	Eccentric punctual	Eccentric punctual	Centric punctual	Centric and eccentric punctual
Enzymatic hydrolysis										
Total glucose %	69.6	92.8	80.2	96.6	86.0	82.3	80.4	69.1	59.5	85.2
Gel properties										
Initial gelatinization temperature (°C)	63.1	60.0	61.9	57.0	57.5	56.7	58.0	61.0	57.4	59.6
Gelatinization temperature (°C)	78.7	78.9	69.1	62.2	64.0	61.9	66.2	76.6	65.2	65.9
Maximum viscosity (BU)	3399	2562	2805	3470	2929	3083	4056	2303	4332	3556
Breakdown (BU)	1049	462	570	2035	1219	1748	2434	673	2339	1715
Setback (BU)	1116	567	693	507	450	334	956	358	826	809

exception of Isaño starch, which presents the absence of large granules, which is why it only shows a spherical shape within the morphology of its starch granule. Starches from chuño, black caya, papalisa, and black Isaño showed greater proportions of large ellipsoidal granules, while starches from tunta, oca, chuño de papalisa, and Isaño showed a greater proportion of small spherical granules. The variation in the morphology and size distribution of the starch granule can be attributed to the biological origin, physiology of the plant, and processes or treatments to which the fresh tubers were subjected to obtain their dehydration (Del Águila Meléndez, 2022; Singh & Kaur, 2004; Valdivieso Molina & Mollinedo, 2021).

3.4 Relationship between amylose content, morphology, and size distribution

The chuño (Group 1), the black caya (Group 2), and the black Isaño (Group 4) had the highest amylose content compared to the other components of the same group, and they are the ones with the highest percentage of starch granules > 20 µm and are ellipsoidal, irregular, and truncated in shape. On the contrary, the starches from tunta (Group 1), oca (Group 2), and Isaño (Group 4) exhibited spherical and ellipsoidal shapes and a higher percentage in starch granules from 1 µm to 10 µm. Therefore, they present the lowest percentage of amylose content with respect to the other samples that are part of the same group. With respect to Group 3, the amylose content between papalisa starch and papalisachuño starch does not present much difference (Table 2); however, with respect to the starch granule size, papalisa starch had the highest percentage of starch granules > 20 µm and the highest content of granules from 1 µm to 10 µm corresponded to papalisachuño starch.

Relating the amylose content (Table 2) to the morphology and size of the starch granule (Table 2), it was observed that at a higher amylose content, the starch granules have a larger size with an ellipsoidal-to-irregular shape, while at a lower content of amylose, starch granules are smaller in size with a spherical or ellipsoidal shape. Singh and Kaur (2004) reported similar characteristics in their study of potato starch fractions according to granule size, in which the large-sized fractions showed higher amylose content and ellipsoidal-to-irregular or cuboidal



Source: Huanca López (2017).

Figure 1. Photomicrographs of starch granules at 20 × obtained with the Optika Vision Pro-Italy microscope.

morphology and the small granules presented spherical or ellipsoidal morphology. It can also be observed that the size of granules and their morphological structure (spherical, elliptical, irregular, and truncated) differ between botanical sources (papa, oca, papalisa, Isaño, and black Isaño). There is a wide diversity in the structure, physicochemical characteristics, and composition of the native starch granule, including significant variations between granules of the same species, possibly due to their different origins, harvest times, plantation environment, climate, and soil, among others (Lagos, 2016; Ren et al., 2020).

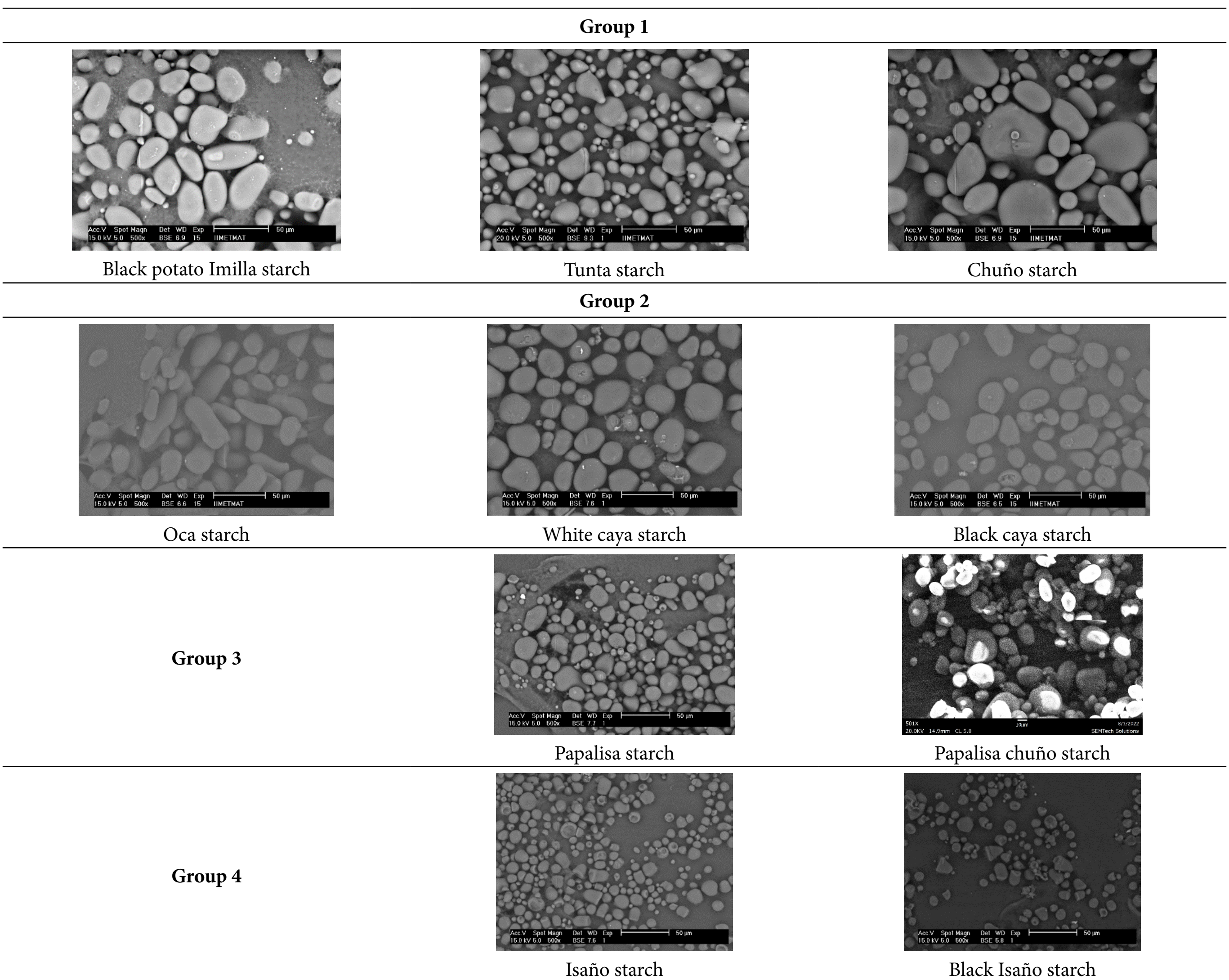
3.5 Enzymatic hydrolysis – Total glucose percentage

The quantification of total glucose of the different hydrolysates, obtained from starches extracted from tubers and dehydrated samples, was carried out using the DNS spectrophotometric method (3,5-dinitrosalicylic acid) based on the construction of the calibration curve or standard curve of the glucose grade pa standard (Graph 2).

Using the glucose standard curve, as shown in Table 2, the percentages of total glucose corresponding to the hydrolyzed samples (using the enzymes amylase, Ban 480L, and Dextrozyme DX 1.5X) from the different starches extracted from fresh and dehydrated tubers can be studied.

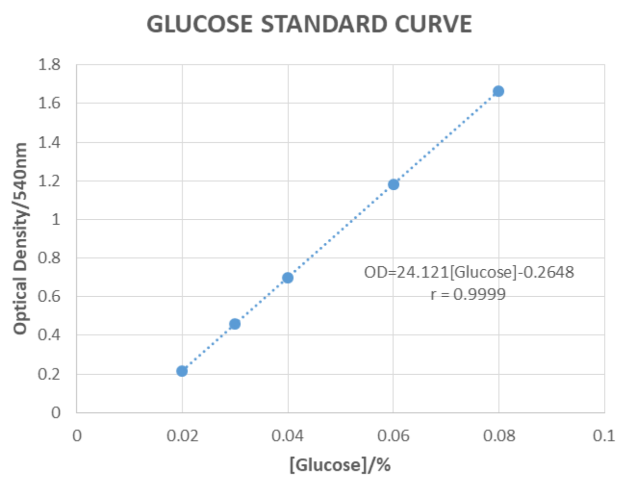
Within Groups 1 and 2, the hydrolysate of tunta starch and oca starch exhibited the highest percentage of total glucose with 92.8 and 96.6%, respectively, compared to the other samples of the same group. In Group 3, the papalisachuño starch hydrolysate presented a higher percentage of total glucose (81.4%) compared to the papalisa starch hydrolysate. In Group 4, the black Isaño starch hydrolysate presented the highest total glucose content (85.2%) in relation to the Isaño starch hydrolysate.

The results obtained in Groups 1, 2, and 3 are in agreement with those by Ochoa-Martínez et al. (2021) since amylose contains only one non-reducing end for the action of the enzyme; a high amylose content starch does not hydrolyze easily. For this reason, the tunta and oca starches within their respective



Source: Huanca López (2017).
Reference bar size, 50 µm.

Figure 2. Scanning electron microscope photomicrograph of starch granules at 500 ×.



Graph 2. Glucose standard curve obtained at 540 nm using the 3,5-Dinitrosalicylic acid spectrophotometric method.

groups reached high values of total glucose percentage in their hydrolysates since both samples have the lowest amylose content compared to the other two samples of their respective groups. Therefore, they have a higher amylopectin content, which in turn results in a greater number of branches and a greater number of reducing ends, which allow the enzymes to have greater points of attack on the substrate and, consequently, a greater degree of hydrolysis and a higher amount of glucose obtained. In Group 3, a difference of 1% was observed between the total glucose content values corresponding to the papalisa chuño starch hydrolysates (total glucose yield [%GT] = 81.4) and the papalisa starch ones (%GT = 80.4), a behavior that is similar to the amylose content, where very close results are also observed (%amylose = 15.8% papalisa starch; %amylose = 16.0% papalisa chuño starch). To explain this minimal difference between both, we analyzed our samples based on the size of starch granules since both starches show notable differences in this measured parameter, with papalisa chuño starch having a higher content of starch granules from 1 µm to 10 µm and papalisa starch having a higher percentage of starch granules > 20 µm. For this reason, the papalisa chuño starch hydrolysate shows a higher total glucose content. Due to the smaller size of the starch granule, the amylose content of the sample is lower and consequently, there are a greater amount of reducing ends and a higher content of glucose in the hydrolysis process (Cuba Canales, 2021; Naguleswaran et al., 2014; Ochoa-Martínez et al., 2021). Black Isaño starch has a higher total glucose content (85.2%) compared to Isaño starch hydrolysate (59.2%), results that show the existence of variations even in samples that are of the same species but a different variety.

3.6 Gel properties

The gel properties of starches extracted from fresh and dehydrated tubers are shown in Table 2.

The granules of the tunta (Group 1), oca (Group 2), papalisa (Group 3), and Isaño (Group 4) starches show lower gelatinization start temperatures in relation to the other samples that make up each group, which indicates that these starches require less energy to begin to lose their molecular arrangement. In this process, the hydrogen bonds of the amorphous region of the granule are broken, allowing water to associate with the free hydroxyl groups of water. This gives way to the gelatinization temperature, where the starch granules are completely hydrated (granule swelling) and have lost all their molecular order, culminating in their disintegration or rupture of the granule (Ochoa-Martínez et al., 2021). According to Ojeda (1998) and Robles Oñate (2012), the amylose content is directly related to the gelatinization temperature (Guzmán Valverde, 2024; Ojeda, 1998; Robles Oñate, 2012). This behavior is observed in the starches that make up Group 1, where the highest gelatinization temperature (78.9°C) and the lowest amylose content (19.2%) correspond to tunta starch, followed by potato starch (gelatinization temperature = 78.7°C, amylose content = 22.7%) and chuño starch that shows the lowest gelatinization temperature (69.1°C) and the highest amylose content (26.4%). In relation to Group 2, the same behavior is observed for black caya starch, which shows a higher amylose content of 20.9% and a lower gelatinization temperature of 61.9°C compared with the oca and white caya starches. However, the highest gelatinization temperature of 64.0°C (white caya starch) does not correspond to the starch with a lower amylose content of 19.2% (oca starch). Similar behaviors are observed in Groups 3 and 4, where higher gelatinization temperatures correspond to starches with a higher amylose content, which could mean that these starches have a higher degree of molecular organization present inside the granule. On the other hand, Salgado-Ordosgoitia et al. (2019) pointed out that the gelatinization temperature is characteristic of each type of starch. Moreover, it is influenced by some factors such as the relationship between amylose/amylopectin, pH, botanical origin of starch, and granule size (Luque Vilca et al., 2021; Salgado-Ordosgoitia et al., 2019). In our study, the behavior observed in Group 3 could be due to the fact that papalisachuño starch has a greater proportion of small granules (1 to 10 µm) (51.7%) and therefore better crystalline organization that requires the application of greater energy to break its structure. With respect to Group 4, it is evident that the gelatinization temperature is characteristic of each type of starch, given that Isaño and black Isaño starches, even though they are samples of the same species, present differences in the amylose content, granule size, and gelatinization temperature.

The maximum viscosity and decomposition viscosity of the starch samples in Group 1 are lower for tunta starch (2562 BU [Brabender units], 462 BU) compared to potato and chuño starches (3399 BU, 1049 BU; 2805 BU, 570 BU, respectively). In Group 2, white caya starch shows lower results for these parameters (2929 BU, 1219 BU) compared to the oca and black caya starches (3470 BU, 2035 BU; 3083 BU, 1748 BU, respectively). Among papalisa and papalisachuño starches (Group 3), the dehydrated one has lower values of maximum viscosity and decomposition viscosity (2303 BU, 673 BU, respectively). In Group 4, made up of samples of the same species but different varieties (Isaño and black Isaño), the maximum viscosity and decomposition viscosity of the starch samples are lower for black Isaño (3556 BU, 1715 BU, respectively) in relation to starch from Isaño.

In general, it is observed that in Groups 1, 2, and 3, the lowest values of maximum viscosity and decomposition viscosity correspond to the starches extracted from the dehydrated samples, which indicates that in this type of samples, the amylose begins to leak from the amorphous regions when the temperature reaches the gelatinization temperature (Li et al., 2022).

The recoil viscosity results from Group 1 to Group 3 are higher for the samples of starches extracted from fresh tubers (1116 BU−−potato starch; 507 BU−−oca starch; and 956 BU−−papalisa starch), compared to Group 4. Isaño starch has a higher recoil viscosity value (826 BU) compared to black Isaño starch (809 BU). The results of this measured parameter indicate that samples that have a higher value of recoil viscosity have a greater capacity for re-association of amylose and amylopectin molecules, which results in a stronger gel network.

3.7 Crystal structure

XRD was used to analyze the crystalline structure of starch, demonstrating that the analyzed samples present characteristic diffraction peaks at $2\theta = 5°, 15°, 17°, 22°$, and $24°$, values assigned to a type B crystallinity pattern typical of tubers. According to Chen et al. (2021) and Lin et al. (2022), indeed, the XRD pattern of type B crystalline starch has the strongest diffraction peak around $2\theta = 17.00°$, and some peaks around $2\theta = 5.6°$; 15.00°, 20.00°, 22.00°, and 24.00 (Chen et al., 2021; Li et al., 2019; Lin et al., 2022).

4 CONCLUSION

The physicochemical characterization of the starch samples corresponding to the fresh and dehydrated tubers was carried out, the microscopic structures were studied, and these parameters were related to the percentage of glucose present in the hydrolysates from the starch. The properties of the gel, such as maximum viscosity, breakdown, and setback, were also studied; these characteristics are important to provide alternative uses, such as the application of starch in the production of various food products. Regarding the XRD diffractogram, all samples presented characteristic peaks corresponding to type B crystalline structures typical of tubers.

Therefore, with the results of the different analyses carried out and on evaluating the observed behaviors, it is concluded that there is a relationship between the morphology of the starch granules and the percentage of amylose/amylopectin, the size of the granule, as well as the enzymatic hydrolysis process. Furthermore, it was observed that the starch yield and the amylose/amylopectin ratio vary between species and even between varieties of the same species, with the latter being an influential factor in the composition, structure, gel properties, and crystallinity of the starch granule.

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