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Exploring starches from varied sorghum genotypes compared to commercial maize starch

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Abstract

Sorghum has been lately attracted the attention for human consumption as a gluten-free cereal rich in bioactive compounds. Sorghum starches isolated from brown (BRS305), red (BRS308), and white (CMSXS180) genotypes were compared to commercial maize starch, based on proximate composition, particle size distribution, microstructure, X-ray diffraction, amylose and total starch content, solubility index, swelling power, pasting, and thermal properties. The carbohydrate content of sorghum starch samples ranged from 98.24 to 99.23 g/100 g (dry basis). The particle size distribution of the CMSXS180 sorghum genotype was very similar to commercial maize starch. The relative crystallinity values varied from 29.83% to 30.39%. The water solubility index and swelling power of sorghum starches were lower than those of maize starch. Sorghum genotypes paste profiles were similar, but greatly differed from maize showing the highest final viscosity (4042.0–4444.5 cP) compared to maize starch (3767.5 cP) and lower gelatinization enthalpy ($\Delta H = 9$–10.5 J/g) than maize starch (11.8 J/g). The results showed some distinct properties of sorghum starches when compared to maize starch, which may contribute to provide alternative uses, particularly in food products requiring very high viscosity and retrogradation.

Practical Applications

Sorghum pericarp is rich in bioactive compounds, which can be used as a main source of ingredients, for example, to the nutraceutical industry. The sorghum grain without pericarp (decorticated), used to produce purified starch (without chemical treatment), may contribute to the total use of this cereal by limiting chemical residues. In addition, sorghum starch has shown particular technological properties different from commercial maize starch, providing alternative uses in the food industry, for example, in products that require high viscosity from hot to cold processes.

KEYWORDS
paste viscosity, Pearson's correlation, principal component analysis, starch isolation, starch swelling

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1 | INTRODUCTION

Sorghum is the fifth most important cereal crop with an annual production of approximately 60 million tons in the world, after wheat, maize, rice, and barley (FAO, 2020). Sorghum (*Sorghum bicolor* [L.]) stands out for being important in food security, as a staple food. It is a gluten-free cereal, carbohydrate-rich with slowly digestible starch and has great potential to increase the consumption as nutri-cereals in human food (Hossain et al., 2022).

In several industrial segments, sorghum cereal has gained considerable attention. For example, it has been considered as a natural antimicrobial ingredient and colorant source, which are particularly found in the sorghum pericarp, as it contains phenolic compounds such as flavonoids (Espitia-Hernández et al., 2022). Flavonoids are also responsible for free radicals scavenging by protecting the human body against oxidative stress (Mawouma et al., 2022). According to Ofosu et al. (2020) even in decorticated sorghum grains there are flavonoids capable of preventing diabetes and obesity.

By using sorghum as a source of bioactive extracted from the pericarp, the resulted endosperm has the potential to be a sustainable alternative of the total utilization of sorghum, which has approximately 70% of starch content (Fărcaş et al., 2021; Singh et al., 2010). Sorghum starch has been virtually not explored as industrial ingredient yet when compared to maize. However, in a study, the use of sorghum starch has been reported as an interesting source of great potential application in food processing in products such as tortillas, porridges, noodles, and couscous showing functional properties as a binder better than acacia gum, for example, (Zhu, 2014).

Several studies have focused either on the characterization of starches from different genotypes or on the evaluation of changes that occur by external physical and chemical agents. However, a physicochemical correlation study between sorghum starches genotypes and commercial maize starch has not been yet carried out. Also, there has been a growing awareness regarding food labels, resulting in increased demand of consumers for "clean-label" ingredients fostering research for non-chemically modified starches.

Therefore, the aim of this study was to isolate starch (non-chemical treatment) from three Brazilian sorghum genotypes and to characterize their physicochemical, particle size distribution, microstructure, pasting, and thermal properties comparing to commercial maize starch, by understating the statistical correlations through principal component analysis (PCA), hierarchical clustering on principal components (HCPC), and Pearson's correlation, therefore evaluating the potential use of sorghum as a commercial source of starch.

2 | MATERIALS AND METHODS

2.1 | Sample preparation

As raw materials for the present work were grown sorghum genotypes BRS305 (brown pericarp), BRS308 (red pericarp), and CMSXS180 (white pericarp), provided by Embrapa Milho e Sorgo, located in Sete Lagoas—Brazil (geographical coordinates: 19°26' S latitude and 44°10' W longitude), with initial moisture content of 13 ± 0.5% (wet basis). The sorghum grains were manually cleaned to remove foreign particles and 355 g of cleaned grains were decorticated in a rice milling machine TM-97 (Suzuki S/A, São Paulo, Brazil), and 300 g were obtained, without the pericarp. Decorticated grains (300 g) were stored in plastic (low-density polyethylene-PE-LD 4) bags, kept under refrigeration at 8°C, for subsequent starch extraction. Commercial maize starch (Maizena®, Unilever, Garanhuns, Brazil) was acquired from the local market of Rio de Janeiro (Brazil).

2.2 | Starch isolation

Sorghum starch isolation was carried out by wet milling and no chemical treatment was used, according to the flowchart showed in Figure 1 and follow the methodology described by da Silva et al. (2020), with modification. The grain to water ratio was increased because it was found that a greater water volume facilitated the separation of starch during sieving.

The Starch yield (%) was calculated according to Silva et al. (2020) using Equation (1):

$$\text{Starch Yield}\,(\%) = [(\text{Dry starch mass g}) \div (\text{Raw material mass g})] \times 100 \quad (1)$$

2.3 | Proximate composition

Moisture and ash were determined using the same equipment, a thermogravimetric analyzer TGA-2000 (Navas Instruments, Conway), corresponding to method no. 925.09 and 923.03 of the Association of Official Analytical Chemists (AOAC) (2005), respectively. Moisture content was determined at 105°C and ash at 550°C. Protein contents were determined in accordance with Kjeldahl method no. 2001.11, of

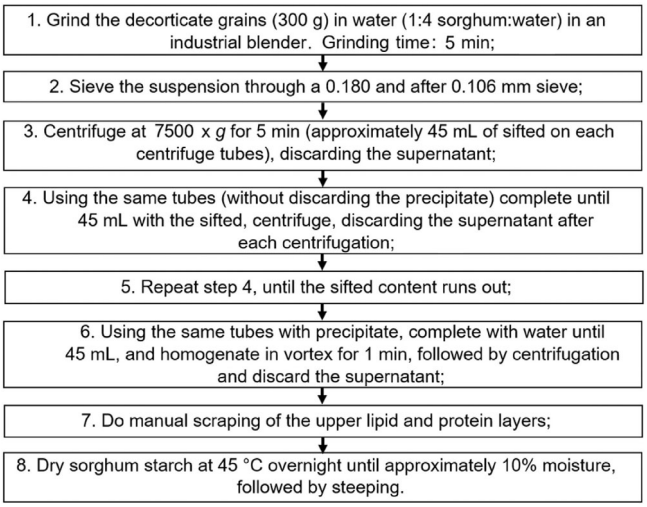

FIGURE 1 Laboratory scale scheme used to isolate starch from decorticate sorghum genotypes (BRS305, BRS308, and CMSXS180), following the methodology described by da Silva et al. (2020) with modification

the AOAC (2005). Lipid content was determined according to the official method Am 5-04 of the American Oil Chemists Society (AOCS), (2005). Carbohydrate content in wet basis (carbohydrate wb) was calculated by the difference (Equation 2) for each sample as below:

$$\text{Carbohydrate wb} = 100 - (\text{Moisture} + \text{Ash} + \text{Protein} + \text{Lipid}) \quad (2)$$

From the calculation of carbohydrate content in wet basis (Equation 2), the carbohydrate content in dry basis (carbohydrate db) was calculated (Equation 3) to disregard the influence of moisture.

$$\text{Carbohydrate db} = (\text{Carbohydrate wb} \times 100) \div (100 - \text{Moisture}) \quad (3)$$

2.4 | Particle size distribution

The particle size distribution of sorghum and maize starches followed the method 55-40.01, with some modification, that are described in the approved methods of the American Association of Cereal Chemist (AACC) (1999). Approximately 0.15 g of starch was previously dispersed in 200 ml of deionized water and then, the solution was placed on a S3500 laser particle size analyzer (Microtrac Inc., Montgomeryville). The average particle diameter was measured in the range of 0.02–2000.00 μm and the particle size distribution expressed in μm.

2.5 | Scanning electron microscopic analysis

The starch granules previously kept in a desiccator with silica gel until 4 ± 0.5% of moisture were carefully deposited on the top of a double-sided adhesive carbon tape, fixed onto an aluminum sample holder. Starch morphology was examined on a scanning electron microscope TM 3000 (Hitachi, Tokyo, Japan) vacuumed and analyzed at an accelerating voltage of 15 kV and on a 50 μm scale (Bernardo et al., 2018).

2.6 | X-ray diffraction and relative crystallinity

Diffraction pattern was performed following the methodology described by Bernardo et al. (2018): Samples were placed in an X-ray diffraction D2 Phaser (Bruker, Rheinfelden, Germany), operating at Cu-K, with 0.154 nm of wavelength, target voltage, and current of 30 kV and 10 mA, respectively. Samples were analyzed from 2° to 32° (2θ) in the range of 0.15° min^{-1}, a step size of 0.02° and with a divergence, scatter and receiving slit width of 0.6, 0.6, and 0.2 mm, respectively; The relative crystallinity (RC) was calculated considering the ratio of the crystalline area and total area using the Diffrac.Suite Eva version 3 software (Bruker AXS, Rheinfelden, Germany).

2.7 | Amylose content

The amylose content was determined by the iodine calorimetric method described by Boudries et al. (2009). Approximately 100 mg of starch were dispersed with 10 ml of urea-dimethyl sulfoxide in a glass test tube. The starch suspension was vortexed using a vortex mixer (Vortex Genie 2, Scientific Industries, Bohemia, NY) and placed in a boiling water bath for 60 min. After cooling, 100 μl of the starch suspension were added to 9.7 ml of distilled water and 200 μl of iodine solution, followed by homogenization. The solution was placed in darkness during 20 min. The absorbance was measured at 635 nm using a spectrophotometer UV-2401 (Shimadzu, Kyoto, Japan).

2.8 | Total starch content

Total starch content was used to determine purity of starch. The analysis was determined following method no. 996.11 of AOAC (2005) and 76.13 of American Association of Cereal Chemist (AACC) (1976), with some modifications. 100 mg of each sample and 200 μl of 80% ethanol were added to a glass teste tube, and then, stirred using Vortex Genie 2 (Scientific Industries, Bohemia, NY). The content of 3 ml of α-amylase was introduced in related glass test tube and incubation in a boiling water bath, with stirring (every 2 min) for 6 min. Cooling was performed, followed by adding 100 μl of amyloglucosidase and the tubes were incubated at 50°C for 30 min. For each sample, the content of tube was made up to 100 ml and centrifuged in a Universal 320R centrifuge (Hettich, Tuttlingen, Germany) at 7500×*g* for 10 min. After that, 50 μl of each sample and 1.5 ml of GOPOD reagent were added into respective eppendorf; as control, 50 μl of D-glucose with 1.5 ml of GOPOD reagent were added into eppendorf, and 50 μl of distilled water (blank) with 1.5 ml of GOPOD reagent were added into another eppendorf. The eppendorfs were incubated at 50°C for 20 min. The absorbance was measured at 510 nm using a spectrophotometer UV-2401 (Shimadzu, Kyoto, Japan) for determination of total starch (%), in wet basis, according to Equation (4) as follows:

$$\text{Total starch}\,(\%) = (\Delta A \times 90) \div (A \times W) \quad (4)$$

where ΔA is the absorbance read of each sample against the blank; A is the absorbance of D-glucose read against the blank; and W is the sample weight (mg).

From the calculation of total starch (%) content in wet basis (Equation 4), the total starch content in dry basis (total starch db) was calculated (Equation 5) to disregard the influence of moisture content in each sample.

$$\text{Total starch db}\,(\%) = (\text{Total starch} \times 100) \div (100 - \text{Moisture}) \quad (5)$$

All results referent to total starch content were expressed in dry basis.

2.9 | Solubility index and swelling power

The estimation of solubility index (SI) and swelling power (SP) at varied temperatures (55–95°C) were determined following the methodology described by Tsai et al. (1997), with some modifications perfumed in triplicate.

1 g of starch (m_i) was stirred in 10 ml of distilled water in a centrifuge tube using a vortex Genie 2 (Scientific Industries, Bohemia, NY). The centrifuge tubes containing starch solution were heated in water bath, at varied temperatures (55, 65, 75, 85, and 95°C, respectively) for 30 min each; followed by cooling in ice and centrifuged on a Universal 320R centrifuge (Hettich, Tuttlingen, Germany) at 7500×*g* for 10 min.

The supernatant was dried at 110°C until dry mater was obtained at constant weight (m_s). SI was expressed as the ratio of dry mater supernatant (m_s) to the initial mass (m_i) of starch (Equation 6).

$$SI = m_s \div m_i \quad (6)$$

SP was described as the ratio of the sedimented starch (m_a) to the initial starch weight (m_i) multiplied by (1 − SI%; Equation 7) as below:

$$SP = (m_a) \div [m_i x\,(1 - SI\%)] \quad (7)$$

2.10 | Pasting properties

The pasting viscosity properties were determined in duplicate using a Rapid Viscosity Analyzer-RVA Series 4 (Newport Scientific, Warriewood, NSW, Australia) following the methodology described in Comettant-Rabanal et al. (2021). Three grams of starch with adjusted moisture content at 14% (wet basis) were added in 25 ml of distilled water in an aluminum canister placed on the equipment running at 160 rpm and starting heating with a temperature of 25°C. The recorded readings of the pasting curve were: pasting temperature (PT), cold viscosity at 25°C (CV.), peak viscosity at 95°C (PV), minimum viscosity after heating (mV), maximum viscosity at cooling (MV), final viscosity (FV); and then calculated: breakdown viscosity (PV-mV) and setback viscosity (FV-mV). The data were analyzed using the Thermocline for Windows version 3.0 (Newport Scientific Pty Ltd, Warriewood, Australia).

2.11 | Differential scanning calorimetry

The thermal properties were determined using a DSC Q200 (TA Instruments, New Castle). Deionized water and approximately 2 mg of starch (water: starch ratio, 2:1) were weighed in a hermetic aluminum pan. The pans were sealed and remained at rest overnight at room temperature. An empty pan was used as reference. Scan occurred in the range from 5 to 110°C at a rate of 10°C/min (Bernardo et al., 2018). The onset (To), peak (Tp), conclusion (Tc), as well as the calorimetric enthalpy (ΔH) were measured from the thermograms using the Advantage software version 5 (TA Instruments, New Castle) and posteriorly calculated gelatinization temperature range (Tc−To). Analyses were performed in duplicate.

2.12 | Statistical analyses

Analysis of variance (ANOVA) was developed to determine the statistical differences ($p < 0.05$) among samples and, when differences were found a multiple mean Tukey test was carried out at a significant level of 5%. PCA was performed after variable standardization to avoid the influence of different magnitude orders. HCPC was conducted using Euclidean distances and Ward's method. Pearson's correlation was used to analyze the interactions of variables. The statistical analyses were performed by using R free statistical software, version 3.2.4 (R Foundation for Statistical Computing, Vienna, Austria).

3 | RESULTS AND DISCUSSION

3.1 | Yield starch and proximate composition

After laboratory-scale starch isolation the yields of sorghum starches were close to 30% between the BRS305, BRS308, and CMSXS180 genotypes with 28.00%, 29.87%, and 28.14%, respectively. Similar values were obtained by Sira and Amaiz (2004) with sorghum starch yields in the range of 27.73% and 30.00%. These low yields may be associated with the lower value of non-carbohydrate compounds, according to the results found below.

The proximate composition of sorghum and maize starches is displayed in dry basis in Table 1, because the moisture content (wet basis) showed statistical difference ($p < 0.05$) among BRS305, BRS308, CMSXS180, and maize starches (9.94, 9.57, 10.06, and 11.70 g/100 g, respectively, in wet basis). Therefore, the values were presented on a dry basis for a correct comparison between the different samples and the data in other studies.

Carbohydrate content (dry basis) range from 98.24 to 99.23 g/100 g while maize starch presented 99.85 g/100 g ($p < 0.5$; Table 1). Similar results were reported by Zhang et al. (2021) that

TABLE 1 Proximate composition (dry basis) of sorghum starch genotypes (BRS305, BRS308, and CMSXS180) and maize starch with R^2 for ash, protein, lipid, and carbohydrate

Composition (g/100 g)	BRS305	BRS308	CMSXS180	Maize	R^2
Ash	0.35 ± 0.02^{ab}	0.62 ± 0.17^{b}	0.59 ± 0.06^{b}	0.00 ± 0.00^{a}	0.89
Protein	0.16 ± 0.05^{b}	0.70 ± 0.00^{c}	0.00 ± 0.00^{a}	0.00 ± 0.00^{a}	0.77
Lipid	0.47 ± 0.14^{a}	0.44 ± 0.12^{a}	0.18 ± 0.00^{a}	0.15 ± 0.02^{a}	0.87
Carbohydrate	99.02 ± 0.20^{a}	98.24 ± 0.29^{b}	99.23 ± 0.06^{ac}	99.85 ± 0.02^{c}	0.96

Note: Values represent mean ± SD ($n = 3$). Different letters in the same row indicate statistical differences ($p < 0.05$) by Tukey test. Values were presented on dry basis (moisture content = 0 g/100 g for all starches) to reduce the effects of moisture content in wet basis.

performed chemical extraction of sorghum starch finding (dry basis) 99.3 g/100 g of carbohydrate, 0.3 g/100 g of protein, 0.2 g/100 g of lipids and 0.2 g/100 g of ashes. In the review of Zhu (2014) in relation to sorghum as a starch source, they reported protein varying from 0.2 to 2.1 g/100 g, lipids from 0.0 to 1.5 g/100 g, ash from 0.1 to 1.6 g/100 g, and carbohydrate from 95.5 to 99.6 g/100 g.

In the analysis of protein content in dry basis, only BRS308 genotype showed a not so low protein content (0.70 g/100 g), which may not be interesting when pure starch is desirable (Table 1), the other sorghum genotypes presented lower protein content close to maize. According to Cardoso et al. (2006), the increasing use of starches by the food industry has grown the interest of removing protein from binding starch granules, since the higher the purity of the granule, the better its performance as an additive.

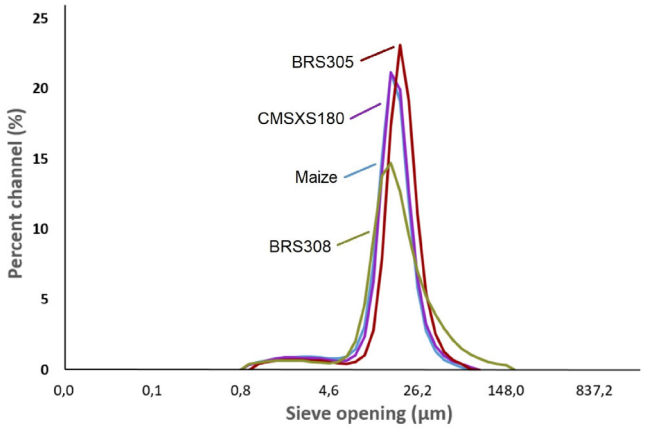

FIGURE 2 Particle size distribution of sorghum starch genotypes (BRS305, BRS308, and CMSXS180) and maize starch

3.2 | Particle size distribution

The particle size distribution of the starch granules is displayed in Figure 2. Sorghum (BRS305, BRS308, and CMSXS180) and maize starches were similar in size, particularly CMSXS180 and maize presented nearly the same particle size distribution. BRS305 exhibited the lowest amount (8.1%) of small starch granules (<10 µm), whereas BRS308 showed the highest amount (14.02%, identical to maize). Concerning the range of large granule sizes (>30 µm), BRS308 also showed the highest value (18.9%).

The range between 10 and 20 µm had the major volume in particle size distribution compared to all ranges. The percentage of granules in this predominant range (10–20 µm), followed this order: 50.51% for BRS308, 51.24% for BRS305, 61.52% for CMSXS180, and 62.93% for maize, confirming that CMSX180 and maize have similar particle size distribution. Kaufman et al. (2018) evaluated the particle size of 19 different sorghum starch genotypes and found the same predominant range (10–20 µm) with 45.66% to 54.58% of particle size distribution.

3.3 | Scanning electron microscopic analysis

Scanning electron micrographs of sorghum and maize starch are displayed in Figure 3.

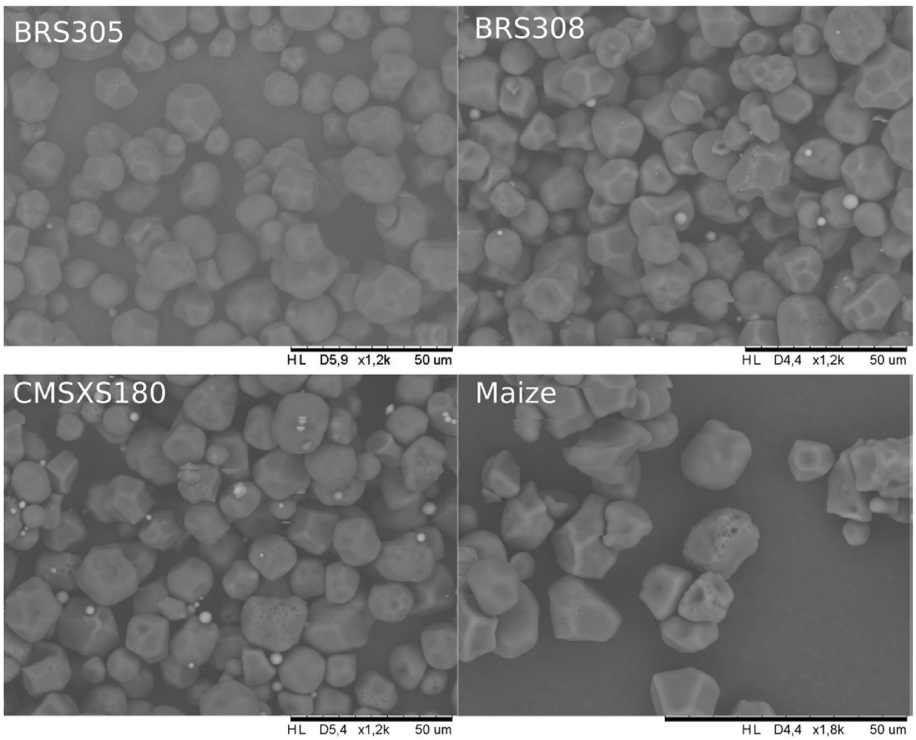

FIGURE 3 Scanning electron micrographs of sorghum genotypes (BRS305, BRS308, and CMSXS180) and maize starch

The image analysis showed that all starch granules presented an irregular polygonal shape and a considerable degree of indentation. Both sorghum and maize starches were similar in size, which corroborates with the results of particle size distribution (Figure 2). Similar findings were reported by Singh et al. (2010), who identified sorghum starch granules with an irregular polygonal shape and spherical form in sorghum cultivars found in India. In addition, the starch morphological characteristics from different plant sources may vary with the genotype and cultivation practices, thus the variation in size and starch granule shape could be assigned to their biological origin.

3.4 | X-ray diffraction pattern and relative crystallinity

Sorghum starch diffractograms were similar to maize starch, which was expected since both plants belong to the same botanical family. Starches, in their native form, presented diffraction peaks corresponding to the Bragg angle (2θ) of 15, 18, and 23°, and a representative crystallinity pattern type A, as previously reported (Cui, 2005).

BRS305 and CMSXS180 showed greater values of relative crystallinity (29.8% and 30.4%, respectively) compared to BRS308 (27.2%) and maize starch (24.3%), which may indicated a higher amount of amylopectin in their starch granules than the amorphous region where amylose molecules are present. The relative crystallinity found was similar to mentioned by Zhu (2014), in sorghum starches ranging from 22.7% to 29.6%.

3.5 | Amylose and total starch content

CMSXS180 (23.39%) and BRS308 (23.16%) showed the highest amylose content, whereas BRS305 had the lowest amylose content (22.86%). These values are similar to those observed by Singh et al. (2010), who studied 15 genotypes of sorghum grown in India. They found that amylose content ranged from 11.2% to 28.5%.

Maize starch had the highest amylose content (24.50%) compared to sorghum. Wang et al. (2022) reported about 25% of amylose in normal maize starch, type A crystalline pattern, which was associated to starches with more compact crystal arrangement, similar to the found in the present work.

It is worth to a note that physical, chemical, and functional properties are influenced by several factors including granule size, genotype, and amylose/amylopectin ratio content. In particular, the amylose/amylopectin ratio can vary among botanical source and starches with 25% amylose can be classified as normal amylose content for cereals (Del Buono et al., 2019).

Total starch content was analyzed in order to determine how pure the starches were. The proposed extraction method led to a small decrease in total starch content for sorghum genotypes BRS305 (90.32%), BRS308 (89.48%), and CMSXS180 (90.03%) in dry basis, when compared to commercial maize starch (93.15%). These variations, in total starch content among samples were not enough to cause a statistical difference ($p < 0.05$).

Wang et al. (2022) reported that the total starch content of normal maize starch without chemical modification was 92.2%, similar to results of sorghum and maize starches analyzed. The same author also mentions a relationship between a decrease in the total starch content and an increase in the damaged starch content by others physical treatments generating damage to the internal structure of starch. For the BRS308 genotype, it was possible to observe this relationship, through the increase of small granules by the particle size analysis, although and decrease of total starch content.

According to Palavecino et al. (2019) sorghum isolated by physical separation exhibited 95.1% to 96.7% of total starch, while others extraction performed with long alkali or sulfur dioxide steeping reported purity from 93% to 99%. These results demonstrate that is possible to obtain high levels of purity in the starches by methods without chemical modification.

3.6 | Swelling power and solubility index

The SP profile of all starch granules (Figure 4a) typically increased from 65 to 95°C. SP of maize starch was higher than sorghum genotypes ($p < 0.05$).

In contrast to maize, BRS305 presented the lowest values of SP with a gradual increase from 55 to 85°C, whereas SP profiles of

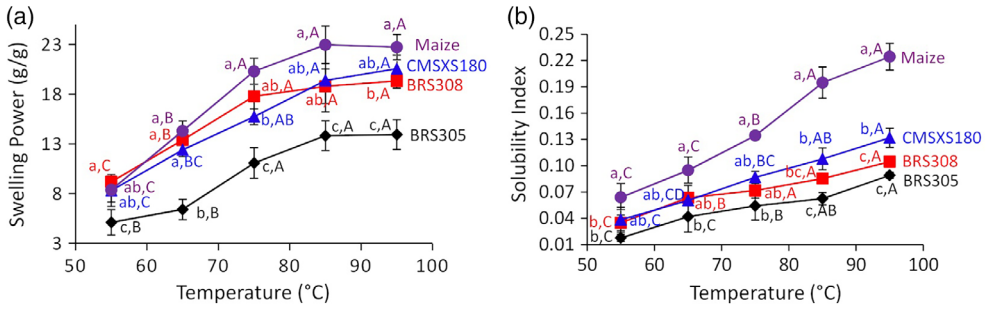

FIGURE 4 Swelling power (a) and solubility index (b) of sorghum (BRS305, BRS308, and CMSXS180) and maize starches. The capital letter means significant differences among different temperatures in the same sample ($p < 0.05$). Small letters indicate significant differences among samples at the same temperature

BRS308 and CMSXS180 were similar to maize starch in almost temperature ranges (Figure 4a). Punia (2020) reports that the swelling behavior of starch cereals is mainly related to the presence of amylopectin, hence higher values of swelling power suggest higher amylopectin content with less rigid granular structure, when compared to starches with lower amylopectin content. In comparison with maize starch, sorghum starch showed to be less susceptible to breakage under extended heating.

According to Liu et al. (2016) and Olayinka et al. (2013), low SP and SI may indicate more granular stability (strong interactions amylose-amylopectin), therefore it was indicated that all studied sorghum starches showed higher stability than maize starch. In addition, the lower swelling power for BRS305 (Figure 4a) within analyzed temperature ranges, indicates that there is interaction among amylose-amylose and amylose-amylopectin chains in heat moisture treatment; generating more rigid structure in double-helical amylopectin side chains, making difficult the interaction with water, inducing to low swelling power (Sindhu et al., 2021).

Both maize and sorghum starches showed an increase of SI with a gradual increase of heating temperature. During heat in excess of water, the starch granules swell, the crystalline structure is broken, and consequently the hydroxyl groups of the amylopectin molecules and mainly amylose bind to water by hydrogen bonds, resulting in increased solubility (Punia, 2020). BRS305 genotype, showed the lowest SI (Figure 4b), which can be attributed to the reduction of leached amylose (Sudheesh et al., 2021). CMSXS180 and BRS308 showed similar solubility values at 65°C. Above this temperature, CMSXS180 and BRS308 presented higher SI compared to sorghum starches, which may be attributed to higher amylose content compared to BRS305.

3.7 | Pasting properties

The paste temperature (PT) in which starches start to swell, shows a sudden increase of viscosity at the beginning of the RVA reading (Figure 5). In addition, PT was similar for all starches, ranging from 74.0°C (CMSXS180), 74.4°C (BRS308 and maize), and 74.5°C (BRS305). Low values of PT, for example, below 70°C indicates less swelling and rupture resistance (Atuobi et al., 2011). PT of sorghum and maize were comparatively higher than other starches such as cassava, wheat, barley, waxy-maize, and rye had comparatively with respective values of 64.0, 54.0, 51.5, 62.0, and 57°C (Atuobi et al., 2011).

The CV was very low for all samples (Figure 5). BRS305 showed the greatest value of 64.0 cP. Low CV values indicate no contamination of other components such us soluble fibers during starch extraction. PV at 95°C was higher for sorghum BRS305 (4602.0 cP) followed by cultivars BRS308 and CMSXS180 with 4097.5 and 4086.5 cP, respectively. The lowest values of PV were presented by maize starch, 2389.0 cP. PV indicates the water binding capacity of the starch and PV is the equilibrium of swell and starch leaching of amylose (Cui, 2005).

The mV values were 1404.0, 1245.5, 1334.5, and 1300.0 cP for BRS305, BRS308, CMSXS180, and maize, respectively (Figure 5).The mV values are used to understand the stability of paste and calculated the breakdown (PV-mV), as well as the retrogradation viscosity, both used to measure functional properties of granules ruptures and polymer alignment (Cui, 2005).

All sorghum starches presented a disturbance of viscosity flow after 14 min run (Figure 5), which may indicate an intermittent formation of entanglements of amylose molecules due to constant shear, thus forming a firm gel that sooner broke and formed again. The gel formation was probably hampered by the formation of complexes between amylose and other residual macromolecules such as lipids and proteins. According to Wang et al. (2020), this could be also caused by some binding capacity of amylopectin to reassociate, which is known to be weak than amylose. Moreover, amylose is the main starch molecule that forms complexes that are formed during heat shear processing protocols (above 90°C) leading to structures of binary complexes (starch-lipid and/or starch-protein) and ternary

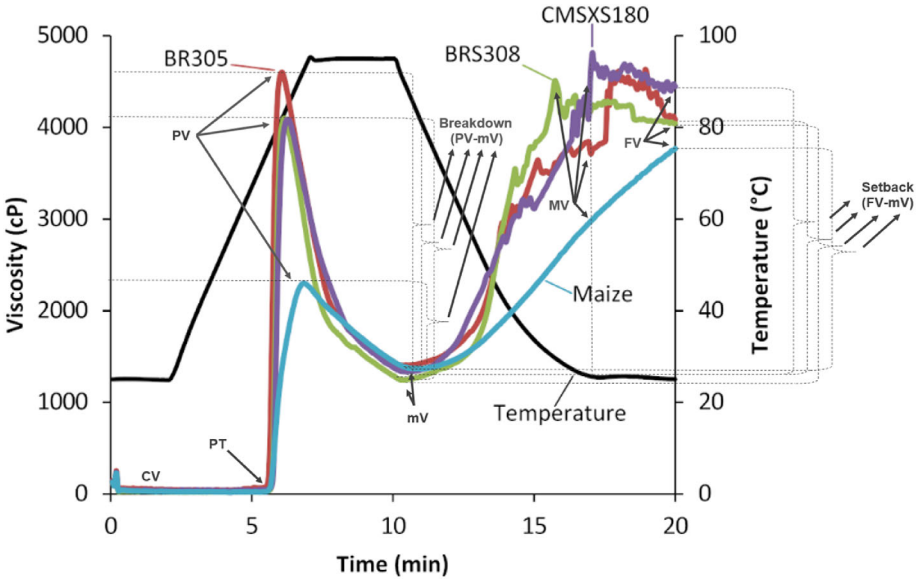

FIGURE 5 Pasting properties: PT, paste temperature; CV, cold viscosity at 25°C; PV, peak viscosity at 95°C; mV, minimum viscosity after heating; MVC, maximum viscosity at cooling; FV, final viscosity; breakdown = PV–mV, breakdown viscosity; setback = FV–MV, setback viscosity; of sorghum starch genotypes (BRS305, BRS 308, and CMSXS180) and maize starch

complexes (starch-lipid-protein) upon cooling hence reducing swelling power, starch solubility, and hindering retrogradation.

The lower swelling power and solubility index and weak gel formation were observed in the different sorghum genotypes (BRS305, BRS308, and CMSXS180) submitted to heating and cooling in this study, when compared to commercial maize starch. Although sorghum starches have less stability in the final gel formation, the FV of sorghum genotypes varied from 4042.0 cP (BRS308) to 4444.5 cP (CMSXS180; Figure 5), showing high retrogradation functionality, demonstrating that the difficulty in gel formation was not great enough to generate the lowest viscosity, while maize presented 3767.5 cP of FV, which was less than shorgum starches.

3.8 | Thermal properties

Sorghum genotype, BRS305 had the lowest value of To (59.63°C) and Tp (68.26°C), indicating that melting occurred at lower temperatures than other starch granules (Figure 6). Also, BRS305 showed the highest value of gelatinization temperature range (17.27°C) compared to other sorghum genotypes, which indicates a higher degree of heterogeneity may be caused by amylopectin chain located in the amorphous lamella regions of the starch granules (Li et al., 2021). Consequently, the conclusion thermal event (complete cooking or melting of starch crystallites), Tc (76.90°C) of, was the lowest than other starches.

BRS308 and CMSXS180 genotypes showed similar values of the thermal event (Figure 6). The sorghum starches BRS308 and CMSXS180 presented the highest To (67.17 and 66.37°C, respectively), Tp (74.58 and 73.95°C, respectively), and Tc (83.97 and 83.47°C, respectively) and lower gelatinization enthalpy (9.00 and 9.64 J/g, respectively). While maize starch showed lower To (64.53°C), Tp (72.74°C), and Tc (81.78°C) and highest ΔH (11.80 J/g), which may indicates reduced stability of their crystallites and smaller length of amylopectin chains (Fredriksson et al., 1998).

Similar findings, To (67.70°C), Tp (73.7°C), Tc (80.90°C), gelatinization temperature range (13.20°C), and gelatinization enthalpy (9.70 J/g) were found by Li et al. (2021) in native sorghum. The authors suggested an influence of amylose content on the integrity of starch granules leading to greater melting enthalpy, which was not observed in this present work.

3.9 | Principal components analysis

The PCAs explained 90.5% of the total variability (Figure 7a,b), resulting in a small loss of information. As reported by Barroso and Artes (2003), the PCA had to explain at least 70% of the total variability to retain enough information.

The PCA was performed using all samples (BRS305, BRS308, CMSXS180, and maize starch; Figure 7a), while the second PCA (Figure 7b) was developed using the physical and chemical variables: relative crystallinity, amylose content, solubility index, swelling power, pasting, and thermal properties. In addition, in Figure 7b, very distant (highest and lowest) values in each physical–chemical variable generated big vectors with greater influence on PCA, they were graphically illustrated by red and orange colors; while close or similar (highest and lowest) values in each physical–chemical had low contribution on differentiation of the samples and generated medium and small vectors, graphically illustrated by yellow and blue colors, respectively.

In relation to the PCA of the samples (Figure 7a), it is possible to mention that CMSXS180 (highlighted in blue) presented low

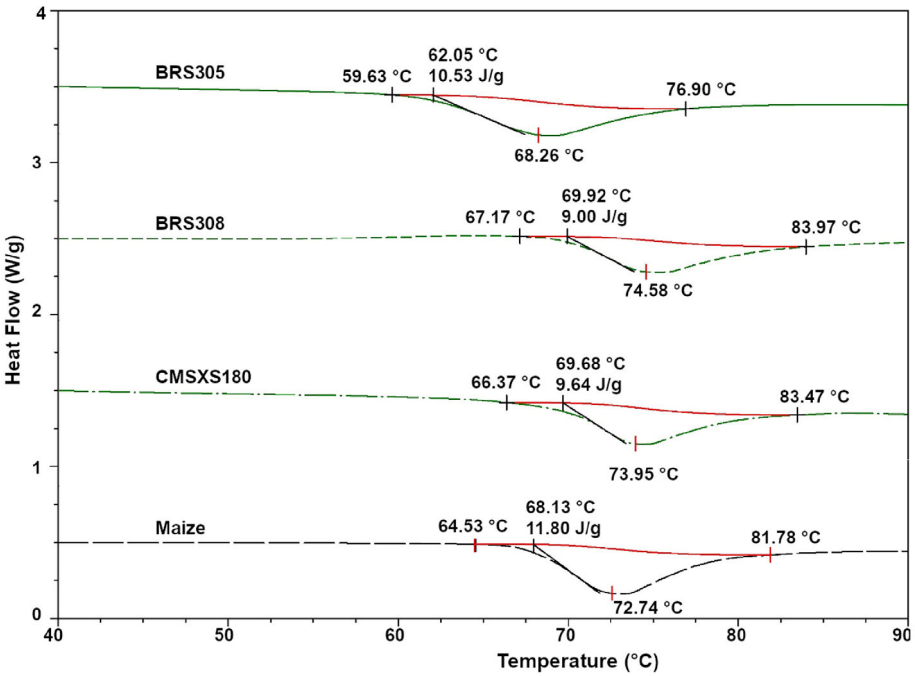

FIGURE 6 DSC thermograms of sorghum starch genotypes (BRS305, BRS308, and CMSXS180) and maize starch

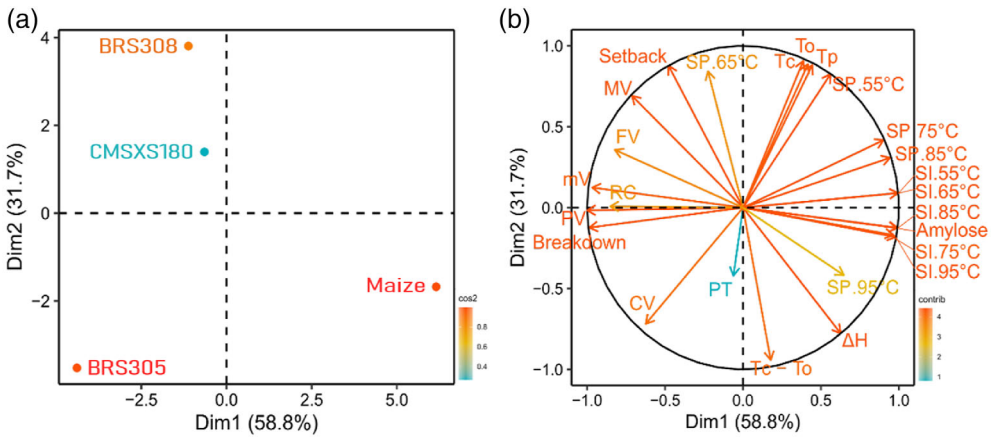

FIGURE 7 PCA from sorghum and maize starches: (a) PCA of samples (BRS305, BRS308, CMSXS180, and maize starches), (b) PCA of physical and chemical properties (relative crystallinity (RC); amylose content; solubility index (SI); swelling power (SP); pasting properties: PT, paste temperature; CV, cold viscosity at 25°C; PV, peak viscosity at 95°C; mV, minimum viscosity after heating; MVC, maximum viscosity at cooling; FV, final viscosity, breakdown and setback and thermal properties: To, onset; Tp, peak; Tc, conclusion; Tc–To, gelatinization temperature range and; ΔH, calorimetric enthalpy)

differentiation. As mentioned in 3.3 and 3.6 sections, CMSXS180 presented the highest values of RC (30.4%) and FV (4444.5 cP), respectively, in addition the highest MV (4507.0 cP) and setback (4444.5 cP); as well as many intermediate values; and the lowest PT (74.00°C). Nonetheless, the resultant vector that determined the geographical position of the CMSXS180 (Figure 7a), generated by all variables in Figure 7b; for example: MV, setback (red vectors), RC and FV (yellow vectors) were not sufficient to cause a considerable difference in CMSXS180 (Figure 7a) compared to other samples.

On the other hand, the resultant vector that determined the geographical position of the BRS308 (orange, in Figure 7a) had differentiation between the samples. This differentiation was caused by the influence of vectors in Figure 7b, due to the highest To (67.17°C), Tp (74.58°C), Tc (83.97°C), SP 55°C (9.2 g/g), and SP 65°C (13.4 g/g; red vectors); some intermediate values; and the lowest values of mV (1245.5 cP), ΔH (9.0 J/g; red vectors), CV (32.5 cP), Tc–To (16.80°C; orange vectors); and SP 95°C (8.4 g/g; medium yellow vector) presented in BRS308.

BRS305 (red, in Figure 7a) had strong influence on sample differentiation; influenced by vectors in Figure 7b, with the highest values of PV (4602 cP), breakdown (3198 cP; red vectors), CV (64 cP), mV (1404 cP), Tc–To (17.27°C; orange vectors), and PT (74.50°C; blue vector). It also presented few intermediate values, and the lowest To (59.63°C), Tp (68.26°C), Tc (76.90°C), amylose content (22.86%), SI (55, 65, 75, 85, and 95°C: 0.02, 0.04, 0.05, 0.06, and 0.09, respectively), and SP (55, 75, and 85°C: 5.10, 11.07, and 13.83 g/100 g, respectively; orange vectors), and SP 95°C (6.40 g/100 g; yellow vector).

Ultimately, maize starch (red, in Figure 7a) had high differentiation, as well as BRS305, caused by the vectors in Figure 7b with the highest values of ΔH (11.8 J/g), amylose content (24.50%), SI (55, 65, 75, 85, and 95°C: 0.06, 0.09, 0.13, 0.19, and 0.22, respectively), SP (75 and 85°C: 20.30 and 22.96 g/100 g, respectively; red vectors), SP 95°C (22.72 g/100 g; yellow vector); few intermediate values; and lowest values of PV (2389.0 cP), breakdown (1089.0 cP), MV (2967.0 cP), setback (2467.5 cP; red vectors), FV (3767.5 cP), RC (24.27%), and SP 65°C (6.09 g/100 g; orange vectors).

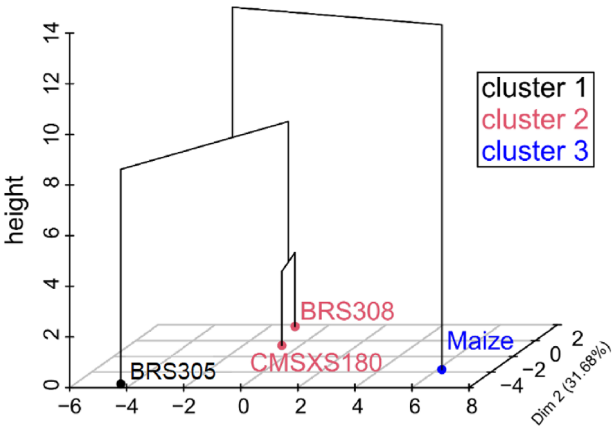

FIGURE 8 Hierarchical clustering from PCA of sorghum starch genotypes (BRS305, BRS308, and CMSXS180) and maize starch

3.10 | Hierarchical clustering on principal components between samples

By applying PCA, it is suggested three groups (Figure 8) of starches based on their physical and chemical similarities. BRS308 and CMSXS180 formed the cluster 2, these samples presented the greatest similar properties. BRS305 (cluster 1) formed a group lonely, due to its characteristics, finally, maize starch (cluster 3) with the least similarity against other samples, indicating the difference related to the physical and chemical parameters analyzed between sorghum and commercial maize starches.

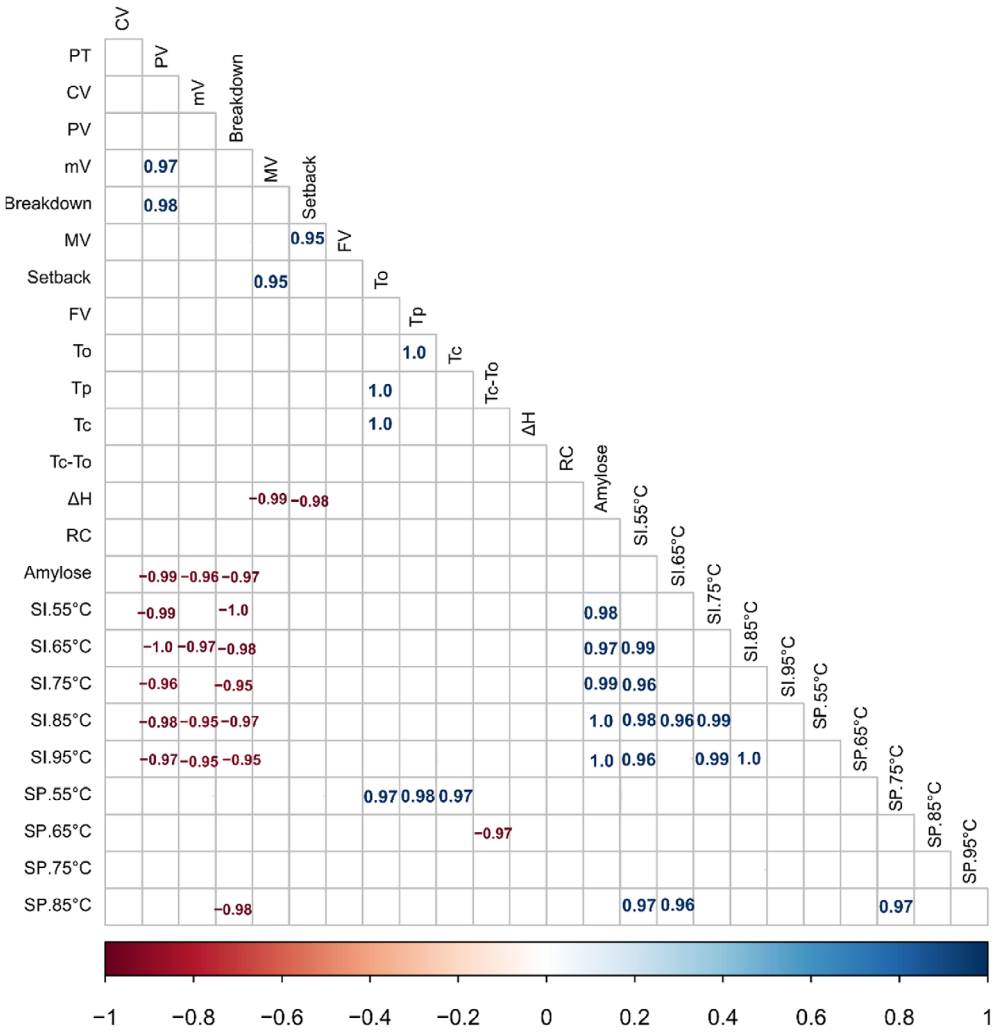

FIGURE 9 Correlogram for the physical and chemical parameters (relative crystallinity (RC), amylose content, solubility index (SI), swelling power (SP), pasting properties: PT, paste temperature; CV, cold viscosity at 25°C; PV, peak viscosity at 95°C; mV, minimum viscosity after heating; MVC, maximum viscosity at cooling; FV, final viscosity, breakdown and setback, and thermal properties: To, onset; Tp, peak; Tc, conclusion; Tc−To, gelatinization temperature range and; ΔH, calorimetric enthalpy). Only significant correlations are presented ($p < 0.05$)

3.11 | Pearson's correlation between physical and chemical properties of starches

Although there were different levels of interaction as shown in HCPC (Figure 8), some physical and chemical variables showed either strong positive or negative correlation among themselves (Figure 9).

According to the scale of strengthens from Pearson's correlation coefficient, there were very high correlations ∼1 (value close to 1, there is a very strong positive correlation while value close to −1 has a very strong negative correlation; Teles et al., 2019). In this regard, there was found a positive and very strong correlation (Figure 9) between To and Tp; To and Tc; SI 85°C and amylose; SI 95°C and amylose; SI 95°C and SI 85°C ($r = 1$). The solubility index at 85 and 95°C also presented a positive correlation with amylose ($r = 1$).

The found correlations could be explained by an expected increase of solubility as the starch granules were submitted to high temperatures in excess of water. Water molecules easily form hydrogen bridges with amylose and amylopectin, exposing their hydroxyl groups, hence increasing starch molecules solubility (Ge et al., 2022).

When compared to the paste viscosity profile, amylose showed a strong negative correlation with PV, mV, and breakdown ($r = -0.99$, −0.96 and −0.97, respectively; Figure 9). In that respect, SI (55, 65, 75, 85, and 95°C) also had strong negative correlations with PV, mV, and breakdown, indicating a strong influence of amylose on reducing the peak of paste viscosity and the solubility index.

From the correlogram (Figure 9), it can be seen a negative correlation (−0.96 to −1.00 at different temperature ranges of SI) between PV and SI values. In other words, an increase of peak viscosity lead to low soluble content in water, which in practical terms and, in accordance to the review of Zhu (2014), sorghum starch could be indicated for the production of noodles. The author mentioned that for a better noodle quality, would be required a less cooking loss (low SI) and would provide better noodle elastic property with correlated with high paste viscosity.

4 | CONCLUSIONS

No chemical treatment was used to isolate sorghum starches, and being possible to obtain high carbohydrate levels and a "clean label starch." The CMSXS180 genotype was highlighted due to protein, lipids, and carbohydrate levels similar to maize starch. Although X-ray diffraction profile and particle size distribution of all sorghum starches

were similar to maize starch, PCA and HCPC showed differences for the physical and chemical variables, and samples analyzed. In Pearson's correlation, the negative interaction among variables PV and SI contributing to sorghum starches being used in starch noodles. The use of each sorghum genotype can be directed in tests to evaluate the texture preference by consumers. Therefore, these starches could be a potential byproduct of bioactive compounds extraction from sorghum; in order to supply the starch demands and encouraging the study of starches not yet commercially established, such as sorghum starch.

AUTHOR CONTRIBUTIONS

Thaís Barbosa dos Santos: conceptualization, data curation, formal analysis, investigation, methodology, software, validation, writing-original draft, and revision; **Raimundo da Silva Freire Neto:** conceptualization, data curation, formal analysis, investigation, and revision; **Nathalia Ferreira Collantes:** conceptualization, data curation, formal analysis, investigation, methodology, and revision; **Davy William Hidalgo Chávez:** data curation, formal analysis, methodology, software, validation, writing-original draft, and revision; **Valéria Aparecida Vieira Queiroz:** conceptualization, project administration, resources, and revision; and **Carlos Wanderlei Piler de Carvalho:** conceptualization, project administration, resources, supervision, and revision.

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CONFLICT OF INTEREST

Authors declare that they have no conflict of interests.

DATA AVAILABILITY STATEMENT

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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