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Effect of planetary ball milling on physicochemical and morphological properties of sorghum flour

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ABSTRACT

Planetary ball milling was applied to white sorghum flour with the aim of modifying its thermal, structural and morphological properties. Median particle size decreased from 57.2 µm to 20.8 µm with increasing milling energy. Particle size-energy models indicated that only part of the energy was used for developing new surfaces. Abrasion of starch granules could be observed by SEM and the increment of damaged starch levels. Gelatinization temperatures measured by DSC were not affected by the process (T_p average 73.4±0.4 °C); yet, gelatinization enthalpy (ΔH) and crystallinity degree (determined by WAXS) decreased with increasing milling energy from 5.54 J/g and 28% to 2.98 J/g and 17.0%, respectively. Then, some pasting parameters significantly changed: final viscosity (from 3947 to 3535 cP) and, consequently, setback (from 2339 to 1896 cP). Planetary ball milling significantly changed the functional properties of sorghum flour and suggested that this method is an alternative to widen sorghum flour food applications.

KEYWORDS

Sorghum flour, planetary ball-milling, modeling, thermal properties, functional properties
1. Introduction

Consumers are increasingly demanding food produced from sustainable and non-GMO crop. Then sorghum is ideal since its hybrids can grow in salty or sandy soils with low amounts of water, fertilizer and pesticides. Although sorghum is the fifth cereal crop produced worldwide, most of African and Asian production is consumed directly as food and the production of USA and Latin America is used as animal feed (Bedford et al., 2017; Léder, 2004). The nutritional properties of sorghum lie in its high amount of starch, phenolic acid and flavonoid; nevertheless, this cereal is not important in the commercial food systems (Hager, Wolter, Jacob, Zannini & Arendt, 2012). The most usual method to produce sorghum flour around the world involves partial dehulling followed by dry milling, since this allows a low-fiber product and avoids the drying process after the wet milling procedure (Sun et al., 2014). Also, the wastewater treatment should be considered despite the high efficiency and the great number of alternatives for bioreactors (Sepehri and Sarrafzadeh, 2018).

The improvement of sorghum flour properties could allow the introduction of this cereal to the food industry and, therefore, to human diets around the world. In this sense, there has been a growing interest over the past few years in physical methods to enhance the functional properties of flour as they increase their applications without resorting to chemical reagents. This turns them into environmentally friendly methods with wider acceptance by consumers. Among these treatments, recent modifications were carried out with high hydrostatic pressure, ultrasound, pulsed electric field and microwaves (Ashogbon and Akintayo, 2014). In turn, simpler methods like particle-size classification and fine grinding were the most common modification strategies to improve gluten-free products (Gómez and Martínez, 2016).

Planetary ball milling is an innovative technology with successful applications in pharmaceutical and nanomaterial industry. However, only few advances were made in food development. This technology can improve material characteristics without producing hazardous materials. Ball
54 milling applied to flour can modify starch structure and behavior (Delogu, Gorrasi & Sorrentino, 2017; M. Loubes & Tolaba, 2014; Shan et al., 2016). Particularly, milling processes like abrasive milling and planetary ball milling have modified crystallinity and, consequently, water absorption, thermal parameters and rheological behavior of amaranth starch-enriched fraction (Roa, Santagapita, Buera & Tolaba, 2014). Liu et al. (2011) and González et al. (2018) found that intense grinding in wheat starch granules decreased enthalpy and temperature of gelatinization and increased soluble material loss as a result of slight depolymerization of amylose and amylopectin.

Grinding modeling aims at building mathematical relationships between feed and product particle sizes that allow mill and grinding system designs. From this, different design and modeling methods have been developed to achieve effective results by simple methods (Austin & Concha, 1994; Mio, Kano & Saito, 2004).

The aim of the present work was to evaluate the effects of planetary ball milling on the physicochemical and morphological properties of sorghum flour and model its relationships.
2. Materials and methods

2.1. Materials

Partially dehulled white sorghum flour from Pannar-8706 W hybrid grown in central region of Argentina was provided by Amylum S.A. (Córdoba, Argentina). The moisture content of the flour was 11.4% and its composition on a dry basis was: lipid 5.4%, ash 1.2%, protein 7.9% and total carbohydrates 85.5% (76.0% starch).

All chemicals were analytical reagent grade and all solutions were prepared by using deionized water.

2.2. Sorghum flour ball milling

The flour was dry ground using a planetary ball mill (PM-100, Retsch, Germany) with five times mass of zirconium oxide balls (5 mm diameter). The jar rotation speed was set at 400 rpm with a change in direction of rotation every 30 seconds and 40-minute breaks every 10 minutes of treatment (to prevent temperature from exceeding 55°C). The grinding energy was set at five levels, considering that the milling power is delivered practically constant for certain conditions, the samples differs in treatments time (Table 1). Because the energy was applied to sample and balls, previous calibration with an empty jar were carried out.

After each treatment moisture content was determined according to the standard method (AOAC, 2000), varying from 11.2% to 8.9%, for 0.26 kJ/g and 5.84 kJ/g, respectively.

2.3. Particle-size distribution

Particle-size distribution of samples was measured with a laser particle-size analyzer (LA-960, Horiba Instruments, USA). Samples were analyzed with the liquid sampler and both circulation velocity and agitation were set at level 10 (out of 15). The sample refractive index was 1.54 and 1.33 for the dispersant. Size distribution parameters $D_{v50}$ (median), $D_{v10}$ and $D_{v90}$ (particle
93 diameters where cumulative volume of particles are 10% and 90%, respectively, mode and
94 mean were measured and expressed in volumetric base. Span was calculated as \((Dv_{90} - Dv_{10})/ Dv_{50}\). Measurements were carried out in triplicate.

2.4. Milling modeling

The energy supplied per unit of processed mass to produce a small change in particle size can
97 be expressed as a function of the power law type, which is a general interpretation of several
98 laws presented by different authors and known as the general law of milling (Snow, Allen, Ennis
99 & Litster, 1999) (Eq. 1).

\[
\frac{dE}{dx} = -\frac{C}{x^n} \quad (1)
\]

where \(E\) is the applied energy, \(x\) is a selected particle size dimension, and \(n\) and \(C\) are constants
102 related to the material. Size parameter mean, median, \(Dv_{10}\) and \(Dv_{90}\) data were fitted to the
103 integrated form of Eq. 1 (Eq. 2).

\[
E = \frac{C}{1-n} (x_f^{1-n} - x_i^{1-n}) \quad (2)
\]

Where \(x_i\) are the initial mean, median, \(Dv_{10}\) or \(Dv_{90}\) and \(x_f\) represents size parameter at different
107 treatment times.

Fitness of fracture to a first-order kinetics was also evaluated with the model proposed by Mio,

\[
\frac{D_t}{D_0} = \left(1 - \frac{D_i}{D_0}\right) e^{-Kt} + \frac{D_i}{D_0} \quad (3)
\]

where \(D_i, D_0\) and \(D_t\) are the values of the mean at time \(t\), time 0 and milling limit time, respectively, 112 and \(k\) is the fracture velocity constant.

2.5. Scanning electron microscopy (SEM)
A scanning electron microscope (Sigma, Carl Zeiss, Germany) was used to evaluate the morphology of flour particles, mainly starch granules. Samples were coated with gold and images were taken at an electron acceleration of 3 kV under high vacuum conditions with main and secondary detectors at different magnifications.

2.6. Damaged starch

Damaged starch (DS) levels were estimated following the methods 76-30A and 80-60 (AACC, 2000). The amount of DS was expressed as percentage of starch subject to enzymatic hydrolysis in the sample, as the mean of two duplicates.

2.7. Water absorption

Water absorption (WA) of flour was determined following Yousif et al. (2012) procedure. Briefly, samples (500±5 mg db) were suspended into water (6 mL), incubated at 25 °C for 30 minutes and then centrifuged, and the supernatant was discarded. The WA determination was performed in triplicate and the mean was expressed as g of absorbed water by g of sample.

2.8. Thermal properties

Thermal properties were evaluated with a differential scanning calorimeter (DSC 823e, Mettler Toledo, Switzerland) controlled by STARe software. Aluminum pans of 100 μL were filled with 10 mg db of flour and 20 μL of deionized water, and then hermetically sealed. The heating ramp was set with a rate of 10 °C/min from 20 to 120 °C. The parameters assessed in the thermograms were: onset (To), peak (Tp) and conclusion temperatures (Tc), gelatinization enthalpy (ΔH, J/g of flour) and peak width and height. Thermal properties were evaluated in duplicate and the results expressed the mean.

2.9. Pasting properties
Pasting parameters were determined with a Rapid Viscosity Analyzer (RVA 4500, Perten Instruments, Australia) 10% w/w of flour in water. The temperature profile consisted in a holding period at 50 °C (1 min), heating to 95 °C (4 min), holding at 95 °C (2.5 min) and finally cooling to 50 °C (3 min) and maintaining (2 min). Stirring speed was 960 rpm for the first 10 s and 160 rpm until the end of the assay. Thermocline software (V 3.15, Perten Instruments, Australia) was used to control the device and acquire the data to obtain pasting parameters: pasting temperature (PT), peak viscosity (PV), trough viscosity (TV), final viscosity (FV), breakdown (BD=PV−TV) and setback (SB=FV−TV). Pasting profile determination were carried out in duplicate.

2.10. Wide-Angle X-ray Scattering

WAXS was carried out with an X-ray diffractometer (PW-1800, Philips, USA) using 40KV and 30mA radiation to scan from 2 to 30º with time steps of 0.02º/2.5 s. The crystallinity degree (CD) is the ratio of crystalline/amorphous phase and was calculated with Peakfit software v4 (Jandel Scientific, USA) through diffractogram deconvolution (Ribotta, Cuffini, León & Añon, 2004). The diffractograms were obtained in duplicate for each sample.

2.11. Statistical analysis

Analyses of variance (ANOVA, multiple comparison test by DSG, α=5 %) were performed using InfoStat software (Version 13p, Di Rienzo et al. 2011) and artwork was made using Excel (2016 version, Microsoft).
3. Results and discussions

3.1. Particle-size distribution changes and modelling

Particle-size distribution of sorghum flour samples was significantly modified by planetary ball milling. Figure 1 shows the particle-size distribution of each sample, where untreated flour (0) has two particle populations forming a bimodal curve with peaks at 18 and 152 µm. Treatments produced a new population of particles with a local maximum at around 100 µm, which remained relatively invariant with the process time at approximately 5% v/v until maximum treatment time. Likewise, the 152 µm decreased and 18 µm increased with applied milling energy with highly milled sample (5 = 5.84 kJ/g) showing a predominant particle population with a peak at 18 µm.

The starch fraction was isolated from the untreated flour following the procedure of Pérez Sira and Lares Amaiz (2004). Sorghum starch fraction showed a monodisperse distribution with a mean of 16 µm, which indicated that the milling treatment breaks the flour particles into separated starch granules.

The mean particle size decreased with the increase in applied energy from 86.6 µm to 36.0 µm (Table 1). In turn, the span (particle size dispersion) oscillated slightly around 3.3 indicating a very wide dispersion in all samples. It can also be observed that the median was considerably lower than the mean for all cases, highlighting their asymmetry. Roa et al. (2014) found similar results for amaranth flour treated in the same mill, although in that case the span was reduced slightly but significantly.

Modeling of the milling procedure helped to understand the process and settle a base to its scale-up. The mean, median, $D_{10}$, and $D_{90}$ were fitted to Eq. 2 and mean obtained the best regression ($R^2 = 0.98$). The $n$ value from this equation is related to the new surface generation efficiency during the milling process (Roa et al., 2014a), and when $n = 2$ the equation correspond to the Rittinger model where energy is proportional to the new surface produced (Snow et al.,...
The value found was 2.44, indicating that only a fraction of the applied energy was used to generate new surfaces; hence, another part was probably dissipated as thermal energy, elastic collisions with fiber particle or particle abrasion. The proportionality constant of the Eq. 2 (C) obtained was 1954 kJ g\(^{-1}\) µm\(^{-n}\). This parameter varies with milling conditions, ball loading and sizes, and indicates how many energy is used to produce the size reduction and new surface creation, therefore an investigation of the optimal milling conditions should aim to minimize this value (Snow et al., 1999; Xu and Wang, 2017).

The experimental data were also adjusted by Eq. 3, reaching a R\(^2\) of 0.98 which proved the first-order kinetics with a K of 0.014 min\(^{-1}\) and D\(_l\) of 27.1 µm. These values indicated a rapid decrease of the normalized media (D\(_l\)/D\(_0\)) at the milling initial stage and a stabilization close to the mean of the highly treated sample. Mio et al. (2004) reported similar behavior in planetary ball milling for several devices and milling conditions. These authors also stated a close relationship between K and specific milling energy particularly useful for simulation and scale-up.

The good fit found for Eq. 3 should be highlighted, since, in this case, the samples do not comply with some of these models assumptions: the start material should be monodisperse and significantly larger than the resulting ones; in addition, most models were developed to weigh particle distributions rather than volumetric (Verkoeijen, A. Pouw, M. H. Meesters & Scarlett, 2002).

### 3.2. Morphologic characterization

The morphology of untreated and highly treated flour is shown in Fig. 2. In the microscopic images of sorghum flour polyhedral and oval starch granules, globular protein bodies and fiber particles could be observed.
From the images acquired by SEM it was possible to observe the breakage of flour particles and the increase of the modification produced in the surface of the starch granules. The planetary milling process produced not only the fracture of flour particles, it also changed starch granule and, therefore, structural, absorptive, thermal and rheological properties. These transformations are summarized in Tables 2 and 3 and will be discussed in the next sub-sections.

Untreated sample (Fig. 2-0A) shows large particles (close to 100 µm), not founded in highly treated sample (Fig. 2-5A). Endosperm fragments, starch granules, fiber particles and protein bodies forming a conglomerate can be observed at a higher magnification (Fig. 2-0B), as described by other authors (Belton and Taylor, 2002; Hager et al., 2012). However, after the most extensive treatment (Fig. 2-5B), granules were no longer attached to each other, no large fiber particles were observed and protein bodies were disintegrated.

In turn, starch granule showed evident damage by planetary mill grinding (Figure 2-5C and D): surface changed from smooth to rough, most of the granules being distorted. Similar results were described by Barrera et al. (2013) for wheat starch treated with a disc mill and Liu et al. (2011) for ball milled maize, highlighting that starch damage could be observed in granules from different sources and through diverse milling devices.

### 3.3. Damaged starch

The milling models focused on the mechanisms of division and fracture, but not on abrasion. In this case, since the treated material is a cereal flour, a large part of the abrasion effect could be known by investigating the damaged starch content. The damaged starch (DS) is quickly hydrolyzed by α-amylases representing an important transformation produced by ball milling since damaged starch generally influences water absorption, thermal properties and rheological behavior (Barrera et al., 2013a; León et al., 2006).
The initial level of DS (5.2%) was produced by the industrial hammer mill during flour production process and planetary ball milling increased starch damage up to 17.9% (Table 2).

The effect of the applied energy on the damaged starch content was investigated. These parameters were adjusted to an exponential equation (Eq. 4) with a high fitting coefficient ($R^2=0.987$). This fitting indicated that the stronger the treatments, the less energy was needed to generate the same change in the amount of damaged starch. For example, between treatments 1 and 2, 0.73 kJ/g was needed to produce a difference of 0.7% of DS, whereas between points 4 and 5 the application of 2.91 kJ/g produced an increase of 7.8% in DS.

$$DS(\%) = 5.4 \cdot e^{0.2 \cdot E} \quad (4)$$

3.4. Water absorption

The water absorption results were summarized in Table 2 and a slight increase in WA was observed with the application of higher milling energy. The highly treated sample showed the highest water retention capacity, 15% more compared with the untreated flour. This increment in the water intake could be explained by the damaged starch generated during the milling process and was reflected in a positive correlation between these parameters ($r=0.99, p<0.05$). The damaged granules possess greater affinity for water and depending on its botanical source (which determines the granule size, pores and shape) a damaged granule could absorb water up to 3 times than native ones (Liu et al., 2011; Roa et al., 2014b).

In turn, Loubes and Tolaba (2014) reported a strong increase of WA in planetary milled rice flour probably related to higher rotational speed and final temperature reached in the processing compared to those used in this study.

3.5. Thermal properties
The gelatinization enthalpy ($\Delta H$) decreased with increasing milling energy (Table 2), in agreement with results published by other authors for wheat, amaranth and rice samples (Barrera, León & Ribotta, 2012; M. Loubes & Tolaba, 2014; Roa, Baeza & Tolaba, 2015).

It was previously demonstrated (Barrera et al., 2013a) that highly damaged starch granules absorb water quickly and leach not only amylose but also amylopectin, giving the ability to hydrate spontaneously in cold water, such as pre-gelatinized starch. Due to these changes damaged starch and the starch granules fragments partially contribute to the endothermic gelatinization energy (Barrera et al., 2012). The reduction of $\Delta H$ and peak height values (Table 2) found in this study could be attributed to partial destruction of the starch structure.

On the other hand, gelatinization temperatures were not significantly affected by planetary milling ($T_o = 67.2\pm1.1 \, ^\circ C$, $T_p = 73.4\pm0.4 \, ^\circ C$ and $T_c = 80.4\pm0.4 \, ^\circ C$), according to studies reported by other authors for wheat starch with DS levels up to 23.8 % (Barrera et al., 2012). In turn, Roa et al. (2015) found only significant differences in $T_o$ for the most intensely treated samples by planetary grinding of amaranth starch (6.52 kJ/g of applied energy) and González et al. (2018) found a decrease in $T_p$ for planetary ball milled rice starch.

### 3.6. Crystallinity degree

Sorghum flour showed a crystalline structure type A, characteristic of cereals with strong refractive peaks at 15° and 23° and a double at 17° and 18°, and no displacement of the peaks was detected with the treatment (Figure 3). In addition, WAXS allowed determining crystallinity loss during the planetary grinding process, in which CD decreased from 28.8 % to 17.0 % as the treatment became more intense (Table 2). This loss in crystallinity and preservation of the diffraction pattern was also reported by other authors for flour and starch with high content of damaged starch, determined by x-ray diffraction and by NMR (nuclear magnetic resonance) (Liu et al., 2011). An intense ball milling treatment produced changes in the crystalline phase and in
the crystallinity of polymeric materials which could led to its total amorphization (Delogu et al., 2017). In this sense, Anzai et al. (2011) found that ball milled potato starch loss entirely its crystalline structure and become amorphous after 17 hours treatment. Liu et al. (2011) reported the disappearance of all the diffraction peaks of maize starch treated during 2 h in a ball mill and reached 2.67% of CD after 3 h.

The gelatinization peak width showed a positive correlation with the damaged starch content \((r=0.85, p<0.05)\) and the peak height with the crystallinity degree \((r=0.88, p<0.05)\). These results indicate that the balls produce the partial disintegration of the crystals, formed mainly from amylopectin, causing reduction of gelatinization enthalpy as was explained above.

### 3.7. Pasting behavior

The heating of starch in excess of water produce the gelatinization of the granules (amylose lixiviation and granule swelling) which increase the system viscosity and its subsequent cooling results in a starch paste composite by a continuous phase of amylose chains with swollen granules dispersed in it. The rapid viscosity analyzer (RVA) allows the assessment of the viscosity evolution while the system was changing (Barrera et al., 2013a; Copeland et al., 2009).

Table 3 summarizes the pasting parameters of all samples and Fig. 4 shows the viscosity profile of untreated and highly treated samples. Regarding the pasting parameters, final viscosity, and consequently, setback showed significant differences between the samples. The PV and TV showed slight differences between samples and breakdown was similar in all samples. In turn, no differences were found for temperature and pasting time, exhibiting average values of 89.4±0.6 °C and 5.8±0.1 minutes, respectively. This indicates that at the beginning of the pasting curve, behavior was similar in all samples, but differed in the cooling stage.

In the pasting curve a second peak can also be observed during cooling stage, which could be ascribed to the presence of free fatty acids, following Zhang and Hamaker (2003, 2005). These
authors describe that samples stored for a certain time (2 months) show this behavior due to the degradation of the lipids present in the flour. In addition, starch-lipid and starch-protein-lipid complexes influence this behavior, varying with the content of fatty acids and the size of their aliphatic chain. In our work, all samples exhibited similar behavior, probably because they were stored during the same period of time and contained the same lipids.

The untreated sorghum flour sample showed a SB of 2339 cP that progressively decreased to 1896 cP for the maximum applied energy sample (5.84 kJ/g). Similar results were found by other authors (González et al., 2018; Z. Zhang, Zhao & Xiong, 2010).

Final viscosity depends on several factors, such as the degree of amylose leached and the volume and stiffness of the remaining granules. According to a previous work, higher starch damage produced weaker and greater disintegration of the granules, and consequently, less volume occupied by the disperse phase and more amylopectin in the continuous phase (Barrera et al., 2013a). In this sense, the study published by Fu et al. (2018) on the thermal and structural behavior of potato starch treated by ball grinding suggests that a small amount of remaining crystals after gelatinization prevents retrogradation.

The FV showed a negative correlation with the applied energy ($r=-0.83$, $p<0.05$) in agreement with the results reported by Loubes et al. (2018) which found that pasting parameters were negatively affected by planetary ball milling time and speed.
4. Conclusions
The planetary milling process affected the particle-size distribution of sorghum flour, reducing considerably its size at high levels of applied energy. In turn, planetary ball grinding significantly affected the thermal and morphological properties of sorghum flour due to the partial destruction of the starch granule structure. In this sense, the amount of damaged starch increased significantly with the application of energy, which increased water absorption, decreased the degree of crystallinity of the granule and hence decreased its gelatinization enthalpy. Consequently, these changes caused differences in the pasting profiles, showing lower final viscosities as the milling process became more intense. At the same time, the modelling results indicated that, during milling, only a fraction of the applied energy was used to generate new surfaces and that this followed a first-order kinetics. This shows that planetary grinding is a suitable method for the production of modified flour, thus avoiding the use of chemical reagents or water.

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441
7. Tables

Table 1. Experimental design and particle-size distribution parameters of planetary ball milled sorghum flour.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Milling time (min)</th>
<th>Energy (kJ/g)</th>
<th>Median (µm)</th>
<th>Mean (µm)</th>
<th>Dv&lt;sub&gt;90&lt;/sub&gt; (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>0</td>
<td>-</td>
<td>57.2&lt;sup&gt;d&lt;/sup&gt;</td>
<td>86.6&lt;sup&gt;f&lt;/sup&gt;</td>
<td>202.9&lt;sup&gt;f&lt;/sup&gt;</td>
</tr>
<tr>
<td>1</td>
<td>5</td>
<td>0.26</td>
<td>66.4&lt;sup&gt;f&lt;/sup&gt;</td>
<td>83.3&lt;sup&gt;e&lt;/sup&gt;</td>
<td>193.8&lt;sup&gt;e&lt;/sup&gt;</td>
</tr>
<tr>
<td>2</td>
<td>20</td>
<td>0.99</td>
<td>58.4&lt;sup&gt;e&lt;/sup&gt;</td>
<td>76.0&lt;sup&gt;d&lt;/sup&gt;</td>
<td>178.5&lt;sup&gt;d&lt;/sup&gt;</td>
</tr>
<tr>
<td>3</td>
<td>40</td>
<td>1.96</td>
<td>34.0&lt;sup&gt;c&lt;/sup&gt;</td>
<td>58.15&lt;sup&gt;c&lt;/sup&gt;</td>
<td>134.9&lt;sup&gt;c&lt;/sup&gt;</td>
</tr>
<tr>
<td>4</td>
<td>60</td>
<td>2.93</td>
<td>27.8&lt;sup&gt;b&lt;/sup&gt;</td>
<td>52.5&lt;sup&gt;b&lt;/sup&gt;</td>
<td>118.8&lt;sup&gt;b&lt;/sup&gt;</td>
</tr>
<tr>
<td>5</td>
<td>120</td>
<td>5.84</td>
<td>20.8&lt;sup&gt;a&lt;/sup&gt;</td>
<td>36.0&lt;sup&gt;a&lt;/sup&gt;</td>
<td>82.5&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

Means with different letters within the same column indicate significant differences among samples (p<0.05).

Dv<sub>90</sub> diameter corresponding to 90 % of cumulative volume
Table 2. Effects of milling energy on damaged starch (DS), thermal parameters (ΔH: gelatinization enthalpy) and crystallinity degree (CD) of sorghum flour.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>DS (%)</th>
<th>WA</th>
<th>ΔH (J/g)</th>
<th>Peak height</th>
<th>Peak width</th>
<th>CD (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (Control)</td>
<td>5.2</td>
<td>a</td>
<td>5.54</td>
<td>d</td>
<td>1.07</td>
<td>b</td>
</tr>
<tr>
<td>1 (0.26 kJ/g)</td>
<td>6.2</td>
<td>b</td>
<td>4.54</td>
<td>c</td>
<td>0.95</td>
<td>b</td>
</tr>
<tr>
<td>2 (0.99 kJ/g)</td>
<td>6.9</td>
<td>b</td>
<td>2.7</td>
<td>a</td>
<td>0.67</td>
<td>c</td>
</tr>
<tr>
<td>3 (1.96 kJ/g)</td>
<td>7.7</td>
<td>b</td>
<td>2.97</td>
<td>b</td>
<td>0.65</td>
<td>c</td>
</tr>
<tr>
<td>4 (2.93 kJ/g)</td>
<td>10.1</td>
<td>c</td>
<td>2.91</td>
<td>b</td>
<td>0.61</td>
<td>c</td>
</tr>
<tr>
<td>5 (5.84 kJ/g)</td>
<td>17.9</td>
<td>d</td>
<td>2.98</td>
<td>b</td>
<td>0.53</td>
<td>a</td>
</tr>
</tbody>
</table>

Means with different letters within the same column indicate significant differences among samples (p<0.05).
Table 3. Effect of planetary ball milling energy on pasting parameters (RVA) of sorghum flour.

<table>
<thead>
<tr>
<th>Treatment</th>
<th>PV (cP)</th>
<th>TV (cP)</th>
<th>BD (cP)</th>
<th>FV (cP)</th>
<th>SB (cP)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0 (Control)</td>
<td>2145</td>
<td>1608</td>
<td>537</td>
<td>3947</td>
<td>2339</td>
</tr>
<tr>
<td>1 (0.26 kJ/g)</td>
<td>2114</td>
<td>1572</td>
<td>542</td>
<td>4057</td>
<td>2485</td>
</tr>
<tr>
<td>2 (0.99 kJ/g)</td>
<td>2241</td>
<td>1685</td>
<td>556</td>
<td>3813</td>
<td>2128</td>
</tr>
<tr>
<td>3 (1.96 kJ/g)</td>
<td>2234</td>
<td>1663</td>
<td>571</td>
<td>3592</td>
<td>1929</td>
</tr>
<tr>
<td>4 (2.93 kJ/g)</td>
<td>2253</td>
<td>1670</td>
<td>565</td>
<td>3584</td>
<td>1914</td>
</tr>
<tr>
<td>5 (5.84 kJ/g)</td>
<td>2212</td>
<td>1639</td>
<td>573</td>
<td>3535</td>
<td>1896</td>
</tr>
</tbody>
</table>

Means with different letters within the same column indicate significant differences among samples (p<0.05). PV, peak viscosity; TV, trough viscosity; BD, breakdown (PV-TV); FV, final viscosity; SB, setback (FV-TV).
Fig. 1. Volume size distribution of planetary ball milled sorghum flour as a function of milling energy (0 = no treatment, 1 = 0.26 kJ/g, 2 = 0.99 kJ/g, 3 = 1.96 kJ/g, 4 = 2.93 kJ/g, 5 = 5.84 kJ/g).
Fig. 2. SEM images of untreated sorghum flour (0 = no treatment) and highly milled sample (5 = 5.84 kJ/g).
Fig 3. X-ray diffraction pattern (WAXS) of planetary ball milled sorghum flour as a function of milling energy (0 = no treatment, 1 = 0.26 kJ/g, 2 = 0.99 kJ/g, 3 = 1.96 kJ/g, 4 = 2.93 kJ/g, 5 = 5.84 kJ/g).
Fig. 4. Pasting profile of planetary milled sorghum flour (0 = no treatment and 5 = 5.84 kJ/g)
Highlights

Planetary ball milling decreased the particle size of sorghum flour

Milling energy increased damaged starch content and decreased final viscosity and setback

Gelatinization enthalpy and crystallinity degree decreased with increasing milling energy