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# PHYSICOCHEMICAL QUALITY CHARACTERISTICS OF BUCKWHEAT FLOUR

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**KEYWORDS**

*Fagopyrum esculentum* Moench, pseudocereal, gluten.

**ABSTRACT**

The aim of this study was to characterize buckwheat flours produced using different processing methods, including with or without tegument removal and sieving with different mesh sizes. Flour A was produced by milling clean grains without husks or any other waste derived from peeling and separation processes and by sieving using 0.21-mm sieves. Flour B was a by-product of flour A, with grain size measuring 0.21–0.25 mm. Flour C was produced by milling whole grains, which after grain separation using 0.21-mm sieves resulted in a fine, whole-grain flour. Flour D was produced from what remained in the 0.21-mm sieves after sieving flour C and was processed using 0.25-mm sieves. Flours were assessed regarding water content, water activity, pH, acidity, color, microscopic characteristics, and protein and ash content. The study design was completely randomized, and differences were tested using an analysis of variance and comparison-of-means tests. Milling and separation procedures changed most quality parameters, except for water activity. Flour D contained the largest proportion of crude protein (19.87%) and showed high solubility and darker coloration than the other flours. The results of this study may be of relevance for consumers and for commercial buckwheat processing to optimize processing methods for food manufacturing.

## INTRODUCTION

Buckwheat (*Fogopyrumn esculentum* Moench) is considered a pseudocereal. It is a dicotyledonous plant belonging to the Polygonaceae family, and it is thus not closely related to common wheat (*Triticum aestivum* L.) as it does not belong to the monocot family Graminaceae. However, both species are commonly used to produce flour for similar usages, and their flours are comparable regarding their chemical composition (Wendler & Simonetti, 2016; Joshi et al., 2019). Buckwheat is known for its high nutritional value and its desirable dietary and medicinal properties, which is why buckwheat flour is frequently used for food production and actually know as a pseudocereal (Leiber, 2016; Mackela et al., 2017; Joshi et al., 2020).

Buckwheat flour does not contain gluten, and as an alternative to wheat flour products, gluten-free baked goods produced from buckwheat can be consumed safely by people suffering from gluten intolerance and/or allergy or celiac disease (Zhu, 2021). In addition, a diet rich in whole grains, which is the case in whole-grain buckwheat flour, may help prevent cardiovascular diseases including atherosclerosis, diabetes, and obesity (Beitane et al., 2018; Huda et al., 2021).

Quality characteristics of flours as water activity for example is an important measurement of how microorganisms react with food water. Pathogens may proliferate faster with increasing water activity; therefore, this parameter is crucial regarding product durability (Damodaran & Parkin, 2018). As a further aspect of product quality, imaging techniques such as microscopy can be used to identify foreign materials that are detrimental to flour quality, such as insect fragments or minerals which were added to powdered spices (Oliveira et al., 2015).

Due to its health benefits, buckwheat is sometimes considered a functional food (Luthar et al., 2020; Luthar et al., 2021; Matsui & Walker 2020). Functional foods are assumed to exert physiological benefits due to their healthy

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constituents (Candido & Campos, 2005). This category of foods belongs to a new nutritional concept originating from Japan where it was introduced in the 1980s through a governmental program with the purpose of promoting healthy foods to increase general health and prolong life expectancy (Anjo, 2004).

Buckwheat contains carbohydrates, dietary fibers, lipids, and phenolic compounds, and its protein content is higher than that of other crops such as rice, corn, and common wheat (Luthar et al., 2021). Moreover, buckwheat protein is attributed a high biological value, about 93.1% according with Luthar et al. (2021) as it comprises all essential amino acids, including lysine. Buckwheat is also a source of energy, which makes it an alternative to other crops used in human and animal nutrition (Leiber, 2016) and rich in flavonoids being dominant the rutin for the common buckwheat (*F. esculentum* Moench) and the Tartary buckwheat (*F.* t*ataricum* (L.) Gaertn.) (Borovaya & Klykov, 2020). Singh et al. (2020) appointed that buckwheat shows an immense potential of commercialization due to presence of essential nutrients and therapeutics and can contribute with nutritional security of world.

Evaluating buckwheat flour production processes and their effects on quality characteristics is thus important for consumers and for commercial processing so as to optimize food production from buckwheat. The aim of this study was to assess physicochemical characteristics of buckwheat flour produced using different milling and separation processes.

## MATERIAL AND METHODS

Grains of the buckwheat cultivar IPR 92 Altar were procured from the Experimental Farm area of the Federal University of Grande Dourados (*Universidade Federal da Grande Dourados*), located in Dourados, Mato Grosso do Sul, Brazil, at 22° 13' 58.656" southern latitude and 54° 59' 28.521" western longitude, and at an average altitude of 420 meters above sea level. Seeds were sown in the last week of February 2017, and no agrochemicals were used for crop management. Grains were harvested by manual cutting 100 days after emergence without using desiccants. Threshing was performed using a soybean harvester, followed by natural drying in an open yard under periodic rotation. Grains were then cleaned using sieves with the following mesh sizes diameters: 1.75 × 22 mm, 3.00 mm, 4.00 × 10 mm, 4.76 × 22 mm, and 5.0 mm (Dias & Oliveira, 2017) and subsequently packed in plastic bags and were stored in a refrigerator at an average temperature of 5 °C until processing.

To produce flour, buckwheat grain subsamples were processed in a testing device used for rice testing (PAZ-1 DTAM; Zaccaria, Limeira, Brazil). This device helps remove all the grains' tegument. The grain husk was subsequently removed using sieves with 0.21 mm mesh size. After cleaning, the grain was milled using a domestic blender for 2 minutes. The product was then sieved through a 0.25-mm mesh to produce flour at commercial-standard fineness.

As an alternative method, flour was produced from whole buckwheat grains (including integument and husk). Milling was also carried out using a domestic blender for the same duration, and the flour was separated using two sieves with 0.21 and 0.25 mm mesh size to produce whole-buckwheat flour.

The flowing four flour types (A, B, C, and D) were produced from these processes:

- Flour A: fine-grained flour from clean grains, free of husks or any other waste removed in the peeling process and sieved at 0.21-mm mesh size.
- Flour B: from waste of flour A, sieved, and with grain sizes between 0.21 and 0.25 mm coarser than flour A.
- Flour C: fine-grained whole-grain flour produced by milling whole buckwheat grains (including tegument and husk), followed by sieving at 0.21-mm mesh size.
- Flour D: from waste of Flour C which was retained in a 0.21-mm sieve and was then sieved again using a mesh size of 0.25 mm.

The four flours were subsequently assessed regarding their physicochemical characteristics. For color analysis, flour samples were placed in petri dishes, and coloration was recorded using a colorimeter device (CR-400; Konica Minolta, Tokyo, Japan) which, through reflectance, captured the coloration and provided luminosity values, as well as green-red and yellow-blue components, indicated by L*, a*, and b*, respectively. Measurements were performed in triplicates of each batch of flour samples. Subsequently, color, chromaticity (C*) and color angle (h) parameters were calculated (Yu et al., 2018).

Water absorption index (WAI) and solubility index were determined according to Anderson et al. (1970). Flour water content was determined using the standard oven method (Brasil, 2009). The amount of ash (%) indicating mineral content was measured by incinerating samples placed in porcelain crucibles, using a muffle furnace at a controlled temperature of about 550 °C for 4 h.

Protein content was measured using the Kjeldahl method by assessing the nitrogen concentration and converting it into a percentage with the factor 6.00 (IAL, 2008). Water activity was determined using Aqua.Lab equipment (BrasEq®) which had been calibrated previously. To measure pH values using a pH meter, approximately 10 g of each sample was diluted in 50 mL distilled water and determinated by direct reading. The hydrogen potential was quantified using a bench pH meter, previously calibrated using standard solution. Titratable acidity was measured in solution of flour and water, which is shown as mg NaOH per g sample.

Flour samples were dried to constant mass in an oven at 65 °C, after which microscopy was performed using a scanning electron microscope (TM 3000; Hitachi, Tokyo, Japan) at 400-fold and 2,000-fold magnification, in collaboration with the Central Analysis Laboratory of the Federal Technological University of Paraná (Campus Pato Branco, Brazil).

The study design was completely randomized, and four types of flours were evaluated in three replicates. The data were subjected to a normality test (Shapiro-Wilk test) and an equality of variance test (Bartlett's test); statistical significance is reported at $p < 0.05$. An F test was used in an analysis of variance followed by a Tukey's means separation test.

## RESULTS AND DISCUSSION

The average values of color parameters of buckwheat flour produced using different processes are shown in Table 1. All color indices which are parameters of colorimetric definition showed significant differences between flours.

TABLE 1. Average color parameters of buckwheat flours; L* luminosity, a* red-green, b* yellow-blue, C* chroma, and H (color angle).

| Flour type | color parameters | | | | |
|---|---|---|---|---|---|
| | L* | a* | b* | C* | H |
| A | 91.12 ± 0.14 a | 0.05 ± 0.03 b | 8.18 ± 0.16 b | 8.18 ± 0.16 b | 89.67 ± 0.23 a |
| B | 82.56 ± 0.51 b | 0.10 ± 0.25 b | 13.42 ± 0.25 a | 13.42 ± 0.25 a | 89.6 ± 0.46 a |
| C | 78.95 ± 0.48 c | 1.84 ± 0.10 a | 8.79 ± 1.30 b | 8.79 ± 1.30 b | 77.92 ± 2.13 c |
| D | 72.29 ± 1.35 d | 1.80 ± 0.17 a | 13.00 ± 0.35 a | 13.00 ± 0.35 a | 82.11 ± 0.53 b |
| p-value | 0.000* | 0.000* | 0.002* | 0.000* | 0.000* |

**Grades:** A: fine flour from cleaned grains, free of husks or any other waste derived from the peeling process; followed by sieving with 0.21-mm mesh size; B: coarse flour from clean grains, free of husks or any other waste derived from the peeling process; grain size between 0.21 and 0.25 mm; C: fine flour separated using 0.21-mm sieves; D: whole-grain flour with grain size between 0.21 and 0.25 mm. Shown are the mean values ± standard deviation; different lower-case letters in each column indicate statistically significant differences (Tukey's test); asterisks indicate significant differences using an F test.

Flour coloration is a crucial information criterion for consumers on which purchasing decisions are based. White or slightly off-white coloration is preferred, which, however, does not necessarily indicate better quality, as coloration results from various factors, some of which are intrinsic to the type of raw material, such as the pigment content (Gutkoski, 2009). With higher average L* values, flour A was whiter than the other flours (L* 100% indicates absolute white and 0% indicates black). This results from flour A containing only milled cotyledons and embryos of buckwheat grain, i.e., completely hulled grains lacking any particles that would darken its coloration. Flour D showed the lowest average L* value (72.29%) and, thus, the darkest coloration with a slightly brown tinge. Flour D was a whole-grain flour produced from grains and husks, and larger mesh sizes permitted incorporation of larger proportions of husk fragments, which also darkened the coloration.

All four flours showed positive a* and b* values, where positive a* values indicate a tendencey to red positive b* values indicate a tendency to yellow. As all respective average values were low, these differences were not distinguishable by the naked eye, and only a slight tendency to yellow was observed.

Color differences between the 4 flour types are shown in Figure 1, indicating that flours sieved with larger mesh sizes are darker (flours B and D) owing to the higher proportions of external components of buckwheat grains such as husk.

A B C D

FIGURE 1. Visual comparison of the four buckwheat flours A, B, C, and D.

Flours A and C (Figure 1 - A and C) were lighter and showed smaller granules, particularly in case of flour A (1). This resulted in white coloration with increasing purity. Flour C from whole grains showed slightly darker coloration with lower L* values (Table 1).

Average ash content corresponds to the amount of minerals, and significant differences between flours were observed (Table 2). Flours A and C showed lower ash content than flours B and D. This is plausible as minerals such as iron, sodium, magnesium, and phosphorus compounds occur at larger quantities in the outermost part of the grain (Germani, 2008). Flours B and D were sieved using larger mesh sizes, thus larger proportions of husks and other external fragments of the buckwheat grain can be expected. According to Germani (2008), the extraction degree has a significant effect on the ash content of flour. The amount of incorporated bran increases with the extraction degree; thus, the ash content is higher. The ash content of flours A, B, and C was below the maximum values permitted by Brazilian legislation (Brasil, 2005), which qualifies the types of flours on the market through their typification and stipulates the maximum ash content. Flour D contained 3.085% ash (Table 2). This high value is due to the processing of the whole grains and exceeds the legal limit in Brazil. Thus, the amount of external material is substantial and increases when using sieves with larger mesh sizes (0.25 mm) which permit incorporation of larger amounts of waste in the flour.

TABLE 2. Average ash content (%), water content (%), water activity (wa), acidity, and pH of buckwheat flours A, B, C, and D; ns: not significant.

| Flour type | Ash (%) | Water content (%) | wa | Acidity (%) | pH |
|---|---|---|---|---|---|
| A | 1.409 ± $^{0.002\ b}$ | 12.34 ± $^{0.001\ b}$ | 0.493 ± $^{0.023}$ | 5.1 ± $^{0,35c}$ | 6.29 ± $^{0.624}$ |
| B | 2.271 ± $^{0.001\ ab}$ | 11.92 ± $^{0.001\ ab}$ | 0.509 ± $^{0.006}$ | 5.8 ± $^{0,16b}$ | 6.18 ± $^{0.552}$ |
| C | 1.267 ± $^{0.001\ b}$ | 12.09 ± $^{0.001\ b}$ | 0.515 ± $^{0.003}$ | 6.2 ± $^{0,14b}$ | 6.36 ± $^{0.294}$ |
| D | 3.085 ± $^{0.001\ a}$ | 11. 36 ± $^{0.001\ a}$ | 0.488 ± $^{0.016}$ | 12.4 ± $^{0,22a}$ | 6.34 ± $^{0.289}$ |
| p-value | 0.000* | 0.000* | 0.056$^{ns}$ | 0.000* | 0.945 $^{ns}$ |

**Grades:** A: fine flour from cleaned grains, free of husks or any other waste derived from the peeling process; followed by sieving with 0.21-mm mesh size; B: coarse flour from clean grains, free of husks or any other waste derived from the peeling process; grain size between 0.21 and 0.25 mm; C: fine flour separated using 0.21-mm sieves; D: whole-grain flour with grain size between 0.21 and 0.25 mm. Shown are the mean values ± standard deviation; different lower-case letters in each column indicate statistically significant differences (Tukey's test); asterisks indicate significant differences using an F test. $^{ns}$: not significant

The average water content of buckwheat flours ranged from 11.36% ± 0.001% to 12.34% ± 0.001%. The adequate water content depends on accurate storage at suitable temperatures in a suitable environment. Average water activity values of the four flours ranged from 0.488 to 0.515 at an average temperature of 24.6 °C (Table 2), and no significant differences between flours were observed. Water activity values ranged around 0.50, which may favor processes such as lipid oxidation, non-enzymatic browning, and other non-enzymatic activities. However, the observed water activity values were not sufficient for the development of fungi and bacteria, as these processes occur when water activity exceeds 0.66.

Flour D, which was produced from milling whole grains and sieving them with 0.25 mm mesh size, showed the highest average acidity value (12.4%). This may be due to the presence of large peel portions and external material and because these outermost compounds are more prone to deterioration.

Furthermore, buckwheat is a source of diverse amino acids, including essential amino acids (Zhu, 2021), and fatty acids, the greater proportion of which are linoleic acid and palmitic acid (Golijan et al., 2019). The pH of buckwheat flours was not influenced by the processing ($p > 0.05$), and the range of pH values was between 6.18 and 6.36, for flours B and C, respectively. Despite the titratable acidity being higher for flours C and D, H+ ion activity was not detected in the pH analysis that considers a variation of ten times in concentration. The buckwheat flours in general have pH of approximately 6.0, which is slightly acidic with good sensorial acceptability for food applications and favors some processes for protein isolation and solubilization-precipitation (Zhu, 2021).

The WAI and the solubility index showed significant differences between flour types (Table 3). The average WAI of flours A and C were lower when compared to the others flours, at 2.004 and 2.155 g gel/g, respectively. We thus suggest that finer flours retain less water due to smaller granular sizes, compared to flours B and D which showed higher water absorption capacities. The food processing industry uses flours with high WAI values, and this characteristic is linked to the number of hydroxyls in starch granules which favors binding of water molecules. Starch granules between 0.21 and 0.25 mm, which were present in flours B and D, showed greater water absorption capacity. For these same reasons, the number of soluble solids found in flours B and D showed higher average solubility index values, which results in reducing starch molecule size and increases solubility in water (Ascheri et al., 2006).

TABLE 3. Average values of water absorption index (WAI) (g gel.g flour$^{-1}$**)**, solubility index (SI), and protein content in buckwheat flour.

| Flour type | WAI (g gel. g flour$^{-1}$) | SI (%) | Protein (%) |
|---|---|---|---|
| A | 2.004 ± $^{0.051d}$ | 0.046 ± $^{0.002c}$ | 5.94 ± $^{1.65\ a}$ |
| B | 2.391 ± $^{0.023\ b}$ | 0.078 ± $^{0.005\ b}$ | 13.52 ± $^{1.81\ a}$ |
| C | 2.155 ± $^{0.075c}$ | 0.052 ± $^{0.001c}$ | 6.68 ± $^{1.34\ a}$ |
| D | 2.752 ± $^{0.033a}$ | 0.096 ± $^{0.003a}$ | 19.87 ± $^{1.95\ b}$ |
| p-value | 0.000* | 0.000* | 0.000* |

**Grades:** A: fine flour from cleaned grains, free of husks or any other waste derived from the peeling process; followed by sieving with 0.21-mm mesh size; B: coarse flour from clean grains, free of husks or any other waste derived from the peeling process; grain size between 0.21 and 0.25 mm; C: fine flour separated using 0.21-mm sieves; D: whole-grain flour with grain size between 0.21 and 0.25 mm. Shown are the mean values ± standard deviation; different lower-case letters in each column indicate statistically significant differences (Tukey's test); asterisks indicate significant differences using an F test.

Regarding the protein content (Table 3), it is noted that the buckwheat flours presented different mean values at the 5% level of significance. In the finer grained flours (A and C) the averages of 5.94 and 6.68% were verified, respectively. Buckwheat flour processed using the seed coat and cotyledon (Flour D) had the best nutritional value considering the crude protein content (19.87%). Buckwheat flours obtained in processes, A and C, when compared to conventional wheat flour (*Triticum* spp) do not adequately show the minimum required by Brazilian regulations, IN nº 8 of MAPA (2005), called "Technical Regulation of Identity and Quality of Wheat Flour" which establishes a minimum of 7.5% for wheat flours classified as Type 1 (granulometry less than 0.250 mm) (Brasil, 2005). The fact, despite being negative, corresponds exactly to what was expected, since the flours under study are derived from buckwheat that does not have the proteins gliadin and glutenin (gluten-forming) in its composition, this characteristic allows the obtainment of so-called weak flours (weak) in addition to presenting low elasticity, the absence of gluten also compromises the protein content. According to Sinkovic et al. (2021) the average contents of crude protein in buckwheat grains are in the range of 12% and 18.9%. Kahlon et al. (2018) presented values in the range of 16% for flour for ancient whole grain buckwheat that was used for development of gluten-free snacks. Thus, one of reason that can be pointed for a value higher than this for the type D flour sample is a higher concentration of protein-rich structures, such as embryonic tissue and aleurone. Both are protective layers of the starchy endosperm of grain and rich in proteins; that are, in this type of processing and separation, responsible for an increase in the total protein content of the flour.

Figure 2 shows microscopic characteristics of different buckwheat flours at different magnifications.

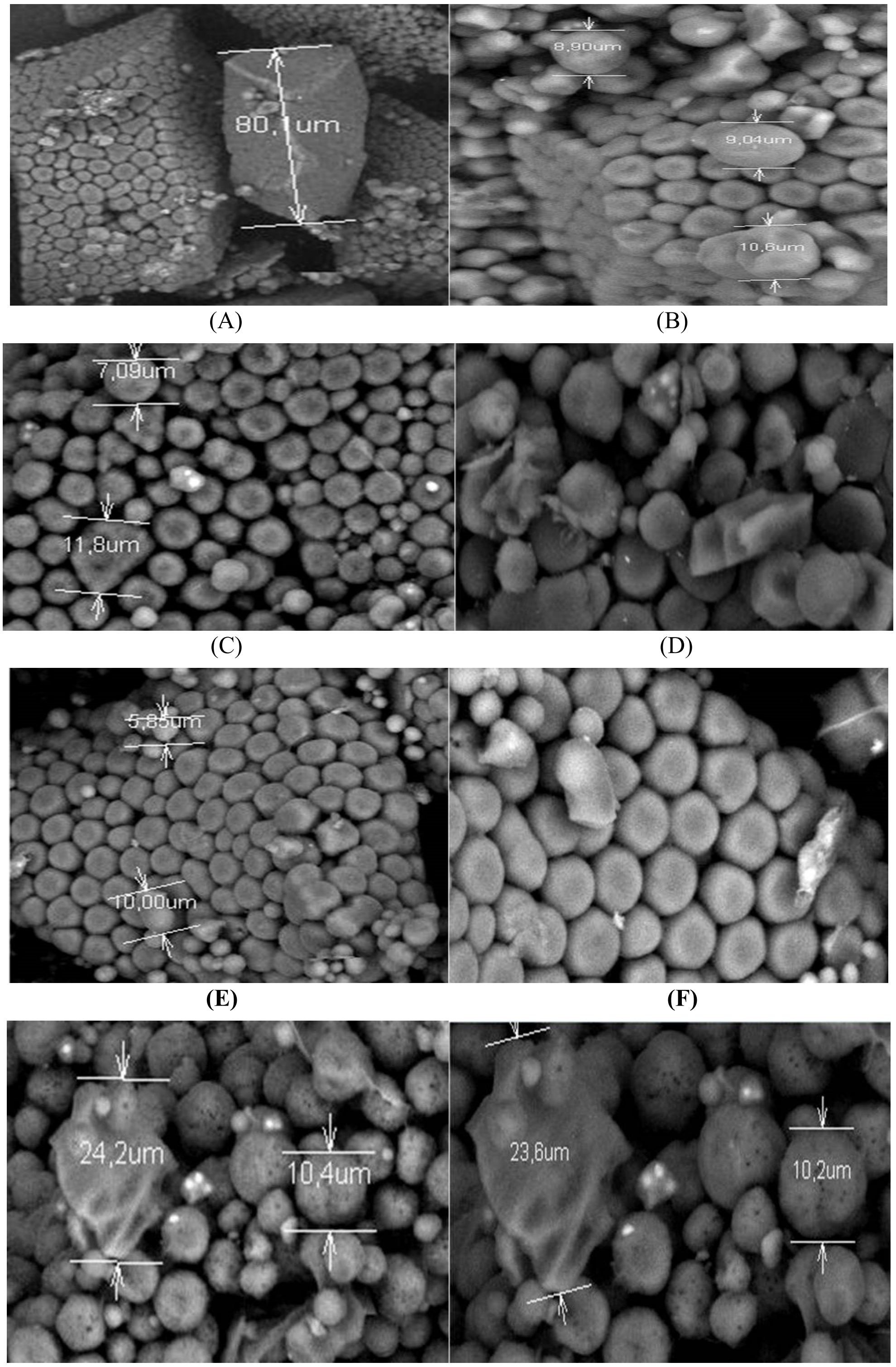


FIGURE 2. Microscopy of buckwheat flour A at 400-fold (A) and 2,000-fold (B) magnification; panels C and D show flour B at 1,200- and 1,800-fold magnification, respectively; panels E and F show flour C at 1,000-fold and 1,800-fold magnification, respectively; panels G and H show flour D at 1,000-fold and 1,800-fold magnification; respectively.

As shown in Figure 2 (A), clusters of various spherical materials can be observed. These clusters are parenchymal starch cells with a size of approximately 80 µm. At higher magnification (2,000-fold), starch grains forming the starch parenchyma are visible in flour produced form hulling and milling of buckwheat, at a minimum size of approximately 9.04 µm (Figure 2 B). In flour A, no foreign material such as insect parts or any other type of dirt was found, which indicates high quality.

Flour B starch grains are larger than those of flour A (Figure 2 C). This is because this flour was passed through a sieve with 0.25 mm mesh size which allowed larger particles to be incorporated. On the right side of the image in panel D of Figure 2, in a different frame of this sample, materials with non-spherical shapes can be observed. These are tegument fragments of processed grains that are more lignified and harder, and which were more resistant to processing and were thus less degraded than amyliferous parenchyma. Furthermore, according with Martínez-Villaluenga et al. (2020) the buckwheat starch can improve the dough development and capacity of globulin protein fraction to form and stabilize emulsions in the flour.

Panels E and F of Figure 2 show starch grains of similar sizes as those found during the processing analysis of flour A. This is because the same mesh size (0.21 mm) was used to sieve flours A and C which thus comprised very small fragments including starch grains of approximately 5.58– 10 µm. Panel F of Figure 2 is a magnification showing some fragments other than starch grains which are parts of the buckwheat husk that was broken down during whole-grain processing. The results suggest the absence of material which does not originate from buckwheat grain. Panels E and F of Figure 2 suggest the absence of dirt and impurities in the flour.

In panels G and H (Figure 2) the material of different shapes and sizes can be observed, which likely originate from the processing of whole grains including husk, as was done to produce flour C. In contrast, however, flour C was sieved using a larger mesh size (0.25 mm). Therefore, parts of husks and of the pericarp occur at larger sizes, and starch grains measured up to 10.4 µm and other parts of the grain up to 24.2 µm. All components shown in the picture originated from the raw material. Furthermore, none of the flours suggested presence of admixtures, which may occur when the original product is mixed with other substances (Oliveira et al., 2015).

## CONCLUSIONS

The physicochemical characteristics of buckwheat flour corresponded to the Brazilian legislation regarding water content, WAI, solubility index, pH, acidity and color, with the exception of water activity. Crude protein and mineral content were higher in flours that contained particles larger than 0.21 mm. Thus, this flour type appears to be recommendable for consumers and for industrial purposes.

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