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Effect of ball milling energy on rheological and thermal properties of amaranth flour

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Abstract

Pearled amaranth grains obtained by abrasive milling were processed by planetary ball milling to produce starch-enriched flours. The influence of milling energy on rheological and thermal behavior of starch-enriched amaranth flour dispersions and stability during refrigerated storage was investigated. The rheological behavior of flour dispersions was determined using a rotational viscometer, while enthalpy change was determined by differential scanning calorimetry. The power law model was found to be suitable in expressing the relationship between shear stress and shear rate. All flour dispersions showed a pseudoplastic behavior. However this character decreased with the storage being dependent on flour concentration and milling energy. Several characteristics of the flour dispersions could be explained: decrease of the consistency coefficient and increase of the flow index with the increasing milling energy; decrease of gelatinization enthalpy showed the loss of crystalline structure due to ball milling, as well as increased stability of the flour dispersions during storage. It was observed, that the stability changed with the concentration of amaranth flours. Thus, more stable dispersions were obtained as the flour concentration increased. The highly milled sample was the most stable sample during the storage. To conclude, a more suitable performance to develop liquid food could be obtained using modified amaranth flour by the application of ball milling procedure.

Keywords: ball milling; amaranth; pseudoplastic behavior; viscosity.

1. Introduction

Over many years the traditional grains has been essential food for mankind. Recently the use of pseudocereals such as amaranth and quinoa has increased as a good raw material for the production of pasta, breakfast food, extruded and expanded products; bread and bakery goods and soup, because of its nutritional, processing and storing characteristics (Bodroza-Solarov et al 2008). Several researchers achieved good results in the preparation of pasta and baked goods by mixing amaranth flour and cassava starch (Fiorda et al. 2013; Saenz-Penella et al. 2012).

The knowledge of rheological properties of flour dispersions is important to control the process conditions, design flow systems and estimate texture of foods. In the same way, the effect of temperature on rheological properties might be documented because a wide range of temperatures is encountered during the process and storage of food dispersions containing starches (Rao 1999a). Therefore, studies on rheological properties of flour dispersions are very important for the purpose of producing products with the desirable qualities. Wang et al (1999) and Bhattacharya (1994, 1996) have studied the rheological properties of rice and maize flour dispersions, respectively. They found that the steady shear of rheological properties of dispersions, depend on concentration, temperature and variety. Consequently, the modification of the rheological properties in flour could increase its industrial use.

In a recent paper, we analyzed the effect of planetary ball-milling on amaranth flour characteristics such as particle size distribution, hydration properties and crystallinity degree. Using X-ray diffraction and FT-IR spectroscopy we found that ball-milling treatment had caused a rise of amorphous structure in starch granules (Roa et al. 2014). It is expected that high homogenization and particle size reduction provided by the planetary ball mill have an influence on rheological behavior of flour

dispersions. However, till preset no attempt has been made to study rheological properties of amaranth flour which was modified by high impact milling.

The first objective of our investigation was to determine, the rheological and thermal properties of modified amaranth flour dispersions affected by milling energy, using a rotational viscometer and a DSC technique. The second objective was to identify the effects of the concentration and milling energy on apparent viscosity and the stability of flour dispersion during 24 hours of refrigerated storage.

2. Materials and methods

2.1 Material

Amaranth grain (***Amaranthus cruentus***) used in this study was harvested on the west of Buenos Aires by Cereales Naturales S.R.L (Lomas del Mirador, Argentina). The grains were screened to remove foreign matter and stored in sealed containers at room temperature previous to their use. The moisture content of amaranth grain was 10.5 ± 0.1 g/100g determined by official method 925.09 (AOAC, 2000).

2.2 Modified amaranth flour

Starch-enriched fraction was first obtained by abrasive milling using a laboratory mill Suzuki MT-95 (Suzuki, Sao Pablo, Brazil), which separates automatically pearled amaranth (starchy fraction yield, 70%) and bran (lipid-protein fraction yield, 28%). Milling conditions involved 100 g of milling load and 90 min of milling time (Roa Acosta et al. 2013). Starch enriched fraction was then milled in a planetary ball mill model PM-100 (Retsch, Haan Mettman, Germany) with one stainless steel milling cylinder (500 mL). The amaranth sample and five times weight stainless steel balls (Φ = 10 mm) were placed into the stainless steel container up to about two thirds of their capacity. The milling was rotated horizontally at constant milling speed of 400 rpm at four levels of energy (0.92 - 1.63 - 2.87 - 6.52 kJ/g of sample) obtaining the starch-enriched flours. The ball milling was changed in rotational direction every 30 seconds.

Milling energy values were selected as a milling variable from the operative menu. Control tests were performed for each energy level in order to discount the energy consumption without sample. Milled samples were sifted in a Testing Equipment, model Zonytest through sieves nº 60 (250 μm), nº 80 (177 μm) and nº 200 (74 μm). The residue (74 μm) contained the flour and was selected for different analysis.

2.3 Particle size

Particle size distribution of amaranth flour was measured by static light scattering (SLS) using a Mastersizer 2000 device equipped with a Hydro 2000MU as dispersion unit, from Malvern Instruments Ltd (Malvern Instruments Ltd, Worcestershire, UK). Five scans were recorded for each sample. Sample dispersion was calculated in term of Span (Eqn 1) as function of diameters Dv10, Dv50 and Dv90 stand for 10%, 50% and 90% of total volume at particles distribution, assuming a spherical shape. Where, Dv10, Dv50, and Dv90 are values calculated by the software using the Rayleigh theory. Mean and standard deviation values of triplicates are reported.

$$\text{Span} = \frac{\text{Dv90} - \text{Dv10}}{\text{Dv50}} \quad (1)$$

2.4 Thermal properties

Thermal properties of amaranth flour were determined by DSC analysis using a DSC 822 (Mettler-Toledo model, Schwerzenbach, Switzerland) with an empty pan as reference. Thermal parameters were recorded in triplicate. Amaranth flour (3 mg aprox, dry weight basis) was placed in a 40-µL aluminum pan, and distilled water was added to give a sample-to-water weight ratio of 1 to 3. The pan was sealed, and the sample was allowed to equilibrate overnight at 4ºC before analysis. In the DSC, the sample was held at 30ºC for 1 min followed by heating from 30ºC to 95ºC at a rate of 10ºC/min. Gelatinization temperatures, onset ($\boldsymbol{T}_o$), peak ($\boldsymbol{T}_p$) and endset ($\boldsymbol{T}_e$), together with gelatinization enthalpy ($\Delta\boldsymbol{H}$) were recorded in triplicate.

2.5 Preparation of amaranth flour dispersion

Amaranth flour dispersions (4% and 8% g/100 mL) were prepared by mixing amaranth flour with distilled water. They were moderately stirred for 30 min at room temperature, and heated at 95 °C in a water bath for 30 min with mild agitation provided by a magnetic stirrer. At the end of the heating period, the cooked amaranth flour dispersion was cooled at 25ºC and immediately transferred to the rheometer cup for the measurements of rheological properties. In order to evaluate the stability of the dispersions with time, they were stored at 4 ° C for 24 hours and then measurements of viscosity were repeated.

2.6 Rheological measurements

Flow properties of heated amaranth flour dispersions were determined using a rotational viscometer (Brookfield DV-LVT; Brookfield Engineering Laboratories, Inc., Middleboro, U.S.A) with coaxial cylinder probes. Measurements were made at several rotational speeds corresponding to a percentage torque ranging from 10 to 100%. For low viscosities, an UL/Y adapter with UL spindle was used. The sample chamber with jacket was connected to a constant temperature bath in order to determine flow characteristics at constant temperature of 25 °C. Rheological tests were triplicated.

The data obtained were fitted to the power law:

$$\sigma = K\dot{\gamma}^n \quad (2)$$

Where σ is the shear stress (Pa) and $\dot{\gamma}$ is the shear rate (s^{-1}). The rheological parameters determined were the consistency index (K; Pa $s^{\boldsymbol{n}}$) and the flow behavior index (***n;*** dimensionless). Using magnitudes of K and ***n***, apparent viscosity ($\mu_a = \sigma / \dot{\gamma}$) at specific shear stress can be calculated.

Statistical analysis

Significance of the effect of ball milling energy on rheological and thermal parameters of modified amaranth flour were evaluated by one-way ANOVA (significance level α = 0.05 %) with Tukey

post-test using Prism 5 (GrapPad Software Inc., San Diego, CA, USA). In some cases, p value was calculated by a non-parametric t test in order to deeper analyze differences between samples.

3. Results and discussion

3.1 Modified amaranth flour

Amaranth flour used in this study was characterized in previous investigations according to some of its functional properties (Roa et al. 2014). However for the actual approach we selected those parameters related to particles size distribution because of the particular relationship that they have with grinding energy, the independent variable chose to generate different samples of modified amaranth flour. Therefore, Table 1 resumes the parameters of particle size distribution of modified amaranth flour as a function of the grinding energy delivered during ball milling. It can be appreciated that median particle size (D_{50}) range from 880 µm (0 kJ/g control) up to 70 µm (6.52 kJ/g) while the Span value changed from 2.05 (0.92 kJ/g) to 1.87 (6.52 kJ/g) stating that the samples were more homogeneous with the increasing energy.

3.2 Thermal properties

The crystalline order in starch granule or starch enriched flour is often a basic factor to influence functional properties. Collapse of crystalline order within the starch granules, generates irreversible changes in properties, such as granule swelling, pasting, loss of optical birefringence, loss of crystalline order, uncoiling and dissociation of the doubles helices, and starch solubility (Tester 1997; Singh et al. 2003; Roa et al. 2014).

Starch gelatinization is the transition of the semi-crystalline structure in native starch granules, to an amorphous structure. This transition occurs on a heating of an aqueous suspension of starch granules. Starch transition temperatures and gelatinization enthalpies by DSC have been extensively investigated by many authors (Chen et al. 2003; Dhital et al. 2010a, 2011; Han 2007, Campanella et al. 2002; Morrison et al. 1994).

Table 1 reports the values of the gelatinization temperatures ($\boldsymbol{T}_{onset}$, $\boldsymbol{T}_{peak}$, $\boldsymbol{T}_{endset}$) and enthalpy gelatinization for the flour samples subjected to different ball-milling energies. Enthalpy values decreased significantly compared to those of the control sample from 7.91 ± 0.15 J/g up to 0.34 ± 0.01 J/g. A very significant reduction in gelatinization enthalpy from 14.0 J/g to 0.13 J/g and from 6.8 J/g to 0.9 J/g was observed by Han et al. (2007) and Loubes et al. (2013) respectively, after of rice starch or starch enriched fraction processing in a planetary ball mill; while Sanguanpong et al. (2003) and Martinez-Bustos et al. (2007) reported that milled native cassava starch was highly amorphous due to the loss of crystallinity in ball milling. Therefore, high impact milling caused a decrease of enthalpy gelatinization, producing a disruption of crystalline regions and double helix content, due to the slight depolymerization of amylose and the breakdown of amylopectin, decreasing the ability to gelatinize.

The onset temperature (49.27 ºC ± 0.78 ºC) and the wide gelatinization range ($\boldsymbol{T}_{endset}$ - $\boldsymbol{T}_{onset}$ = 29.83 ºC) of the highly treated sample, was significantly different from those shown in Table 1. This behavior obeys to a decrease on the crystallinity degree (Roa et al. 2014), which causes an unstable

structure and makes the granule less resistant. Another reason is due to irregularly shaped granules produced after ball-milling. A decrease of thermal parameters with an increase of milling time was also reported by Han et al. (2007) and Huang et al (2007), who studied the effect of milling time on thermal parameters of rice starch and cassava starch respectively. While Singh et al. (2003) and Kaur et al. (2002) reported lower transition temperatures due to irregular shape potato starch granules.

3.3 Rheological properties

The effect of milling energy on the viscosity of the flour dispersions, as function of shear rate, is shown in fig. 1 and 2 at different starch concentrations. The apparent viscosity decreased with the increasing shear rate, implying that all the samples analyzed behaved as shear thinning systems. The highest viscosity values were obtained at lowest shear rate, and the lowest at highest shear rate. Similar behaviors were observed by Ibanoglu et al. (1999) and Manohar et al. (1998) when they studied semi-liquids breakfast food.

The data of apparent viscosity at different shear rates was used to determine the behavior of consistency coefficient and flow index, by power law model (eq.2). Consistency coefficient (***K***) and flow index (***n***) values on time (0h – 24h) are given in the table 2 and 3 respectively.

All processed samples (4% - 8%) at time (0h) showed an increase in values of ***n*** as the milling energy increased. This behavior is due to the ball milling, which provoked the breakage of the intra and intermolecular associative bonding system in the starch network, increasing the starch solubility and affecting the process of hydration and swelling of the starch granule. Hasjim et al. (2012) showed similar results when they studied rice milling using hammer and cryogenic milling.

The flour solutions 8% showed a significant increase in flow index (***n***) during storage for 24h (i.e. samples from 0.0kJ/g to 2.87kJ/g). This decrease in pseudoplastic behavior was probably induced by rearrangement of amylose, which increased free-water of system during storage. However, the highly milled sample (6.25kJ/g), showed a lower flow index (***n*** = 0.48) after storage compared to flow index (***n*** = 0.56) before storage. This indicates that it was the most stable sample after storage.

The (***K***) values increased as the concentration increased, due to a higher presence of amylose and amylopectin which facilitates network starch formation (Autio and Eliasson 2009). This ability is lost with increasing grinding due to the changes caused on structure of starch granule. Therefore, the (***K***) value decreased as the milling energy increased (table 2). However, the highly milled samples showed higher stability during storage. This stability is reflected in lower variation on (K) value before and after storage.

4. Conclusions

This study showed that ball milling processing achieved particle size reduction, causing rupture of starch granule, loss in the gelatinization enthalpy and an increasing solubility. These features induced the decreasing pseudoplastic behavior and a higher stability of the soups during storage. The stability of modified amaranth starch represents a technological advantage in the development of liquid food in comparison with the native starch; since it avoids or delays the phase separation in the product. The decrease in viscosity of soups with shear rate levels would facilitate the flow and exchange of heat during

the process, generating low energy demand for the process. The ball milling is a simple process that presents minimal environmental problems and is easy to operate. By selecting appropriate operation conditions, ball milling can provide amaranth starch enriched fractions with some special functional properties that may be of potential use in the food industry.

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Table 1. Particle size distribution characteristics and thermal parameters of starch-enriched amaranth flours obtained by planetary ball milling as function of milling energy.

Milling energy (kJ/g)	Median Diameter D_{50} (μm)	Span (dimensionless)	T_{onset} (ºC)	T_{peak} (ºC)	T_{endset} (ºC)	$\Delta \boldsymbol{H}$ (J/g)
0.0	929.7 ± 76.4[a]	0.247 ± 0.026[e]	65.18 ± 1.17[a]	73.48 ± 0.20[a]	80.82 ± 1.18[a]	7.91 ± 0.15[a]
0.92	201.0 ± 3.3[b]	2.032 ± 0.029[b]	67.52 ± 0.74[a]	74.51 ± 0.11[a]	80.32 ± 0.23[a]	7.04 ± 0.09[b]
1.63	150.4 ± 0.5[c]	2.104 ± 0.004[a]	64.85 ± 5.51[a]	74.26 ± 0.85[a]	80.22 ± 0.11[a]	5.43 ± 0.01[c]
2.87	121.7 ± 0.6[d]	1.909 ± 0.011[c]	67.84 ± 1.14[a]	74.29 ± 0.43[a]	79.69 ± 0.22[a]	3.39 ± 0.23[d]
6.52	68.0 ± 0.5[e]	1.874 ± 0.014[d]	49.27 ± 0.78[b]	57.07 ± 0.20[b]	79.10 ± 0.0[a]	0.34 ± 0.01[e]

Different letters indicate significant differences ($p<0.05$) on each column. Mean and standard deviation values of triplicates are reported

Table 2. Consistency index (equation 2) of amaranth flour dispersions at 25°C as function of flour concentration and storage time[1].

Milling energy (kJ/g)	Consistency index (***K***) Flour dispersion 4% (0h)[1]	Consistency index (***K)*** Flour dispersion 4% (24h)[1]	Consistency index (***K)*** Flour dispersion 8% (0h)[1]	Consistency index (***K)*** Flour dispersión 8% (24h)[1]
0.0	172.0 ± 18.0^{a}	56.21 ± 3.07^{c}	5845 ± 590^{a}	3773 ± 102^{b}
0.92	158.7 ± 14.3^{ab}	53.23 ± 3.19^{c}	4899 ± 159^{a}	3462 ± 141^{bc}
1.63	128.9 ± 3.7^{ab}	37.52 ± 0.48^{d}	4736 ± 96^{a}	3277 ± 66^{cd}
2.87	125.8 ± 2.9^{b}	34.54 ± 1.57^{d}	3856 ± 214^{b}	3079 ± 65^{d}
6.52	57.9 ± 7.7^{c}	24.24 ± 1.72^{e}	606.3 ± 28.3^{e}	678 ± 15^{e}

Different letters indicate significant differences ($p<0.05$) on each concentration. Mean and standard deviation values of triplicates are reported

.

Table 3. Flow behavior index (equation 2) of amaranth flour dispersions at 25°C as function of flour concentration and storage time[1].

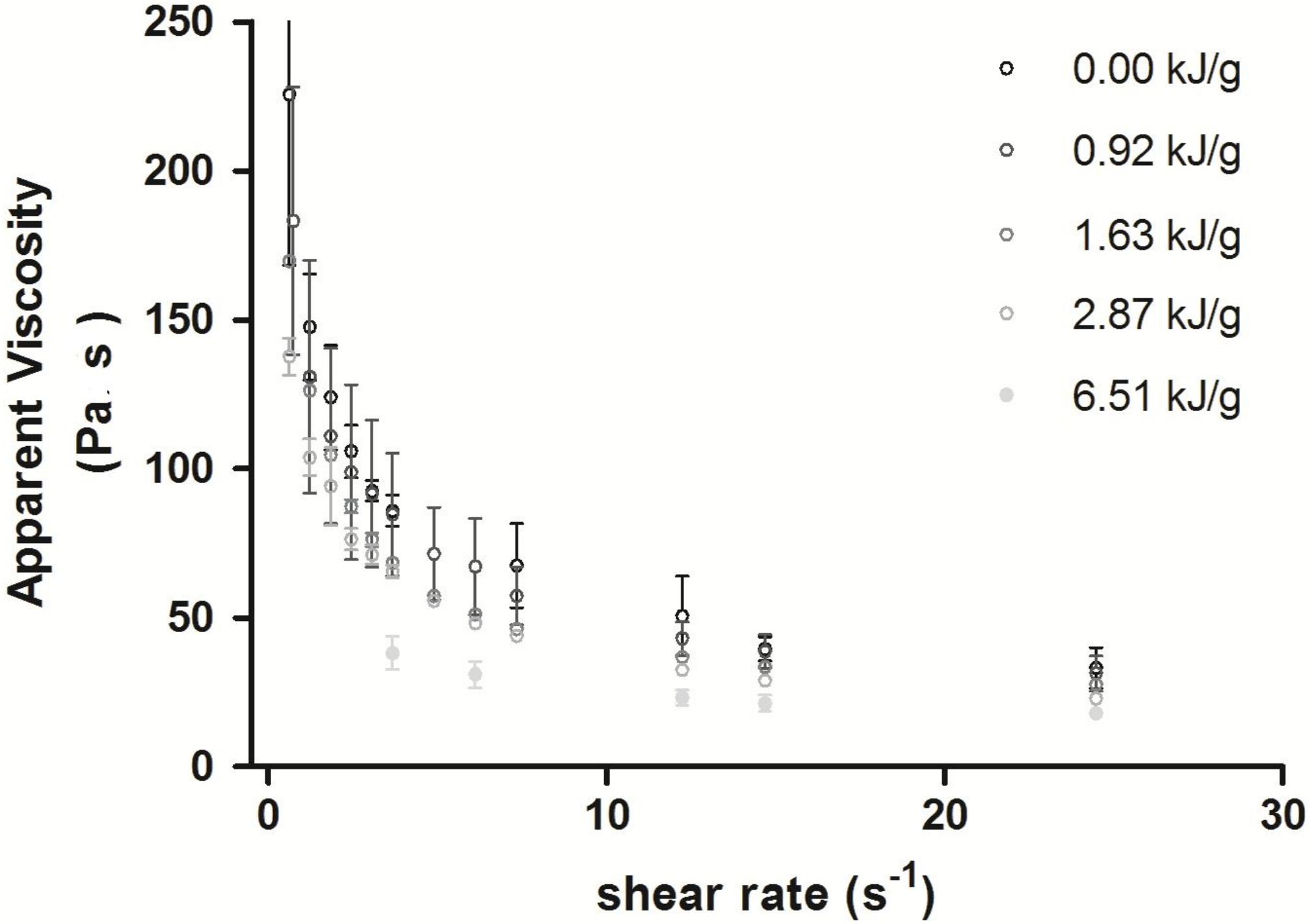
Milling energy (kJ/g)	Flow index (***n***) Flour dispersion 4% (0h)[1]	Flow index (***n***) Flour dispersion 4% (24h)[1]	Flow index (***n***) Flour dispersion 8% (0h)[1]	Flow index (***n***) Flour dispersion 8% (24h)[1]
0.0	0.47 ± 0.01^{b}	0.46 ± 0.09^{b}	0.28 ± 0.05^{d}	0.60 ± 0.04^{a}
0.92	0.48 ± 0.04^{b}	0.43 ± 0.02^{b}	0.36 ± 0.02^{cd}	0.58 ± 0.02^{a}
1.63	0.49 ± 0.04^{b}	0.42 ± 0.09^{b}	0.39 ± 0.01^{c}	0.56 ± 0.02^{a}
2.87	0.51 ± 0.01^{b}	0.40 ± 0.07^{b}	0.48 ± 0.02^{b}	0.55 ± 0.05^{a}
6.52	0.64 ± 0.04^{a}	0.30 ± 0.08^{c}	0.56 ± 0.02^{a}	0.48 ± 0.02^{b}

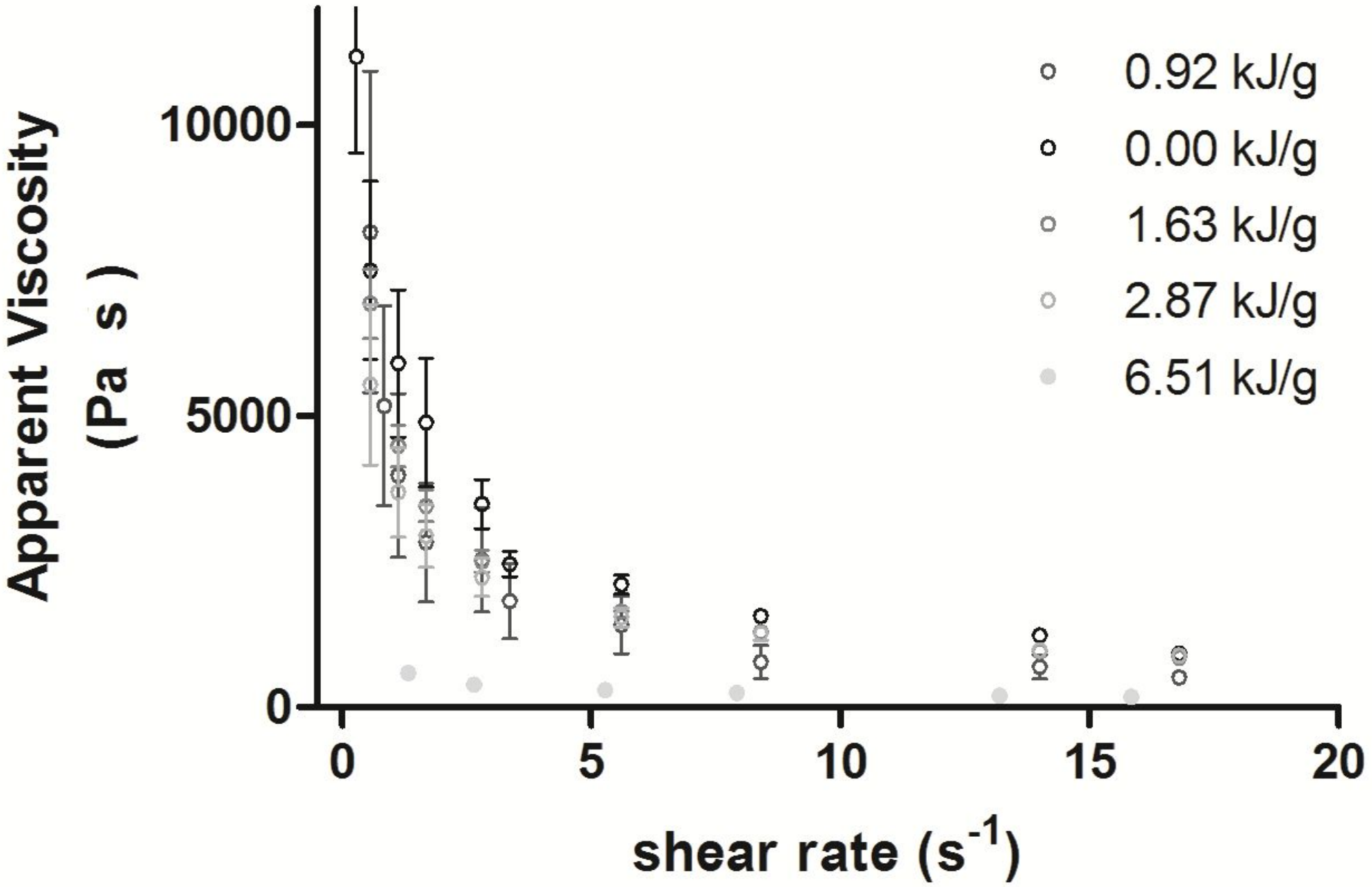
Different letters indicate significant differences ($p<0.05$) on each concentration. Mean and standard deviation values of triplicates are reported.

Figure legends

Figure 1. Apparent viscosity of starch-enriched amaranth flour dispersions (4 g modified flour/100 mL) at 25°C as function of shear rate for different levels of milling energy.

Figure 2. Apparent viscosity of starch-enriched amaranth flour dispersions (8 g modified flour/100 mL) at 25°C as function of shear rate for different levels of milling energy.


Apparent Viscosity (Pa. s)
250
200
150
100
50
0
0
10
20
30
shear rate (s-1)
0.00 kJ/g
0.92 kJ/g
1.63 kJ/g
2.87 kJ/g
6.51 kJ/g


Apparent Viscosity
(Pa s)
10000
5000
0
0
5
10
15
20
shear rate (s-1)
0.92 kJ/g
0.00 kJ/g
1.63 kJ/g
2.87 kJ/g
6.51 kJ/g