Chapter

Physicochemical Characterization of Mesquite Flour (*Prosopis laevigata*), Particle Size Distribution, Morphology, Isosteric Heat, and Rheology

Sadoth Sandoval Torres, Larissa G. Reyes López, Lilia L. Méndez Lagunas, Luis Gerardo Barriada Bernal and Juan Rodríguez Ramirez

Abstract

Mesquite pods were dried and milled. The physicochemical properties of mesquite flour were characterized. The pods were dried at 60°C, 15% RH, and 2 m/s airflow. After drying, two types of milling were applied: (1) industrial blade mill and (2) Blender, and the nutritional composition was determined. The sorption isotherms were obtained at 30, 35, 40, and 45°C for a range of water activity of 0.07–0.9. The particle size distribution and the average particle size of the flours were characterized by means of diffraction of blue laser light; furthermore, the morphology was analyzed by (SEM). The powders were also analyzed by DSC. Alveography was applied to study the rheology of the flour. Mesquite powders are highly hygroscopic, and the (GAB) model displays a good description of the experimental data. Flours expose different morphologies depending on the milling technique; a more homogeneous powder was obtained from the industrial blade mill. Rheological characterization indicates that mesquite flour decreases the tenacity and extensibility of the flour mixture. According to DSC, the flours are very stable over a wide temperature range from 0 to 120°C, and the thermograms indicate a transition of proteins affected by high-molecular-weight carbohydrates and moisture content.

Keywords: ethnic food, drying, milling, powder properties, nutritional

1. Introduction

Mesquite (*prosopis spp*) trees grow in dry environmental conditions, counting 44 species in the world [1, 2]. Mesquite tree appertains to genus *Prosopis* and is present in semiarid lands of America [3]. It has been documented that *Prosopis* pods have been food intakes of people from arid and semiarid regions in South America [4], and according to [5], the *Prosopis* pods are highly palatable to humans.

In Mexico, mesquite is found in different regions of the country. In the past, the native people utilized mesquite pods as food, to prepare flour, syrups, and bread [6]; nevertheless, the pod's morphology varied substantially across different regions [7].

According to [1], mesquite pods have a complex morphological structure; the important protein content and the sucrose content reveal a potential for the production of new ethnic foods. As a raw material mesquite pods can be used for baking, snack food, sweetener, gum, and protein concentrate.

Prosopis pallida and *Prosopis juliflora are varieties with especially large and sweet fruits, and* were studied by [8]. The authors explored different applications for every component of the fruit (exocarp, mesocarp (pulp), and endocarp), the episperm, endosperm, and cotyledon of the seed. They affirm sucrose is the main sugar component in the pulp; galactomannan is the most important polysaccharide in the endosperm, and glutamic acid, arginine, aspartic acid, leucine, proline, and serine were identified in the seed cotyledon. In the pulp, vitamin C, nicotinic acid, and calcium pantothenate were identified. In other study [9], authors characterize the phenolic antioxidants occurring in the pod mesocarp flour of Chilean *Prosopis*; they conduct an HPLC-MS/MS analysis identifying the presence of eight anthocyanins and 13 phenolic compounds including flavonol glycosides, C-glycosyl flavones, and ellagic acid derivatives. The antioxidant activity and the phenolic composition of this product reveal its potential as a functional food.

The structural and functional properties of *P. alba, P. chilensis, and P. flexuosa* were assessed by other author [10]. The flours were characterized by granulometric analyses, water absorption, oil absorption, solubility, and color. Drying and milling process allows ultrastructural changes, modifying the membranes of proteins and changing their capacity to absorb water. The values of solubility reveal flours can be used for the elaboration of liquid foods and candies. According to [2], *Prosopis* mesocarp flour contributes to the browning, color, aroma, and flavor of baked products.

In other work [11], the authors studied the fruits of *Prosopis alba* and *P. pallida*. A drying process was applied at 60°C for 60 h, then a hammer milling process was used. The findings reveal proteins, calcium, iron, dietary fiber, and sugars as the principal constituents of the pulp. The efficiency of the milling and sieving for *P. alba* and *P. pallida* were 54.5% and 55%, respectively. The total sugar content was higher in *P. alba* than *P. pallida* and protein content was higher in *P. pallida* than *P. alba*.

In another study, the authors investigated the particle size, morphology, rheology, physicochemical, and mineral composition of *Proposis julifrora* [3]. Drying at 60°C was applied, after that the pods were analyzed. Sieves with a meshing of 32–150 were used. The flour exhibited an important concentration of fibers, calcium, and phosphorous. The results of rheology indicated that mesquite flour is suitable for cookie production.

In other work, the genotoxicity of *prosopis* flour was addressed [4]. The authors affirm sucrose constitutes the main sugar in flours obtained from *P. alba* and *Prosopis nigra*. *Prosopis* extracts did not reveal any mutagenic effect with and without metabolic activation. The authors conclude *Prosopis* flour is a rich source of antioxidant compounds that could avoid pathologies related to oxidative stress.

For the elaboration of bakery products and/or confectionery, it is indispensable the use of flours with the ideal characteristics that can satisfy the culinary necessities and that in turn can be conserved [10]. Currently, the use of mesquite pods in the food industry is uncommon [2, 5]. Then, the aim of this work was to assess the physicochemical and rheological characteristics of mesquite flours (*Prosopis laevigata*) harvested in Oaxaca State (Southern of Mexico) in order to develop foods with important nutritional value without gluten and take advantage of the agro-food resources of semiarid zones. The particle size distribution, the particle morphology, the isosteric heat of sorption, and the thermal stability were also studied.

2. Materials and methods

Pods of *P. laevigata* were harvested between April and August 2016 in the community of Santiago Sulchiquitongo (Oaxaca, Mexico). Three stages of maturation were identified [12]. Pods in stage three of maturity were used for drying. The drying process of pods was performed using a convective tunnel dryer [13]. The drying conditions were as follows: an airflow at 60°C, with a relative humidity of 15%, and an air velocity of 2.0 m/s. After drying, pods were stored in a desiccator.

The pods reached a final moisture content of 0.12 g of water/g dry matter. Once dried, the mesquite was milled by implementing two techniques; 1) an Osterizer blender, model 465–15 for 20 seconds and 2) a mill blade pulverizer Model HC-2000Y, for 20 seconds. The mesquite powder was passed through # 60 (0.250 mm) and # 80 (0.177 mm) sieves and each mill was stored in low-density polyethylene bags in a vacuum desiccator for 24 hours. A more homogeneous material was obtained from the mill blade pulverizer, then, powders from this milling technique were subsequently analyzed.

2.1 Chemical characterization of flours

The chemical-proximal and nutritional composition of the flours were obtained. The moisture content, the total raw protein content, the reducing and direct sugars, the total fat extraction, the raw fiber, and the ash were determined by methods published in [12].

2.2 Particle size distribution

The size distribution of a particulate product is dependent on the shapes of its particles [13, 14]. The particle size distribution determines the critical chemical and physical properties of particulate systems [15]. Particle size induces many properties of powder materials and is a significant indicator of quality and performance. For this reason, the particle size distribution of mesquite flours was analyzed using the principle of blue laser light diffraction measurement [16]. For this purpose, we use a Microtrac Blueray M3551-1 W-BU00 in a humid medium, with a measuring range of 10 nm up to 2000 microns. The method is presented in [12].

2.3 Morphology of flour particles

Images from samples of mesquite flour were obtained. A scanning electron microscope (SEM) JEOL brand, model JIB-4601F, with a spatial resolution of 1.2 nm, a focused ion beam, and a digital camera (CIIDIR-Oaxaca, Mexico), was used in this work. The flour samples were placed in small graphite plates and introduced into the SEM vacuum chamber. In our analysis, a secondary electron detector E-T (Everhart-Thornley) and a backscattered electron detector were used. A range of magnification from 50x to 2500x was used for the images.

2.4 Sorption isotherms

According to [17], powder processing must be conducted under controlled relative humidity and temperature in order to enhance the storage, handling, and processing. As the relative humidity of the surrounding air is increased, powders tend to absorb water, which may form liquid bridges between powder particles and result in greater powder cohesion. The sorption isotherms of mesquite powders (*Prosopis Laevigata*) were assessed by the gravimetric static method with water activities ranging from 0.07 to 0.97 at four temperatures: 30, 35, 40, and 45°C. The salts used in this work were the following: NaCl, $MgCl_2 \cdot 6H_2O$, KOH, KCl, KI, K_2SO_6, and $Mg(NO_3)_2 \cdot 6H_2O$ [18, 19] The details of this method are presented in [12]. Experimental data was fitted to the (GAB) (Guggenheim-Anderson-Deboer) model (Eq. (1)). The theoretical fundamental for the GAB sorption isotherm is the assumption of localized physical adsorption in multilayers without lateral interactions [20]. The parameters for this model were estimated by implementing the equation in excel (GRG nonlinear).

$$\mathrm{Xeq} = \frac{\mathrm{Xm} \cdot \mathrm{C} \cdot \mathrm{k} \cdot \mathrm{aw}}{(1 - \mathrm{k} \cdot \mathrm{aw}) \cdot (1 - \mathrm{k} \cdot \mathrm{aw} + \mathrm{C} \cdot \mathrm{k} \cdot \mathrm{aw})} \tag{1}$$

where *Xeq* is the equilibrium moisture content (g H_2O/g dry basis), X_m is the monolayer moisture content (g H_2O/g dry basis), and C is a heat-related constant of the monolayer, *k* is a constant related to the sorption heat of the multilayer, and *aw* is the water activity.

2.5 Isosteric heat of sorption

The estimation of energy consumption during drying needs the knowledge of the enthalpy of water sorption in the entire range of moisture contents. Certainly, the use of the enthalpy of vaporization of pure water can give inaccurate results [21]. The isosteric heat of sorption is a useful expression, notably in the design of drying operations, as heats of sorption rise well in excess of the heat of vaporization of water as food is dried to low moisture contents [22]. The net isosteric heat of sorption measures the binding energy of the forces between the water vapor molecules and the solid phase [23]. It gives information for the comprehension of the sorption mechanism [20]. The gap between the amount of energy necessary to remove water from the flour and the amount of energy needed for normal water vaporization is defined as the net isosteric heat of sorption. The isosteric heat of sorption (Q_{st}) was estimated by using the equation derived from Clausius Clapeyron [22–24]; in order to calculate the enthalpy change associated with the sorption process (Eq. (2)).

$$\left[\frac{\partial \ln (a_w)}{\partial (1/T)}\right]_{CHE} = -\frac{Q_{st} - \lambda}{R} = -\frac{q_{st}}{R} \tag{2}$$

where q_{st} is the isosteric heat of sorption, R is the universal constant of the gases (0.00831 Joules/K·mol), a_w is the water activity, T is the temperature (K), and λ is the latent heat of vaporization of pure water at room temperature. For isosteric heat of sorption, we used the experimental information of the sorption isotherms.

2.6 Differential scanning calorimetry (DSC)

The scanning calorimetry provides a direct estimate of the overall enthalpy change of transitions without requiring knowledge of the thermodynamic mechanism; moreover, the sample preparation is minimal [25]. Four samples of mesquite flours (*P. laevigata*) previously conditioned at a relative humidity of 7%, 32%, 51%, and 67% were prepared for a DSC analysis (TA Instruments, model Q2000). 17 mg of flour was placed inside hermetic aluminum capsules and sealed by a press. The experimental conditions consisted in running the samples at an initial temperature of 0.0°C, followed by a heating rate of 2°C/min up to an end temperature of 250°C. The thermograms were analyzed by using the TA Instruments DSC software.

2.7 Alveography

Alveograph method is useful to estimate the potential performance of flours [26]. The Alveograph test provides a test sample of dough, which, under air pressure, forms a bubble. The test recreates the deformation of a dough when is subjected to carbondioxide during fermentation [27].

Alveographic characteristics were analyzed for three mixtures of wheat/mesquite flour. Experiments were conducted in triplicate in alveographic equipment (Chopin Technologies, France), following the AACC Method 54–30.02 [28], quantifying the following parameters: tenacity (P), extensibility (L), and dough deformation energy (W).

The tenacity is the capacity to resist deformation, extensibility is the maximum volume of air that the bubble is able to enclose, and deformation energy corresponds to the dough baking strength.

3. Results and discussion

The pods show different shades of color during maturity; these changes are accompanied by variations in the organoleptic and physical properties. According to [12], pods reveal three stages of maturity: pods in stage 1 showed a green coloration, in stage 2 the pods were brighter with a reddish coloration, and in stage 3 the pods increased in brightness compared to the previous stages, reddish and yellow coloration was still observed, cream color is characteristic of this last stage. For the study, we used exclusively pods in stage 3 of maturation.

3.1 Chemical composition of flours

The nutritional composition of mesquite flours is shown in (**Table 1**). The nutritional compositions of mesquite flours present a complete composition of macronutrients. Some differences according to the type of milling were observed, the fiber content was higher for milling with a blade mill; however, the proteins decreased slightly. Both flours are suitable for use as complement flour or as a natural supplement.

Components	Blender (1)		Blade (2)	
	Average (g/100 g)	Standard deviation	Average (g/100 g)	Standard deviation
Energy content (kcal/100 g)	170.97	0.07	198.72	0.50
Carbohydrates (g)	24.27	0.09	26.18	0.08
Sugars (g)	7.48	0.03	10.18	0.02
Proteins (g)	12.4	0.08	11.77	0.12
Fats (Lipids) (g)	2.16	0.03	2.90	0.06
Fiber (g)	16.9	0.2	17.25	0.11
Ashes (g)	3.12	0.01	3.45	0.05
Humidity (g water/g dry matter)	0.10	0.01	0.10	0.01

Table 1.
Nutritional composition of mesquite flour (Prosopis laevigata) *obtained by two different grindings.*

3.2 The particle size distribution

In **Figure 1** we show the size distribution of the flours. During the procedure, the aggregates were dispersed with the help of the sample dispersion accessory, and a power of 30 watts was applied for 30 s.

For the flour obtained from the blender (**Figure 1a**), two oblations were identified. Particles of 1.291 microns with 95% up to 657 microns and a cumulative 10% of particles of 28.93 microns were measured.

For the blade mill (**Figure 1b**), the particle size distribution showed a smooth and unique Gaussian distribution. The sample was dispersed very nicely with the simple agitation of the circulatory without forming aggregates. The fine particles are presented from 28.53 microns with a cumulative 10% of particles of 64.23 microns. There are particles as large as 497 microns, with a cumulative 95% of 302.5 microns. The powders showed a homogeneous distribution with adequate particle size. Mesquite flour can be used for the elaboration of baking and confectionery products.

3.3 Morphology of flour particles

The (SEM) images of flour particles are shown in **Figure 2**. The two methods of milling produce different morphologies of powders. Diverse shapes and sizes of mesquite particles with irregular surfaces are identified, and the smooth and striated parts are shown.

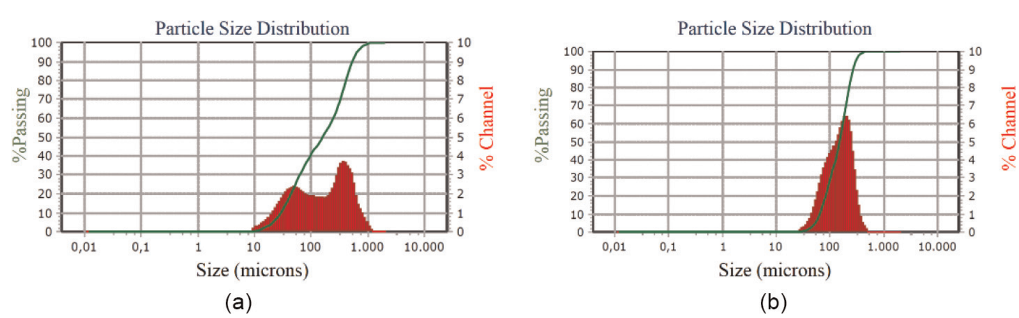

Figure 1.
(a) Particle size distribution – Blender; and (b) Particle size distribution – Blade mill pulverizer.

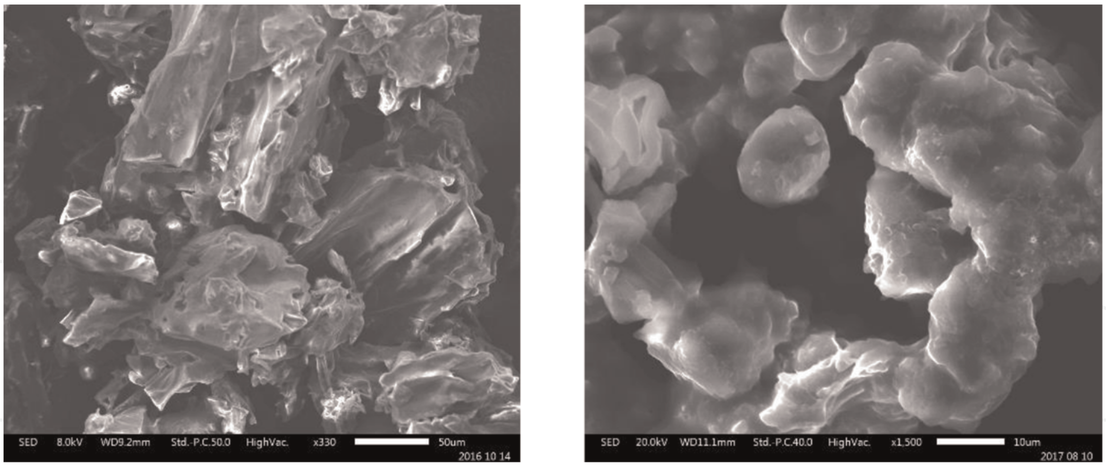

Figure 2.
(a) Micrographs of mesquite flour (Blender), 330×; and (b) Micrograph of mesquite flour (Blade mill), 1500 ×.

Figure 2a shows the morphology of powders obtained from the blender. It shows a slight agglomeration and striated parts forming a tortuous, irregular, and rocky agglomerate. It can be observed the presence of some cavities that can allow the moisture adsorption. **Figure 2b** shows the morphology of powders obtained by blade mill. It reveals the surface of a particle with a rounded structure, better defined, with smooth surfaces without forced cuts.

3.4 Sorption isotherms

The experimental information and the simulations of the GAB model at 30, 35, 40, and 45°C are shown in **Figure 3**, which display a Type II isotherm shape, and indicate a likely small adsorption force in the monolayer [29, 30]. This type of isotherm has been presented in materials containing fibers and polysaccharides of cereals (wheat,

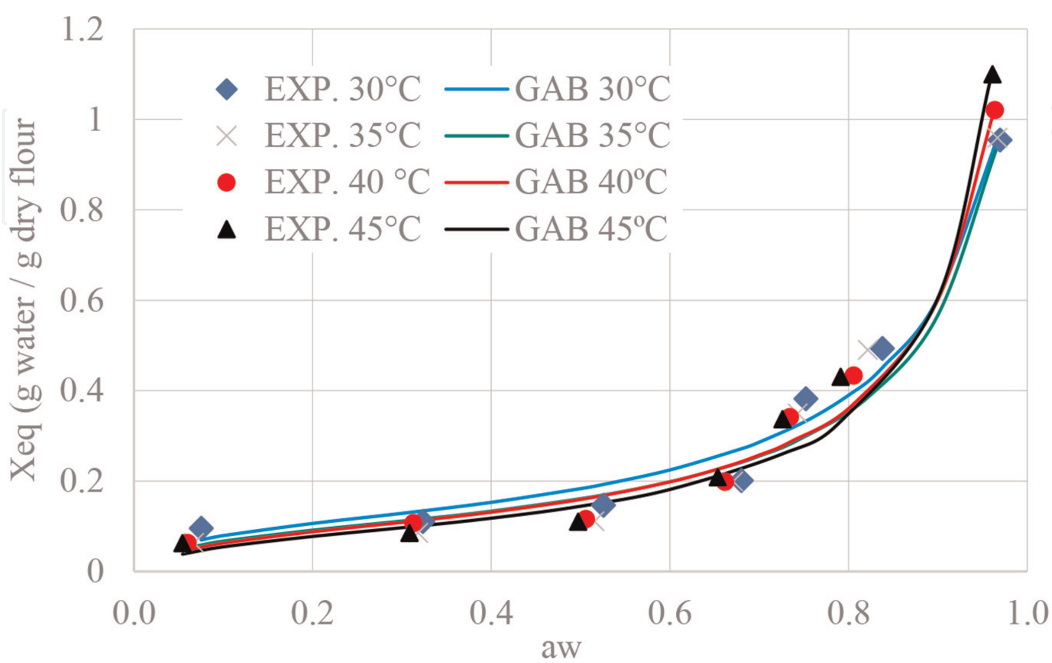

Figure 3.
Sorption isotherm of mesquite flour (Prosopis laevigata) and simulated GAB model.

Model	Parameters	30 °C	35°C	40°C	45°C
GAB	Xwa (g water/g dry matter)	0.1039	0.0905	0.0894	0.0824
	C	22.1997	19.9075	16.6142	15.2432
	k	0.9219	0.9355	0.9475	0.9656
	r^2	0.9898	0.9772	0.9817	0.9773
	s	0.0555	0.0741	0.0592	0.0703

Xwa = Monolayer value; C and k = constants for the model; r^2 = correlation coefficient; and s = standard error.

Table 2.
Estimated parameters for the GAB model.

rice), tubers (potato, cassava), proteins (soybean, maize), and starches, these components are present in the mesquite flour. In the experimental curves, it was observed that the equilibrium moisture content notably increases for aw = 0.67–0.97. The GAB model correctly represented the experimental data for all experimental conditions, giving a %Error of 10–15%. **Table 2** shows the parameters estimated from the GAB model.

According to [20], parameter C indicates the strength of binding of water to the primary binding sites. The larger the C, the stronger water is bound in the monolayer, and the larger the difference in enthalpy between the monolayer molecules and multilayer molecules. In the case of parameter K, when it approaches one (our case), there is almost no divergence between multilayer molecules and liquid molecules. In that case, the water molecules beyond the monolayer are not structured in a multilayer, but have the same characteristics as the molecules in the bulk liquid, as discussed by [31].

3.5 Isosteric heat of sorption

Figure 4 shows the evolution of isosteric heat (Qst) versus the moisture content (Xw) of mesquite flour (*Prosopis laevigata*). We observe Qst decreased rapidly with the increase of Xw; being the lowest value of Qst at 47.69 kJ/mol at 0.25 of Xw. This situation indicates a low availability of active sites and liaison forces on the surface of the powder [23]. When the moisture content is 0.15 g of water/g of dry matter, the heat needed to evaporate the water from mesquite flour would be 57.03 kJ/mol., without affecting the stability of the powder. The net isosteric heat of sorption estimates the binding energy of the forces between the water vapor molecules and the solid phase. It allows a better comprehension of the sorption mechanism.

Figure 4 depicts positive quantities, manifesting the endothermic behavior of desorption. The isosteric heat of sorption decreases as moisture content increases. This fact refers to the intermolecular attraction forces between sorptive sites and water vapor.

The higher the moisture content, the less energy is necessary to remove water molecules from the flour. As drying continues sorption will perform at active sites demanding higher interactive energies [20].

3.5.1 Differential scanning calorimetry (DSC)

Since food powders are complicated mixtures of compounds, it is regularly difficult to identify their phase’s transitions accurately. **Figure 5** shows the DSC curves for

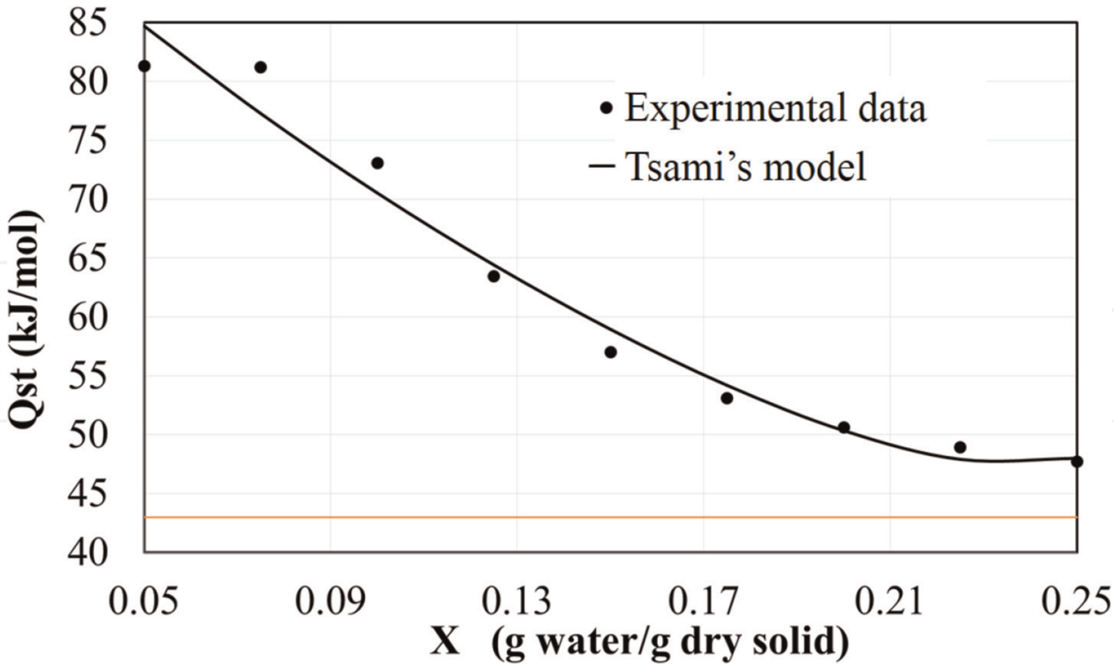

Figure 4.
Isosteric heat of sorption.

mesquite flours exposed at four RH (6.3, 31.8, 48.5, and 66.1%). If it is true that flour contains protein, the heat denaturation temperatures of proteins *in solution* are normally below 100°C; nevertheless, proteins become stable toward heat when the moisture content is low. A clear example of moisture effect on protein denaturation is published in [32]. A DSC thermogram for wheat flour is presented by [17]; in this work, a crystallization peak was observed near 190°C, which is referred by the authors as a decomposition of the flour. In our work, the glass transition (Tg) of the mesquite flour was not observed, due to possible flexibility and mobility of the glucose and fructose chains, provoked by the increase of moisture content of the powders when exposed at different RH (6.3, 31.8, 48.5, and 66.1%). According to [33], the glass transitions of high molecular weight carbohydrates and proteins arise well above 100°C and approach thermal decomposition temperatures of the food powder. Moreover, the plasticization effect of water leads to depression of the glass transition temperature causing noticeable changes in the physicochemical and crystallization properties of the material.

The transitions shown in **Figure 5** display a first-order behavior. The transition phase that was observed in all the flour samples was the crystallization peaks (Tc) from 140–157°C. Likewise, the heat flow of the endothermic transitions increased as the moisture of the flours increased, corroborating the effect of the relative humidity in the modification of the structure of the flours. It is also well-known sugars affect the protein thermal properties [25]. The thermograms of mesquite flour showed significant endotherms in the range of 130–180°C, being associated with a melting of the simple sugars present in the flour.

According to Barba de la Rosa et al. [34], sugars such as glucose, maltose, L-arabinose, and sucrose are found in high proportion in mesquite pods, so the transitions for this food can be related to a binary water-carbohydrate system.

The thermograms in **Figure 5** show the transition of proteins affected by high molecular weight carbohydrates and moisture content of powders.

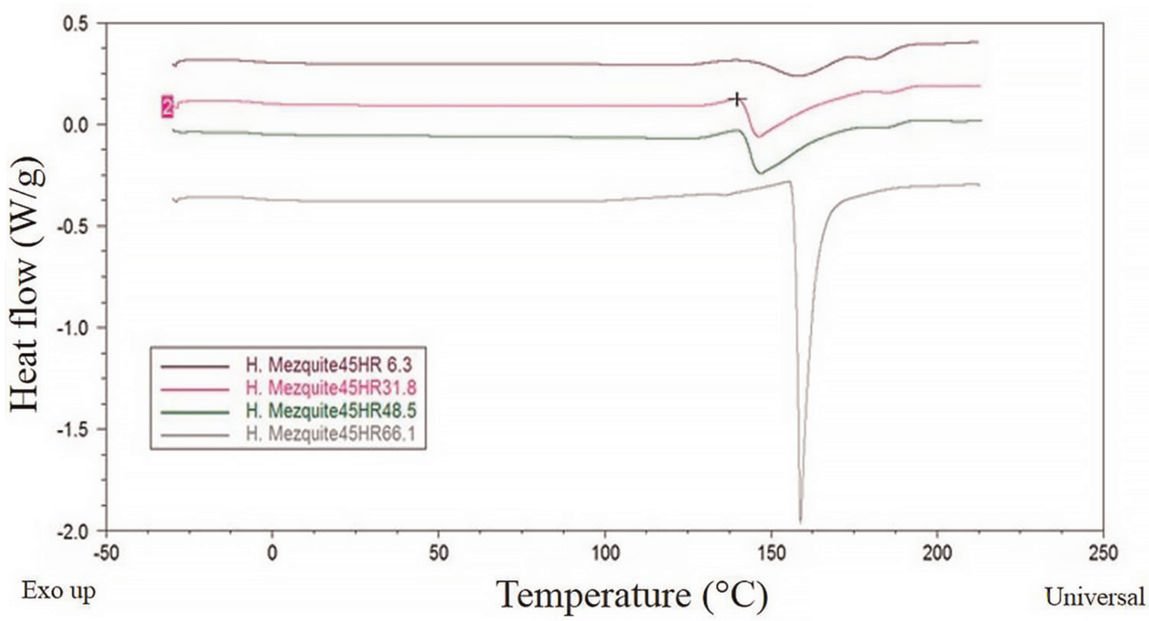

Figure 5.
*DSC curves of mesquite flours (*Prosopis laevigata*).*

3.6 Alveography

The water absorption of mesquite flour can significantly affect the rheological properties of a mixture. According to our results (**Table 3**), the dough's tenacity ranged 18–83 mmH_2O, decreasing with the increase of mesquite flour content of 0–15% (wt). According to [35], tenacity values for standard wheat quality range 60–80 mm H_2O, and very good wheat quality 80–100 mm H_2O, then mesquite flour decrease the quality of the mixture, for this reason, it should be mixed at low concentrations for baking applications.

Extensibility characterizes the average length of the alveogram from the point at which the bubble starts to inflate to the point at which the bubble breaks. Extensibility (L) decreases as mesquite content increases, so mesquite flour impacts the handling properties of the dough. According to [3], when the concentration of mesquite flour is increased, tenacity and extensibility are reduced, and this fact can be explained by the weakening of glutenin protein (a protein responsible for elasticity and extensibility), and also due to a lower water absorption due to the high fiber content of mesquite flour. The higher the addition of mesquite flour with wheat flour, the dough may have a lower capacity to retain the gas generated during fermentation and rising of the bread. The energy of deformation (W) ranged 2.6–4.2, indicating that as mesquite

Mixture % wt Wheat flour + % wt Mesquite flour	Tenacity (P) Average ± SD	Extensibility (L), Average ± SD	Deformation energy (W), Average ± SD
100% + 0%	83.0 ± 6.0415	74.6 ± 7.8930	4.20 ± 3.2985
95% + 5%	35.4 ± 2.6076	81.0 ± 6.2449	2.63 ± 2.5467
90% + 10%	22.4 ± 5.0793	50.4 ± 32.9135	2.74 ± 0.8354
85% + 15%	18.4 ± 3.1304	19.0 ± 5.3385	2.67 ± 2.0255

Table 3.
Results of the alveography.

proportion increases the baking strength decreases. W is frequently referred to as flour strength, dough strength, baking strength, or flour protein strength [36]. W is one of the industrially most applied alveograph parameters, as it is used for prediction of processing behavior of flour cultivars [35]. For example, according to [37] bread flours are characterized by larger W values compared to biscuit flours. W is positively related to the water absorption of the flour [38]. According to our results, an increase in mesquite flour content reduces water absorption, then, this effect should be considered on the type of application, either in baking or as biscuits.

4. Conclusions

Dried pods were milled by two milling techniques. The two techniques of milling produce different morphologies of powders. Particles from the blade mill were more homogeneous, they showed a smooth and unique Gaussian distribution, and SEM images reveal a rounded structure, better defined, without rocky parts nor forced cuts. The flour from blade mill has an adequate particle size to be used as flour for baking and confectionery products. Flours from the two different grindings have an important content of protein, sugar, and fibers. Mesquite flours showed a type II isotherm for the three experimental temperatures, indicating a possible small adsorption force in the monolayer. Flours are notably hygroscopic, and this phenomenon is related to the sugar content and the powder's microstructure. The isosteric heat reveals the endothermic behavior of desorption and the energy required to remove the water molecules from the powder.

The calorimetric data of the flours showed thermal stability in a wide temperature range; however, for temperature > 130°C crystallization peaks (Tc) were observed (from 140–157°C), which show the transition of proteins affected by sugars and moisture content.

Our results confirm that, as the mesquite flour proportion increases in the mixture, the flour became poorer. Tenacity values for standard wheat quality range 60–80 mm H_2O, whilst our mixtures showed lower values. According to the values for extensibility, the presence of mesquite flour affects the handling properties of the dough. The values of deformation energy show mesquite flour develop a weak baking strength, then it should mix at low concentrations for baking applications since an increase in mesquite flour content reduces water absorption. The nutritional composition of the flour releases useful attributes for many applications in food industry, however, this information should be carefully studied, depending on the application of mesquite flour, either in baking or as biscuits.

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