



Characterization, mathematical modeling of moisture sorption isotherms and bioactive compounds of Andean root flours

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ABSTRACT

Andean roots can be used as an alternative to gluten-free food. The objective of this study was to enhance the technological and nutritional properties of Andean root flours to promote their industrial applicability. The water content and activity of the flour were lower than those required to prevent mold growth. The bulk density of the flour was comparable to that of wheat flour. The flour of *Ipomoea batatas* (L.) Lam. exhibited the lowest water absorption capacity of the tested samples. However, both this flour and *Tropaeolum tuberosum* Ruiz & Pavón showed a higher fat absorption capacity. The samples exhibited type-II isotherms, indicating that the flours were highly hygroscopic. The Guggenheim, Anderson, and de Boer GAB model showed a higher coefficient of determination in mathematical modeling. The chroma of *T. tuberosum* Ruiz & Pavón flour was higher than the other samples, which was related to total carotenoids and lycopene. Furthermore, *I. batatas* (L.) Lam. exhibited the highest phenol value.

1. Introduction

Celiac disease is an immune-mediated enteropathy that causes inflammation of the small intestinal mucosa after gluten ingestion (Oxentenko and Rubio-Tapia, 2019). Individuals with celiac disease develop antibodies against gliadin, the major component of wheat gluten, and in an autoimmune response, generate antibodies against the host tissue transglutaminase (de Lorgeril and Salen, 2014). Individuals genetically susceptible to celiac disease experience chronic diarrhea after consuming gluten. In the medium term, this disease can cause weight loss, anemia, osteoporosis, and neurological disturbances (Caio et al., 2019). Celiac disease is a common lifelong disorder, affecting approximately 0.5%–1% of the general population worldwide (Cab-anillas, 2020). Prevalence rates vary between countries, with higher rates observed in younger and older individuals from developed countries (Mäki et al., 2003; Vilppula et al., 2009). However, this disease appears to be less prevalent in sub-Saharan Africa and is rare among African Americans (Ludvigsson and Murray, 2019). Some studies have suggested that the prevalence and incidence of celiac disease may be

influenced by the host's genetic makeup and the consumption of considerably quantities of gluten (up to 20 g/day) (de Lorgeril and Salen, 2014; Gatti et al., 2020). The gluten protein portion primarily comprises gliadin (67%) and glutenin (33%) (Lexhaller et al., 2017; Wieser et al., 2023). Glutenin provides strength and elasticity to dough, whereas gliadin is responsible for causing intolerance. This presents a challenge for the food industry, as gluten-free foods must be developed. However, the absence of gluten affects the crust, crumbs, and other parameters of baked products (Matos and Rosell, 2015).

An alternative to preparing gluten-free foods is to use Andean roots (Fig. 1). These crops contain significant dietary fiber and resistant starch (Fonseca-Santanilla and Betancourt-López, 2022; Jimenez et al., 2015), which are crucial characteristics of gluten substitutes. Furthermore, these roots contain a variety of vitamins (A, C, B, E, and K), minerals (Ca, Mg, K, P, and Na), and bioactive compounds such as phenolic acids, carotenoids, saponins, glycoalkaloids, and anthocyanins (Chandrasekara & Josheph Kumar, 2016; Saranraj et al., 2019). The use of root flours can result in functional foods that offer health benefits beyond basic nutrition (Leidi et al., 2018). These flours have been used to create

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various gluten-free products, including bread (Espino-Manzano et al., 2015), bread made with hydrocolloids and enzymes (Calle et al., 2020), muffins (Salazar et al., 2021), spaghetti (Giri and Sakhale, 2022), milk pudding (Hendek Ertop, Atasoy and Akin, 2019), tomato ketchup (Sit et al., 2014), second- (Acurio et al., 2023), and third-generation snacks (Acurio et al., 2023). However, Andean roots play an essential role in the diet of the residents of the area, as they are an excellent source of energy. Despite this, their use at an industrial level is still limited due to a lack of knowledge regarding their outstanding technological and nutritional properties. These flours offer the opportunity to develop many gluten-free products with technological and nutritional characteristics equal or better than those made with wheat.

Flour is a key ingredient in baked goods and pasta, making it an essential food worldwide, and crucial for improving food and nutritional security. One of its advantages is its low water activity and extended shelf life. A study was conducted on the drying processes of various Andean roots. According to the authors, these flours should be dried until the humidity level is below $10 \text{ g}_{\text{water}}/\text{g}_{\text{drymass}}$ mass to maintain their initial characteristics (Acurio et al., 2023).

The purpose of this study was to investigate the technological and nutritional properties of underutilized root crops for potential use in food processing. The properties of these crops depend on genetic and environmental variations; however, the results will provide baseline information for worldwide use. Specifically, this work aims to elaborate flours of sweet purple potato (*Ipomoea batatas* (L.) Lam.). This study analyzed the influence of roots on various properties of four Andean tubers: potato (*Solanum tuberosum* L.), ulluco (*Ullucus tuberosus* Caldas), mashua (*Tropaeolum tuberosum* Ruiz & Pavón), and three varieties of oca (*Oxalis tuberosa* Molina) (white, yellow, and red) (2). The properties analyzed included the water content, water activity, particle size distribution, bulk density, tap density, Hausner ratio, Carr index, hygroscopicity, water and fat absorption index, water solubility index, optical properties, and bioactive compounds. This project aims to showcase the exceptional technological and nutritional properties of these flours, positioning them as viable raw materials for the production of various processed foods. Furthermore, these findings are expected to benefit global growers of the analyzed root and tuber crops.

2. Materials and methods

2.1. Andean roots

The study purchased the sweet purple potato (*Ipomoea batatas* (L.) Lam.), mashua (*Tropaeolum tuberosum* Ruiz & Pavón), and three varieties of oca (*Oxalis tuberosa* Molina) (white, yellow, and red) from a local market in Ambato, Ecuador.

2.2. Flour manufacturing

The roots were washed, peeled, and sliced into 2 ± 0.1 mm thick pieces. The slices were microwaved (750 W/20 s) and soaked in cold water (4 ± 1 °C) for 20 s to prevent enzymatic browning. The drying process was performed in a convective dehydrator (Gander mtn, CD 160, Saint Paul, MN, USA; air velocity 2 ms^{-1} , metallic mesh 450×450 mm) at 65 °C for 8 h. The dehydrated slices were ground at three 10-s intervals using an electric mill (Hamilton Beach, model: 80,393, Picton, Canada) (Acurio et al., 2023). The resulting flour was stored in individual aluminized bags.

2.3. Root characterization

2.3.1. pH and brix

Approximately 10 g of freshly peeled slices was washed with distilled water and homogenized using a Thermomix (TM 21, Vorwerk, Valencia, Spain) for 30 s at 5200 rpm. Homogenized samples were used to measure the pH, degree Brix, and water activity. The pH was determined using a Basic 20 pH meter (Crison, Spain) and the degree of Brix was measured using a refractometer (Abbat 200, Anton Paar, Austria) at 20 °C. All measurements were performed in triplicate.

2.3.2. Water content (x_w) and water activity (a_w)

Water content (x_w) was determined using fresh peeled slices (2 ± 0.1 mm thickness) and a Vacuotem vacuum oven (J.P. Selecta, Barcelona, Spain) at 103 °C for 48 h. Water activity (a_w) was measured using an AquaLab PRE hygrometer (Decagon Devices Inc., Pullman, WA, USA). All measurements were performed in triplicate. The water content (x_w) and water activity (a_w) of the flours obtained by the procedure reported in Section 2.2 were also determined using these methodologies. Flour samples weighing approximately 1 g were used in this study.

2.4. Flour characterization

2.4.1. Particle size distribution

The distribution of flour particle sizes was determined in accordance with the ISO13320 standards (AENOR, 2009) using laser diffraction. Malvern Instruments Ltd. A Mastersizer 2000 particle size analyzer equipped with a dry sample dispersion unit (Malvern Instruments Ltd., Scirocco, 2000) was used. The results were analyzed using Mastersizer 2000 software (version 5.6) and expressed as the volume mean diameter (D [4,3]), as well as the standard percentiles d (0.1), d (0.5), and d (0.9) (García-Segovia et al., 2020).

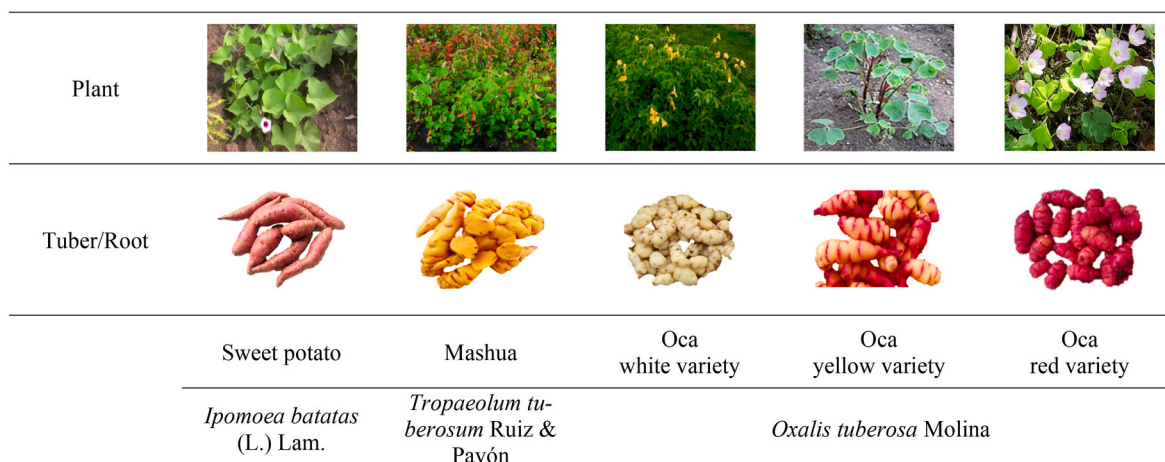


Fig. 1. Andean plants and roots.

2.4.2. Bulk density (ρ_b), tap density (ρ_t), Hausner Ratio (HR), and Carr index (CI)

The study utilized the methods proposed by Sotelo-Díaz et al. (Sotelo-Díaz et al., 2023) to determine the ρ_b , ρ_t , HR, and CI of flour. The bulk density (ρ_b) was calculated by dividing the mass of the flour, which was weighed using a precision balance (± 0.001 g) from Mettler Toledo, Greifensee, Switzerland, by the volume measured using a 10 mL test tube. The results were expressed as g/m^3 . The graduated test tube was mechanically tapped, and the tapped density (ρ_t) (g/cm^3) was calculated by dividing the flour mass by the occupied volume after tapping. The HR was calculated as the ratio of tap density to bulk density (Equation (1)). The CI was determined using bulk and tap density values according to the Carr method (Carr, 1965). The CI (%) was calculated using Equation (2). The reported results represent the average of three measurements.

$$\text{HR} = \frac{\rho_t}{\rho_b} \quad 1$$

$$\text{CI} = 100 \times \frac{\rho_t - \rho_b}{\rho_t} \quad 2$$

2.4.3. Hygroscopicity (Hy)

Hygroscopicity (Hy) was determined using the Cai and Corke method (Cai and Corke, 2000). The flour samples were placed in a Petri dish in a desiccator with a saturated Na_2SO_4 solution (81% relative humidity at 25 °C). After 24, 96, and 168 h, each sample was weighed using a precision balance (± 0.001 g) (Mettler Toledo, Greifensee, Switzerland). The Hy values were expressed in grams of water per 100 g of dry solids.

2.4.4. Water absorption index (WAI), fat absorption index (FAI), and water solubility index (WSI)

The WAI and WSI were determined using the methods proposed by Singh and Smith (Singh and Smith, 1997) and Uribe-Wandurraga et al. (Uribe-Wandurraga et al., 2020), respectively. To prepare the samples, flour (2.5 g) was mixed with 25 g of distilled water and stirred for 30 min using a magnetic stirrer. The mixture was then placed in centrifuge tubes (50 mL) and centrifuged at 3000 rpm for 10 min. The weights of the sediment and dissolved solids in the supernatant were measured to determine the WAI and WSI using Equations (3) and (4), respectively.

$$\text{WAI} = \frac{\text{weight of sediment}}{\text{weight of dry solids}} \quad 3$$

$$\text{WSI} (\%) = \left(\frac{\text{weight of dissolved solids in supernatant}}{\text{weight of dry solids}} \right) \times 100 \quad 4$$

The FAI was determined using the method proposed by Navarro-González, García-Valverde, García-Alonso, and Periago (Navarro-González et al., 2011), with some modifications (Noguerol et al., 2022). The flour sample (4 g) was placed in a centrifuge tube and 24 g of sunflower oil was added. The mixture was stirred at 3000 rpm for 30 s every 5 min until 30 min elapsed. After stirring, the samples were centrifuged at $1600 \times g$ for 25 min and the free oil was decanted. The FAI values were expressed as $\text{g}_{\text{oil}}/\text{g}_{\text{sample}}$.

2.4.5. Determination of moisture sorption isotherms and isosteric heat of sorption

The equilibrium moisture content was determined using a gravimetric method. This method uses the saturated salt solutions listed in Table 1 (Greenspan, 1977). The saline solutions used were of reagent grade, and the preparation technique of W. Spiess and Wolf (Spiess and Wolf, 2017) were used in this study. Thymol was added to $(\text{NH}_4)_2\text{SO}_4$ solution to inhibit microbial growth. The samples were weighed regularly until a constant weight (± 0.0005 g) was achieved using a precision analytical balance (Mettler Toledo, Greifensee, Switzerland). When the samples reached a stable weight, their moisture content was considered

Table 1

Saturated salt solutions were used to determine the moisture sorption isotherms.

Name	Nomenclature	a_w^a
Lithium chloride	LiCl	0.1178
Potassium acetate	$\text{CH}_3\text{CO}_2\text{K}$	0.2982
Magnesium chloride	MgCl_2	0.3425
Sodium bromide	NaBr	0.5732
Ammonium sulfate	$(\text{NH}_4)_2\text{SO}_4$	0.8012

^a Values were determined using an AquaLab 4 TE water activity meter (Decagon Devices Inc., Pullman, WA, USA).

to have reached equilibrium (approximately 4 weeks).

The experiment was conducted at temperatures of 20 °C, 30 °C, and 40 °C (± 1 °C), which were selected based on the average ambient temperature in Ecuador's three most critical climatic zones. Furthermore, testing at 40 °C is necessary because high temperatures can cause irreversible quality changes (Saleh et al., 2018).

The data obtained by the gravimetric method were fitted to the Brunauer, Emmett, and Teller (BET) and Guggenheim, Anderson, and de Boer (GAB) models (Table 2).

The net isosteric heat of sorption (q_s) was determined using Equation (7), as proposed by Clausius–Clapeyron (Tsami, 1991). The value of q_s was calculated from the slope of the plot between $\ln(a_w)$ and $1/T$ at constant moisture.

$$d(\ln a_w) = -\frac{q_s}{R} d\left(\frac{1}{T}\right) \quad 7$$

Where, a_w is the water activity, R is the ideal gas constant (8.314 J/mol K), and T is the absolute temperature (K).

2.4.6. Optical properties

The optical properties of the flour were measured using a Minolta spectrophotometer (CM-3600 d, Tokyo, Japan) with D65 standard light and observer 10 standard. The results were expressed in $\text{CIE}^*L^*a^*b^*$ color coordinates (spectra 400–700 nm), and the chroma (C^*) and hue (h^*) were also analyzed.

2.4.7. Bioactive compounds

The total carotenoids (TC) were quantified using the AOAC spectrophotometric method (AOAC, 2000) and expressed as mg of β -carotene per 100 g of sample (Olives Barba et al., 2006). Lycopene (LP) was determined from the TC extract at 503 nm using the Khamis et al. (Khamis et al., 2017) method and expressed as mg/100 g of the sample.

Total phenols (TP) were measured at 765 nm using a UV-3100 PC (VWR, Radnor, Philadelphia, PA, USA). The results were expressed as mg gallic acid (GA) per 100 g of dry solids (Iguar et al., 2016). Antioxidant capacity (AC) was determined using the DPPH method at 515 nm (Iguar et al., 2016). The AC results are expressed as milligrams of Trolox equivalents (TE) per 100 g of dry solid sample. The study calculated an average of three measurements for each bioactive parameter.

2.5. Statistical analysis

Variance (ANOVA) and Tukey's multi-range test were performed using Statgraphics Centurion XVII software (Statgraphics Technologies, Inc., The Plains, VA, USA - version 17.2.04) to evaluate differences among roots. The coefficient of determination (r^2), root-mean-square error (RMSE), and mean relative percentage deviation (MRPD) were used to evaluate the goodness of fit in the modeling of the sorption isotherms. Pearson correlation analysis was conducted between the parameters at a 95% significance level.

Table 2
Equations used for modeling sorption isotherms.

Model	Equation	Equation	References
Brunauer, Emmett, and Teller (BET)	$X_e = \frac{X_0 \times C \times a_w}{(1 - a_w) \times (1 + (C - 1) \times a_w)}$	5	Stephen Brunauer, Emmett, and Teller (1938)
Guggenheim, Anderson, and de Boer (GAB)	$X_e = \frac{X_0 \times C \times K \times a_w}{(1 - (K \times a_w)) \times (1 + (C - 1) \times (K \times a_w))}$	6	Van den Berg and Bruin (1978)

X_e is the equilibrium moisture content ($\text{g}_{\text{water}}/\text{g}_{\text{dry mass}}$), X_0 is the monolayer moisture content ($\text{g}_{\text{water}}/\text{g}_{\text{dry mass}}$), C is an empirical constant (dimensionless) for the BET and GAB equations, and K is the second empirical constant (dimensionless) for the GAB equation.

3. Results and discussion

3.1. Root characterization

Based on the pH values, these roots can be classified as neutral foods. IbP and Tt had lower pH values ($p < 0.05$) than other roots (Table 3). The roots of IbP, OtW, OtY, and OtR had °Brix values ranging from 11 to 13.3, with no significant differences ($p > 0.05$), whereas Tt had a lower value (7.7 ± 0.6). According to various studies, the Ib tuber contains 15%–35%, Tt contains 21.7% soluble sugar, and Ot contains 18.9% soluble sugar (Pang et al., 2021; Valcárcel-Yamani et al., 2013). These high percentages give the roots a sweet taste. The x_w value of Tt was higher than the other samples, whereas IbP had the lowest value. The x_w values of the three Ot varieties were significantly similar ($p > 0.05$). All samples had similar a_w values ($p > 0.05$). Tt has the highest a_w value, which is related to its high water content.

Correlational statistical analyses were conducted to determine the influence of °Brix on x_w and a_w . In this study, a negative correlation was found between x_w and °Brix (-0.7358 , $p < 0.05$). The concentration of soluble solids, mainly sucrose and inverted sucrose, such as glucose and fructose, has been shown in various studies to decrease x_w because of the bonds these compounds form with water. This is easily verified by Raoult's law of mole fraction (Fennema, 1996; Gabriel, 2008).

3.2. Flour characterization

3.2.1. Water content and water activity

Table 4 shows that the flour had x_w values ranging from 0.044 to 0.114 $\text{g}_{\text{water}}/\text{g}_{\text{dry mass}}$. The drying process reduced the humidity of most samples to below 0.08 $\text{g}_{\text{water}}/\text{g}_{\text{dry mass}}$, except for Tt, which had the highest value (0.114 $\text{g}_{\text{water}}/\text{g}_{\text{dry mass}}$). Although the moisture content of this sample is high, it is still below the threshold of a 0.12 $\text{g}_{\text{water}}/\text{g}_{\text{dry mass}}$, where mold growth is typically observed in cereal flours (N. Abdullah et al., 2000; Awol et al., 2024). According to various studies, it is recommended to prevent the amount of water in the flour from exceeding 0.19 $\text{g}_{\text{water}}/\text{g}_{\text{dry mass}}$. This is because it can cause superior particle cohesion, carotenoid degradation (due to lipoxidase activity), and an

Table 3

Mean values (standard deviations) of fresh roots: pH, Brix, water content (x_w), and water activity (a_w).

Sample	pH	Brix	x_w ($\text{g}_{\text{water}}/\text{g}_{\text{dry mass}}$)	a_w
IbP	6.39 (0.06) b	11.7 (0.6) a	2.0 (0.3) c	0.983 (0.003) ab
Tt	6.65 (0.03) b	7.7 (0.6) b	5.4 (0.2) a	0.989 (0.005) a
OtW	7.25 (0.14) a	11.0 (1.7) a	2.40 (0.09) bc	0.9836 (0.0006) ab
OtY	7.2 (0.4) a	13.3 (0.6) a	2.8 (0.3) b	0.984 (0.002) ab
OtR	6.1 (0.3) ab	12.3 (1.2) a	2.9 (0.1) b	0.977 (0.003) b

Different lowercase letters represent significant differences ($p < 0.05$) by samples (IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety).

Table 4

Mean values (standard deviations) of flour water content (x_w) and water activity (a_w).

Sample	x_w ($\text{g}_{\text{water}}/\text{g}_{\text{dry mass}}$)	a_w
IbP	0.0589 (0.0012) c	0.397 (0.003) c
Tt	0.114 (0.003) a	0.454 (0.009) a
OtW	0.062 (0.002) c	0.3467 (0.0012) d
OtY	0.0735 (0.0013) b	0.407 (0.003) c
OtR	0.0437 (0.0006) d	0.433 (0.005) b

Different lowercase letters represent significant differences ($p < 0.05$) by samples (IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety).

increase in fat acidity, resulting in dough with reduced strength and increased plasticity (Doblado-Maldonado et al., 2012; Guan and Zhang, 2009; Lancelot et al., 2021). Table 4 shows that the water activity values ranged from 0.347 to 0.454, with Tt having the highest value. However, all flours had values below 0.62 to 0.7, which is considered the limit to prevent fungal growth (Hill and Lacey, 1983).

3.2.2. Particle size distribution

Fig. 2 shows the volume particle size distributions of the samples. Most particles in the Tt sample fell within the range of 100–2500 μm , accounting for 8.5% of the volume. Similarly, the OtY sample exhibited this trend between 100 and 2200 μm , representing 6.2% of the volume. There were no significant differences in the particle size distributions of OtW, OtR, and IbP in the range of 200–2200 μm , accounting for 5.5% of the volume. The particle size range of wheat flour was mostly between 40 and 500 μm (Ma et al., 2021), whereas hard wheat flour had a range of 51–300 μm (W. Kim et al., 2004). These findings follow those of previous studies. For example, potato powder showed a pronounced bell between 900 and 2000 μm (Hu et al., 2020), cassava (*Manihot esculenta* Crantz) fermented mash flour between 710 and 1000 μm (Nwaiwu and Onyeaka, 2022), and dried *Dioscorea hispida* tuber fiber between 100 and 2000 μm (Hazrati et al., 2021). The OtW and OtR samples also exhibited a slight bell curve between 10 μm and 100 μm , representing 3% of the volume. Similarly, the OtY samples showed this trend in the

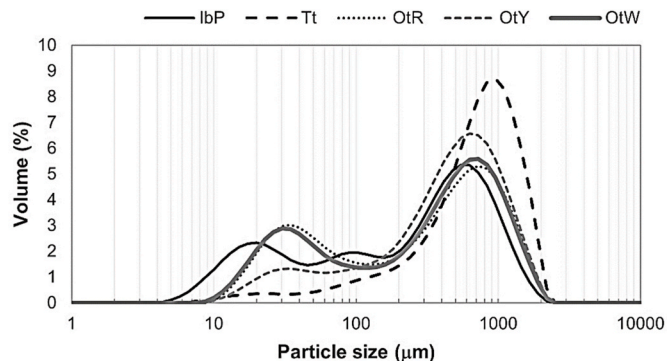


Fig. 2. Volume particle size distributions (representative curves). Samples, IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety.

range of 10–60 μm , representing 1.5% of the volume. In contrast, the IbP sample displays two bell curves, one between 5 and 40 μm and the other between 60 and 160 μm .

Table 5 lists the values obtained for the mean volume diameter [D (4,3)]. This parameter represents the diameter of the sphere of the volume equivalent to the flour particles formed after the grinding process (Guo et al., 2021). Tt had the highest volume particles (723 \pm 23 μm), which was significantly different ($p < 0.05$) from the other samples. IbP had the lowest volume (364 \pm 27 μm), which was also significantly different ($p < 0.05$) from that of the other samples. The three varieties of Ot exhibited significant differences ($p < 0.05$). OtY had a higher value (509 \pm 52 μm), whereas OtW and OtR showed no significant difference ($p < 0.05$) between them (434 \pm 22 μm and 436 \pm 28 μm , respectively).

Table 5 also shows the particle size distributions at the standard percentiles of d (0.1), d (0.5), and d (0.9). The particles were in the range of 15.6–1353 μm . The d (0.5) percentile represents the data median, which is the central value when all the data are ordered from smallest to largest. The highest median was observed in Tt flour (680 \pm 28 μm) and the lowest in IbP flour (270 \pm 27 μm), with a significant difference ($p < 0.05$) between them. The Tt sample showed that 90% of the particles [d (0.9)] had a size of less than or equal to 1353 \pm 28 μm , which was significantly different ($p < 0.05$) from IbP, which showed a percentile value of 881 \pm 68 μm . The Ot samples showed no significant difference ($p < 0.05$) in this percentile. These results are consistent with those reported for maize semolina (approximately 900 μm) (Benković et al., 2013) and durum wheat semolina (800–1000 μm) (Petitot et al., 2010).

An inverse relationship exists between particle size and the amount of starch in the roots. For example, IbP root had 12.7%_{w.b.} Starch (Acurio et al., 2023), and this flour had a d (0.9) value of 881 \pm 68 μm , whereas Tt root sample had 7.1%_{w.b.} Starch (Acurio et al., 2023), and this flour had a d (0.9) value of 1353 \pm 28 μm . This correlation was also observed in soft wheat flour ($r = -0.90$) (Siliveru et al., 2017).

3.2.3. Bulk density, tap density, hausner ratio, and Carr index

Bulk density (ρ_b) is related to the relative volume of the packaging flour and its porosity (Ngoma et al., 2019). The flours presented ρ_b values ranging from 0.57 to 0.67 g/cm^3 (Table 6), and there were no significant differences ($p > 0.05$) among the samples. These values are similar to those reported for hard and soft wheat flours (0.658 and 0.553 g/cm^3 , respectively) (Bian et al., 2015). Comparable values for flours made with the roots under study have been reported in different studies. For example, IbP was reported to have a bulk density of 0.46–0.48 g/cm^3 in flour made from steamed sweet potato (Patria et al., 2013) and between 0.52 and 0.55 g/cm^3 in sweet potato flour, whose pieces were pretreated with hot water, steam, and grill and dried at 65 $^\circ\text{C}$ for 48 h (Akinjide Olubunmi et al., 2017). Furthermore, Ot and Tt reported bulk densities of 0.71 \pm 0.01 and 0.65 \pm 0.01 g/cm^3 , respectively (Salazar et al., 2021).

The tap density (ρ_t) is related to flour compaction during transportation and handling (Bian et al., 2015). The flour presented ρ_t values

Table 5

Mean values (standard deviations) of volume mean diameter D (4,3), standard percentiles d (0.1), d (0.5), and d (0.9).

Sample	D (4,3) (μm)	d (0.1) (μm)	d (0.5) (μm)	d (0.9) (μm)
IbP	364 (27) ^d	15.6 (0.4) ^d	270 (27) ^d	881 (68) ^c
Tt	723 (23) ^a	138 (8) ^a	680 (28) ^a	1353 (28) ^a
OtW	434 (22) ^c	24.5 (0.7) ^c	340 (32) ^c	1026 (39) ^b
OtY	509 (52) ^b	46 (11) ^b	433 (63) ^b	1076 (68) ^b
OtR	436 (28) ^c	25.6 (0.7) ^c	324 (39) ^c	1056 (51) ^b

Different lowercase letters represent significant differences ($p < 0.05$) by samples (IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety).

Table 6

Mean values (standard deviations) of flour: bulk density (ρ_b), tap density (ρ_t), Hausner ratio (HR), and Carr index (CI).

Sample	ρ_b (g/cm^3)	ρ_t (g/cm^3)	HR	CI (%)
IbP	0.596 (0.013) ^a	0.718 (0.012) ^b	1.206 (0.012) ^{ab}	17.08 (0.72) ^{ab}
Tt	0.57 (0.05) ^a	0.65 (0.06) ^b	1.148 (0.009) ^b	12.92 (0.72) ^b
OtW	0.67 (0.05) ^a	0.83 (0.05) ^a	1.24 (0.02) ^a	19.17 (1.44) ^a
OtY	0.66 (0.05) ^a	0.76 (0.04) ^{ab}	1.15 (0.04) ^b	13.33 (2.89) ^b
OtR	0.67 (0.02) ^a	0.83 (0.02) ^a	1.24 (0.02) ^a	19.17 (1.44) ^a

Different lowercase letters represent significant differences ($p < 0.05$) by samples (IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety).

ranging from 0.65 to 0.83 g/cm^3 (see Table 6), with significant differences ($p < 0.05$) among samples. IbP, Tt, and OtY had the lowest values. ρ_t was higher than the bulk density due to particle friction during tapping, which caused particle rearrangement and reduced trapped air (E. C. Abdullah and Geldart, 1999). These values are similar to those reported for hard and soft wheat flours (0.718 and 0.677 g/cm^3 , respectively) (Bian et al., 2015). IbP has been reported to have a ρ_t between 0.68 and 0.72 g/cm^3 (Patria et al., 2013).

The HR is also an indicator of food powder compaction due to vibration during handling (Bian et al., 2015). The samples presented HR values ranging from 1.24 to 1.15, with significant differences ($p < 0.05$) between them. A ratio of less than 1.25 classifies this powder as free-flowing (Senden and Verkooijen, 1983). OtW and OtR presented higher values (1.24 \pm 0.02). These values are comparable to those reported for hard and soft wheat flours (1.09 and 1.22, respectively) (Bian et al., 2015).

The CI is an important factor that affects the characteristics of foods made with flours and the design of processing machines (Khan and Saini, 2016; Meng and Kim, 2020). The CI values for OtW and OtR were higher ($p < 0.05$) at 19.17% \pm 1.44%, which is similar to the values reported for hard and soft wheat flour (20% and 21%, respectively) (Blancher et al., 2005).

Table 6 shows the direct relationship between ρ_t , HR, and CI. Flours with high ρ_t values (OtW and OtR) also had high HR and CI values, and vice versa. Table 5 indicates an inverse relationship between the specific volume of the particles [D (4,3)]. Because the particles have a lower specific volume, there is more space between them (air), resulting in greater reorganization when tapping is generated. This led to a higher tap density.

Correlational statistical analyses were conducted to investigate the impact of water content (x_w) on the bulk density (ρ_b), tap density (ρ_t), and CI. The results showed that x_w was negatively correlated with ρ_t (-0.853 , $p < 0.05$) and CI (-0.6544 , $p < 0.05$). The high water content and heterogeneity in particle size likely resulted in reduced interparticle voids, increased aggregation, and, ultimately, lower CI (Maaroufi et al., 2000). This effect has also been demonstrated in roasted Bengal gram (Cicer arietinum) flour (R. K. Raigar and Mishra, 2015) and potato flour (Rakesh Kumar Raigar and Mishra, 2017).

3.2.4. Hygroscopicity

Fig. 3 shows the trend of the hygroscopicity data over time (24, 96, and 168 h). The Ot samples (OtW, OtY, and OtR) exhibited a similar trend, whereas IbP and Tt exhibited different behaviors from the rest of the samples. Between 24 h and 96 h, hygroscopicity increased considerably in Tt, OtW, OtY, and OtR, with an average of approximately 3.6 g water/100 g dry mass. In IbP, the increase was less significant during this period (1.8 g water/100 g dry mass). During the second period (96–168 h), there was a smaller increase in all samples, averaging 1.9 g water/100 g dry mass.

Studies have shown that products with smaller particle sizes are usually more hygroscopic because of their increased surface area,

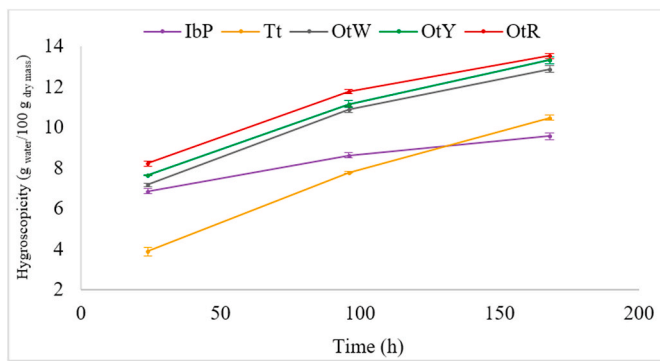


Fig. 3. Hygroscopicity of each flour sample regarding assay time. Samples, IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety.

exposing hydrophilic groups to form covalent or hydrogen bonds with environmental water (Khushbu et al., 2020; Lucena de Araújo & da Silva Pena, 2020). This can explain why Tt was the most hygroscopic sample and IbP was the flour with the lowest capacity to absorb water from the environment (Figs. 2 and 3). In addition, this behavior depends on the grinding process because of damage to the starch granules and breaks in the molecular starch structure (Jung et al., 2018).

3.2.5. Water absorption index, fat absorption index, and water solubility index

WAI is a measure of the ability of flour to absorb water during food preparation (Osungbaro et al., 2010), which affects its sensory properties. The flours in Table 7 had WAI values ranging from 3.51 to 8.1, with significant differences ($p < 0.05$) among the samples. Wheat flour typically has WAI values between 2.12 (Tharise et al., 2014) and 4.89 (Khan and Saini, 2016), whereas composite flour made from cassava, rice, potato, soybean, and xanthan gum has values ranging from 2.36 to 2.63 (Tharise et al., 2014). The flours that were pretreated typically exhibited a higher water absorption capacity than the untreated samples. This is due to the exposure of polar or charged side chains. For instance, a study demonstrated that IbP pretreated with sodium metabisulfite and citric acid at varying concentrations exhibited a higher water absorption capacity than the control sample (Ngoma et al., 2019).

The fat absorption capacity (FAI) of the Tt and IbP samples was significantly higher than the Ot samples ($p < 0.05$). This means that when preparing fried products, these flours absorb more fat, which can affect the final product's characteristics. However, although the FAI values of Tt and IbP samples are high (0.86 and 0.85 g oil/g sample, respectively), they are still lower than those observed in a mixture of cassava, potato, and soybean flours (1.88–2.08 g oil/g sample) (Tharise et al., 2014).

The WSI was determined by the interaction between amorphous and crystalline flour particles. The flours in Table 7 had WSI values ranging from 17.7% to 28.9%, with significant differences ($p < 0.05$) among the

Table 7

Mean values (standard deviations) of flour: water absorption index (WAI), fat absorption index (FAI), and water solubility index (WSI).

Sample	WAI	FAI (g oil/g sample)	WSI (%)
IbP	3.51 (0.03) ^c	0.85 (0.07) ^a	17.68 (0.02) ^c
Tt	8.10 (0.45) ^a	0.86 (0.02) ^a	19.3 (1.2) ^c
OtW	3.54 (0.36) ^c	0.711 (0.002) ^b	27.16 (0.07) ^{ab}
OtY	4.94 (0.14) ^b	0.76 (0.03) ^{ab}	28.9 (0.5) ^a
OtR	3.97 (0.19) ^{bc}	0.72 (0.05) ^b	25.5 (0.4) ^b

Different lowercase letters represent significant differences ($p < 0.05$) by samples (IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety).

samples. High solubility indices are associated with high soluble sugar content and greater starch leaching during pretreatment and drying (Ojo et al., 2017). Furthermore, the value of this property depends on the type of pretreatment. For example, those subjected to grilling and steam showed higher values than those obtained by blanching with hot water or those not pretreated (Akinjide Olubunmi et al., 2017). The amount of starch present in the samples also plays a role; Ot contains $56.82 \pm 1.01\%$ starch, whereas Tt contains $41.35 \pm 1.60\%$, which is why Ot samples have higher WSI values. Furthermore, Tt starch is more easily digested than starch from Ot (Valcárcel-Yamani et al., 2013).

Correlational statistical analyses were conducted to investigate the impact of x_w , ρ_b , ρ_t , and CI on the WAI and WSI. The results showed a direct correlation between x_w and WAI (0.9274) and between a_w and WAI (0.7088) ($p < 0.05$). Furthermore, an inverse correlation was observed between x_w and ρ_t (-0.7228 , $p < 0.05$). Previous studies have shown a correlation between water solvent retention capacity and the overall absorption characteristics and quality of wheat flour (Kweon et al., 2011). WSI was directly related to ρ_b and ρ_t (0.6454 and 0.6417, respectively; $p < 0.05$). This effect was also observed in flours from different chickpea cultivars (Kaur and Singh, 2006), which may be attributed to their more significant hydrophobic constituents.

3.2.6. Moisture sorption isotherms and isosteric heat of sorption

The samples exhibited type-II isotherms, classified according to Brunauer (Fig. 4) (S. Brunauer et al., 1940). This isotherm is also sigmoidal because of the presence of an inflection point. Comparable results have been reported for various types of flour, including maize (Oyelade et al., 2008), cassava (Navia et al., 2011), durum wheat (Chuma et al., 2012), oat, and rice flours (Brett et al., 2009). The isotherm curve indicated that the matrix was highly hygroscopic. The capacity of flour for water molecule adsorption increases with higher relative humidity in the environment. This behavior suggests that the drying and grinding processes cause structural changes in the food matrix, leading to an increase in the active points for water adsorption (Martins Oyinloye and Byong Yoon, 2020). Both processes increase the contact surface, which contributes to an increase in hydroxyl groups present in starch or any other polar groups located on the surface (Alamri et al., 2018).

The BET model was adjusted to an a_w of 0.57, whereas the GAB model was adjusted within the evaluated range. The GAB model exhibited a higher r^2 value. Similar results were observed in sweet potato flour (*Ipomoea batata* L.) (Saavedra Layza, 2022), tapioca flour (a secondary product from cassava flour) (Chisté, Silva, Lopes, & da Silva Pena, 2012), and dehydrated bagasse yacon (*Smallanthus sonchifolius*) (Carvalho Lago and Noreña, 2015). The parameters of both models are listed in Table 8. The moisture of the monolayer (X_0) determines the bound humidity of food (Gutierrez Balarezo et al., 2019). The X_0 values obtained using the BET and GAB models were similar for all samples.

The C values range from 4.443 to 84.3. This constant, known as the sorption heat, is related to the active sites of the food matrix and the water molecules of the atmosphere. The lowest value was observed for the OtY sample with the BET model at 20 °C (4.44), and the highest value was observed at 30 °C (84.3). The obtained C values were comparable to those reported for whole wheat flour (13.59 at 20 ± 0.1 °C with the GAB model) (Martín-Santos, Vioque and Gómez, 2012), wheat flour (9.7 at 20 °C with the GAB model) (Moreira et al., 2010), and cassava starch (12.86 with the BET model and 19.93 with the GAB model at 30 °C) (Perdomo et al., 2009). The shape curve is also related to the C value, with a value > 2 indicating an inflection point. Therefore, the isotherm was classified as type II, and the food exhibited multilayer water adsorption capacity. The correction factor for the multilayer sorption constant (K) of the GAB model should be equal to or less than 1, as it represents the interaction of water molecules in the multilayer (Ceballos et al., 2009). The K values in this study ranged from 0.9 to 1.

The net isosteric heat of sorption (q_s) is defined as the energy required to remove water from the substrate relative to the energy

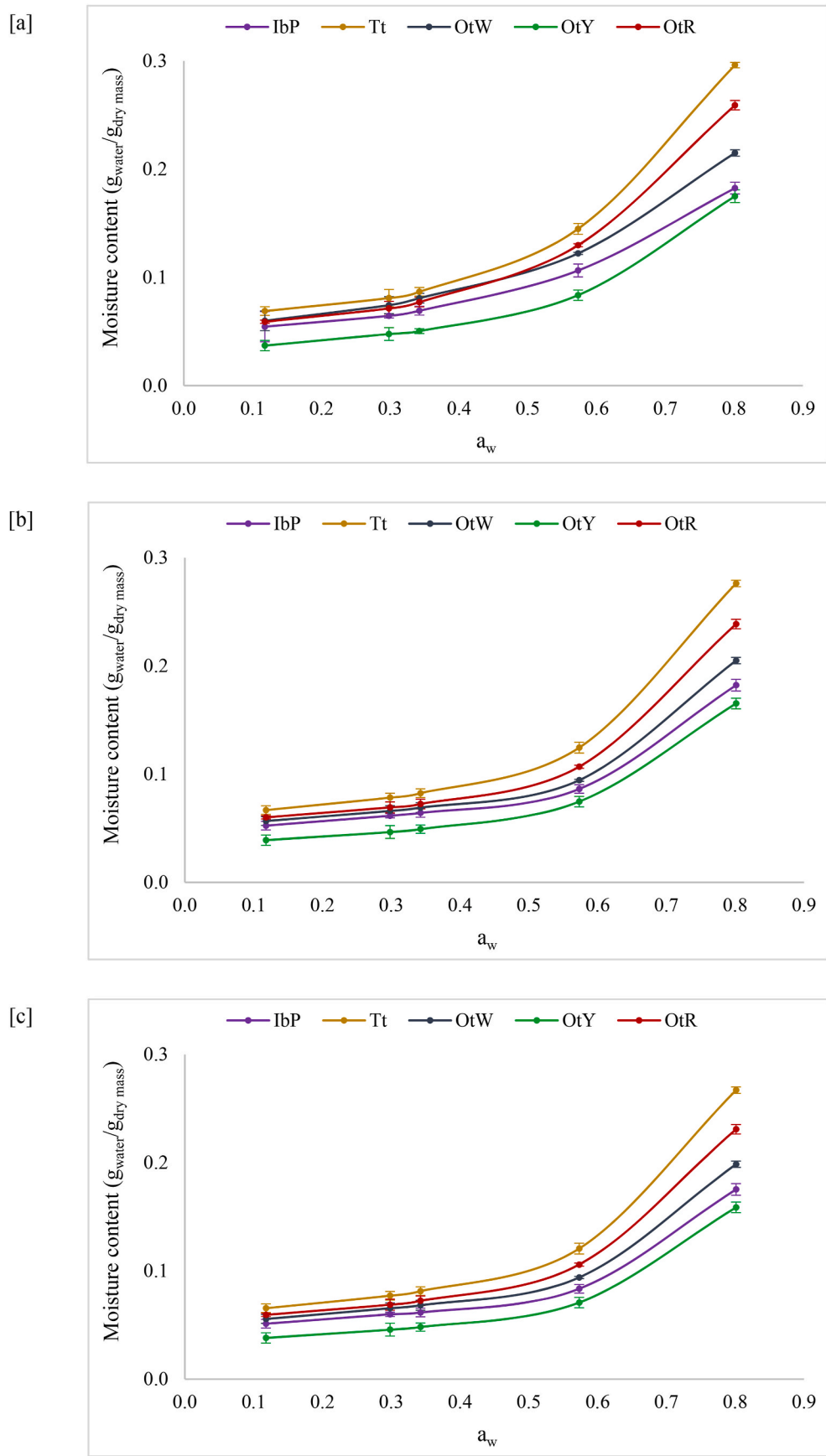


Fig. 4. Experimental moisture sorption isotherms at [a] 20 °C, [b] 30 °C, and [c] 40 °C (IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety).

Table 8
Parameters obtained in moisture sorption isotherm mathematical modeling.

Samples	Parameter	20 °C		30 °C		40 °C	
		BET	GAB	BET	GAB	BET	GAB
IbP	Model constants	X_0 : 0.04	X_0 : 0.05	X_0 : 0.04	X_0 : 0.04	X_0 : 0.04	X_0 : 0.04
		C: 17.88	C: 17.79	C: 19.43	C: 38.63	C: 18.81	C: 36.79
	Adj. r^2	0.98	0.99	0.987	0.998	0.997	0.998
	RMSE	0.02	0.03	0.013	0.005	0.02	0.005
	MRPD %	19.98	3.55	21.98	7	21.36	6.18
Tt	Model constants	X_0 : 0.07	X_0 : 0.07	X_0 : 0.05	X_0 : 0.05	X_0 : 0.05	X_0 : 0.05
		C: 16.4	C: 11.47	C: 64.11	C: 60.48	C: 50.81	C: 57.87
	Adj. r^2	0.99	0.99	0.999	0.998	0.999	0.999
	RMSE	0.004	0.005	0.02	0.02	0.01	0.009
	MRPD %	19.99	3.45	14.98	8.13	13.59	7.25
OtW	Model constants	X_0 : 0.05	X_0 : 0.06	X_0 : 0.04	X_0 : 0.04	X_0 : 0.04	X_0 : 0.04
		C: 12.5	C: 13.82	C: 25.05	C: 37.48	C: 22.2	C: 42.48
	Adj. r^2	0.98	0.98	0.998	0.98	0.998	0.998
	RMSE	0.003	0.005	0.01	0.02	0.01	0.01
	MRPD %	7.05	5.23	23.6	11.9	23.36	11.77
OtY	Model constants	X_0 : 0.05	X_0 : 0.05	X_0 : 0.03	X_0 : 0.03	X_0 : 0.03	X_0 : 0.03
		C: 4.44	C: 4.8	C: 84.3	C: 77.6	C: 48.32	C: 60.78
	Adj. r^2	0.98	0.97	0.99	0.99	0.999	0.999
	RMSE	0.02	0.003	0.008	0.007	0.006	0.004
	MRPD %	12.72	3.26	12.78	8.52	11.82	5.59
Otr	Model constants	X_0 : 0.06	X_0 : 0.06	X_0 : 0.05	X_0 : 0.05	X_0 : 0.05	X_0 : 0.05
		C: 21.89	C: 20.26	C: 38.51	C: 44.62	C: 38.06	C: 48.7
	Adj. r^2	0.99	0.998	0.999	0.998	0.998	0.998
	RMSE	0.01	0.007	0.008	0.01	0.01	0.01
	MRPD %	12.56	7.2	8.27	7.05	8.7	6.57

Different lowercase letters represent significant differences ($p < 0.05$) by samples (IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, Otr: oca red variety).

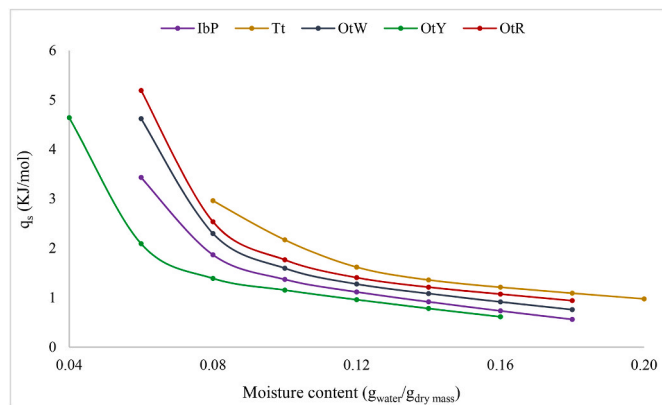


Fig. 5. Net isosteric heat of sorption for different moisture contents. Samples, IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, Otr: oca red variety.

Table 9
Mean values (standard deviations) of flour color coordinates (L^* , a^* , b^* , C^* , and h^*).

Sample	L^*	a^*	b^*	C^*	h^*
IbP	72.06 (0.03) ^c	6.581 (0.002) ^b	10.82 (0.09) ^d	12.66 (0.08) ^d	58.7 (0.2) ^e
Tt	65.96 (0.07) ^c	9.89 (0.06) ^a	38.8 (0.3) ^a	40.03 (0.25) ^a	75.70 (0.06) ^d
OtW	85.46 (0.11) ^a	-0.32 (0.02) ^d	18.84 (0.15) ^c	18.85 (0.15) ^c	90.97 (0.06) ^b
OtY	67.36 (0.02) ^d	3.708 (0.005) ^c	30.010 (0.014) ^b	30.238 (0.014) ^b	82.957 (0.009) ^c
Otr	78.95 (0.3) ^b	-0.655 (0.006) ^e	9.66 (0.14) ^e	9.68 (0.14) ^e	93.88 (0.02) ^a

Different lowercase letters represent significant differences ($p < 0.05$) by samples (IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, Otr: oca red variety).

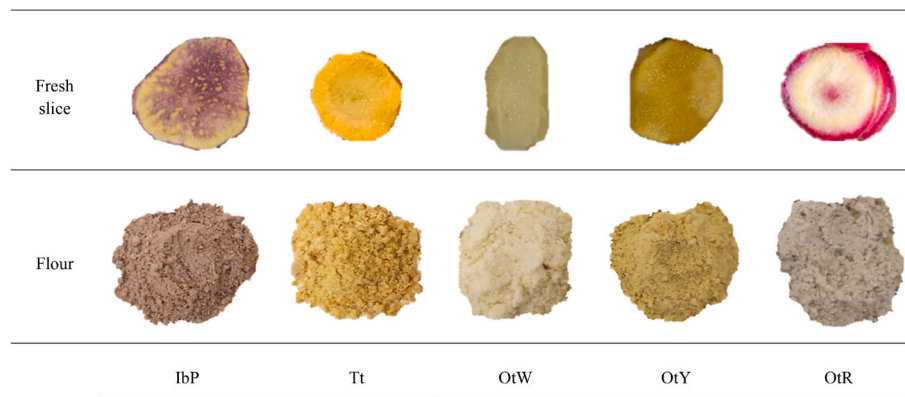


Fig. 6. Appearance of fresh root slices and flour. Samples, IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety.

Table 10

Total carotenoid (TC), lycopene (LP), total phenol (TP), and antioxidant capacity (AC) mean values (standard deviations).

Sample	TC (mg β carotene/ 100 g)	LP (mg/ 100 g)	TP (mgGA/ 100 g)	AC (mgTE/ 100 g)
IbP	2.60 (0.03) ^c	1.78 (0.04) ^c	761 (9) ^a	59.1 (0.8) ^b
Tt	34.82 (0.17) ^a	6.15 (0.02) ^a	564 (9) ^b	66.9 (0.7) ^a
OtW	2.80 (0.06) ^c	1.65 (0.04) ^d	201 (4) ^d	18.2 (0.3) ^d
OtY	10.98 (0.03) ^b	2.53 (0.02) ^b	264 (4) ^c	29.2 (1.2) ^c
OtR	2.66 (0.03) ^c	1.66 (0.03) ^d	197 (4) ^d	14.5 (1.7) ^c

Different lowercase letters represent significant differences ($p < 0.05$) by samples (IbP: purple sweet potato, Tt: mashua, OtW: oca white variety, OtY: oca yellow variety, OtR: oca red variety).

3.2.7. Optical properties

Table 9 shows the color coordinates (L^* , a^* , b^* , C^* y h^*). The OtW flour had higher luminosity values (85.46 ± 0.1) than the Tt flour which has lower values (65.96 ± 0.07). Wheat flour has L^* values of 95.02, whereas composite flours from cassava, rice, potato, soybean, and xanthan gum have values ranging from 95.71 to 97.1 (Tharise et al., 2014). The OtW and OtR flours were located in the second quadrant of the CIE $L^*a^*b^*$ space, resulting in negative a^* values. The chroma (C^*) value of the Tt flour was higher (40.03 ± 0.25) than the OtR sample, which had a lower C^* value (9.68 ± 0.14) ($p < 0.05$). Fig. 6 shows the intense yellow color of Tt flour, especially when compared to the OtR sample. The hue values (h^*) indicate the locations of the samples in the CIE $L^*a^*b^*$ space. The OtW and OtR samples had similar values because both flours were pale yellow. In contrast, the Tt and OtY samples had h^* values closer to the saturated yellow zone.

3.2.8. Bioactive compounds

Table 10 presents the results of the study. The Tt sample exhibited higher TC, LP, and AC values than those of the other samples ($p < 0.05$). Carotenoids have been extensively studied because of their antioxidant properties, particularly low-density lipoproteins (Kaczor et al., 2016). Furthermore, they are pigments that can be converted into vitamin A through metabolism, particularly when ingested through foods containing β -carotene (Meléndez-Martínez, 2019). The Tt flour exhibited a TC value of 34.82 ± 0.17 mg β -carotene/100 g, which is higher than the values reported in the literature. Previous research on 27 Peruvian mashua morphotypes showed that yellow varieties have values ranging from 2.02 to 7.15 mg/100g_{d.b.} (Jacobo-Velázquez et al., 2022). Castañeta, Miranda, Bascopé, and Peñarrieta (Castañeta et al., 2024) reported

value of 16.82 mg β -carotene/100g_{d.b.} for the same variety. The AC in this sample was influenced by the concentrations of vitamin C and glucosinolates, which confer antifungal, antibacterial, antioxidant, and anticarcinogenic properties (Vig et al., 2009). Moreover, glucosinolates are responsible for crop pest resistance (Grau et al., 2003).

IbP had the highest TP value and the lowest TC value compared with the other samples ($p < 0.05$). Studies have shown that the high phenolic content in these roots can be attributed to the presence of non-, mono-, and di-acylated anthocyanins. Di-acylated anthocyanins constitute at least 81.5% of the total anthocyanin content (H. W. Kim et al., 2012). Specifically identified phenols include di-acylated peonidin-3-(caffeoylferuloyl sophoroside)-5-glucoside, peonidin 3-(dicaffeoyl sophoroside)-5-glucoside, and peonidin 3-(caffeoyl-p-hydroxybenzoyl sophoroside)-5-glucoside (Gras et al., 2017; Lee et al., 2013).

The OtW and OtR samples exhibited the lowest values among the four analyzed compounds. In contrast, the OtY sample exhibited significantly higher values. This yellow variety contains more bioactive compounds, despite belonging to the same genus, Ot. OtY had the highest TC and LP values after Tt. In a study conducted on 14 genotypes of *Oxalis tuberosa* Molina, varieties with yellow flesh and skin had higher values of these compounds (between 10 and 25 μ g/g) (Campos et al., 2006).

Correlational statistical analyses were performed to explain the influence of TC, LP, and TP on the AC. TP played a significant role in AC (0.9135 , $p < 0.05$), followed by LP (0.6845 , $p < 0.05$) and TC (0.6501 , $p < 0.05$). These findings indicate that TP may be a crucial contributor to the AC of the roots studied, as has been widely demonstrated in various research (Deng et al., 2013; Hwang et al., 2015; Sreeramulu and Raghunath, 2010).

4. Conclusions

This study shows these flours have excellent technological and nutritional characteristics, making them suitable raw materials for producing various processed foods. For instance, flours had water content and activity levels below the threshold required to prevent mold growth. Furthermore, all samples had bulk densities similar to those reported for hard and soft wheat flours. The flour made from IbP showed a lower capacity to absorb water from the environment than wheat flour. However, it still exceeds the reported value for wheat flour. The Tt and IbP samples exhibited a greater capacity for fat absorption than the Ot sample. This parameter can influence the characteristics of fried food made from this raw material.

The samples exhibited type-II isotherms, indicating the high hygroscopicity of the flour. The GAB model demonstrated a higher coefficient of determination for the mathematical modeling of moisture sorption isotherms. Tt flour displayed a higher chroma value than the other samples, which may be attributed to its TC and lycopene content.

Conversely, IbP exhibited the highest phenol value among all the samples. According to various studies, the high phenolic content of the roots is attributed to the presence of di-acylated anthocyanins. The TP content played a significant role in antioxidant activity because phenols are essential contributors to the AC of the roots' and tubers.'

Author contributions

Conceptualization, P.G.-S., J.M.-M., and M.I.; methodology, L.A. and M.I.; software, L.A., D.S., and M.E.G.; formal analysis, L.A., D.S., and M.E.G.; investigation, L.A., D.S., and M.I.; resources, P.G.-S., J.M.-M., and M.I.; data curation, L.A., D.S., M.E.G., and M.I.; writing—original draft preparation, L.A., D.S., and M.E.G.; writing—review and editing, P.G.-S., J.M.-M., and M.I.; supervision, M.I.; project administration, P.G.-S., J.M.-M., and M.I.; funding acquisition, P.G.-S., J.M.-M., and M.I. All authors have read and agreed to the published version of the manuscript.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

Marta Igual reports financial support was provided by Polytechnic University of Valencia. Liliana Acurio reports financial support was provided by Technical University of Ambato. If there are other authors, they declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

All data is contained in the article.

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