

Modeling of particle size and energetic requirement in amaranth grain ball-milling

Modelado del requerimiento energético y del tamaño de partícula en la molienda de bolas del grano de amaranto

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ABSTRACT

Amaranth flour is of high nutritional value, which makes it a potential food. Grinding of the grains is a necessary operation to obtain products with physical properties that provide the food products with adequate characteristics. To analyze the effect of grinding velocity and time on the particle diameters and physical properties of Amaranth flour by ball mill, a Doehlert design with triplicate at the central point was used. The tests were carried out with the mass ratio (balls/samples) (R1:5). Granulometry curve of each design system was fitted to the Rosin-Ramler-Bennet and Holmes-Hukki equations. A found a very significant effect of the velocity on the particle diameters (D_{50} , D_{63} and D_{80}). The flour obtained were modeled satisfactorily ($r^2 > 0.99$) by using the Rosin-Ramler-Bennet equation, where the homogeneity index of (n_1) was obtained, which was directly influenced by the milling energy. By using the Holmes-Hukki model, were able to model the characteristic diameters with the grinding energy; a critical region

was observed between 100 μ m and 200 μ m, where lost efficiency in the size reduction. The excess energy, released in the critical region, caused the decrease in starch crystallinity and structural changes in the protein, which affect the functional properties of the flour. The planetary mill is emerging as an effective mean of modifying the functional properties in the development of new food products.

Keywords: rosin-ramler-bennet model; holmes-hukki model; water absorption index; water solubility index; granulometry of amaranth grain.

RESUMEN

La harina de amaranto es de alto valor nutricional, convirtiéndola en un alimento potencial. La molienda de los granos es una operación necesaria para brindarle a los productos alimenticios unas adecuadas propiedades físicas. Para analizar el efecto de la velocidad y el tiempo de molienda sobre el diámetro de particular y las propiedades físicas

de la harina obtenida por molienda de bolas, se usó un diseño experimental Doehlert con réplica en el punto central. En las pruebas de molinería se tuvo en cuenta la relación masa de bolas/masa de muestra (R1:5). Las curvas de granulometría de cada punto del diseño experimental fueron modeladas por las ecuaciones de Rosin-Ramler-Bennet y Holmes-Hukki. Se encontró un efecto muy significativo de la velocidad de molienda sobre los diámetros característicos (D_{50} , D_{63} y D_{80}). El modelo de Rosin-Ramler-Bennet ajustó satisfactoriamente ($r^2 > 0.99$), además, se obtuvo el índice de homogeneidad (n), el cual fue afectado directamente por la energía de molienda. El uso del modelo de Holmes-Hukki permitió relacionar el diámetro de partícula con la energía de molienda y se logró observar una región crítica entre $100\mu\text{m}$ y $200\mu\text{m}$, donde hay una reducción en la eficiencia de la reducción de tamaño de partícula. El exceso de energía liberada en la región crítica causó el descenso en la cristalinidad del almidón y provocó cambios en la estructura de las proteínas, lo cual, modificó las propiedades físicas de la harina. El Molino planetario es una técnica emergente y efectiva para modificar las propiedades funcionales en el desarrollo de nuevos productos alimenticios.

Palabras clave: Modelo de Rosin-Ramler-Bennet; Modelo de Holmes-Hukki; índice de absorción de agua; índice de solubilidad; granulometría del grano de amaranto

INTRODUCTION

The grinding operation of a material is a complex process that coordinates the technical parameters, the quality of the grinding surface and the characteristics of the material to be reduced (Guerrini *et al.* 2017; Ivanova, 2016). Despite the large number of studies in the field of fracture systems, there is no single known formula that effectively predicts the energy required to reduce a material from an initial particle size to a smaller size (Suresh *et al.* 2016). However, there are three semi-empirical models, each effective in its own range of work, whose predictions can reach the experimental values in the grinding tests (Roa *et al.* 2014; Rhodes, 2008). These three classic models are known as Milling Laws and are represented by the Rittinger, Kick and Bond equations (Rhodes, 2008). These laws can be expressed by the general equation developed by Holmes and Hukki:

$$dE = -C \frac{dx}{x^{n+1}} \quad \text{equation 1}$$

E: milling energy
C: milling constant
x: granularity
n: milling index

Based on this model, several authors have suggested that, the Kick equation can be used to determine the energy requirement in the grinding of large particles, while the Rittinger model can be used in the milling of fine particles (Roa *et al.* 2014; Rhodes, 2008). The granularity of flours can be represented graphically by a distribution of frequency or accumulated distribution. In literature, different functions have been reported to simulate the size distribution,

such as the Gates-Gaudin-Schumann equation and the Rosin-Rammler-Bennett model, among others (De la Cruz *et al.* 2015; Sanchez *et al.* 2008; Cajas *et al.* 2015). Materials with a normal distribution are relatively rare in the food area and are found mainly in powders obtained by chemical processes such as condensation and precipitation. However, the importance of the normal function is that it provides an idealized distribution of the error, based on the assumption that the elementary errors are combined at random to produce the observed effect. The Rosin-Ramler-Bennet equation (Eq. 2) was introduced in 1933, and is widely employed in the characterization of the particle size distribution when there are departures from the normal distribution (De la Cruz *et al.* 2015; Sanchez *et al.* 2008). Usually the function is presented in terms of accumulated frequency and involves two parameters:

$$F(x) = 1 - \exp\left[-\left(\frac{x}{x_{63}}\right)^n\right] \quad \text{equation 2}$$

Where $F(x)$ is the cumulative distribution, (x) is the characteristic particle size, (x_{63}) is the characteristic particle size corresponding to 63.2% of the cumulative distribution and (n) is defined as the index of homogeneity. Low values of (n) indicate a wider dispersion of size; while high values of (n) indicate less dispersion (Rhodes, 2008; De la Cruz *et al.* 2015; Ceron *et al.* 2016). The planetary ball mill is a new technology used in the development of new materials and that recently has been employed in the pharmaceutical area, it is important to study the application of this technology in the food area (Yaozhong *et al.* 2019; Limin *et al.* 2018). For this reason, the work objectives are as follows: study the relationship between the flour granularity and the energy requirement of the planetary mill by using mathematical models and then describe the effect of high impact milling on some physical properties of the flours.

MATERIALS AND METHODS

Materials. The material used was amaranth grain (*Amaranthus cruentus*), provided by S.R.L Natural Cereals (Lomas del Mirador, Provincia de Buenos Aires, Argentina). The grains were sifted (ASTM # 5) in order to remove the foreign material and then stored in polypropylene bags at 4°C .

Milling methods

Method (I) High impact grinding as a function of time and rotation speed. A planetary PM-100 ball mill (Retsch, Haan Mettman, Germany) equipped with a cylindrical stainless-steel jar (500mL) was used. The samples of amaranth (154g) and stainless-steel balls ($\varphi=10$ mm) in a relation of (1:5) were placed inside the cylindrical jar filling it to a third of its capacity. The grinding was carried out as a function of time and speed of rotation. To analyze the effect of speed, time and milling energy on the indexes of size reduction (D_{50} , D_{63} and D_{80}) as well as on homogenization index (Span and n), the experimental design matrix of Doehlert^{9,11} was used. The speed ranges of (250rpm - 450rpm) and time (5 min - 33 min) ranges were estimated based on previous tests (Roa *et al.* 2014).

Method (II) High impact milling based on operating energy. The planetary ball mill PM100 has the energy function which to

determine the total energy provided to the grinding vessel. The energy is defined as the difference between the energy consumed for driving the full milling vessel (material + balls) and the energy required for the empty milling vessel (baseline energy). In the first place, the energy consumed at idle is determined for a short period of time, which is considered by the equipment's electronics as the point zero or baseline. The energy that exceeds the idle level is considered the grinding energy. The mill provides the grinding energy value in kJ, the specific grinding energy was obtained by kJ/g considering the sample mass introduced in the milling jar. The milling was carried out at a constant rotation speed of 450rpm and at different specific energy levels (0kJ/g – 6.5kJ/g).

Modeling of granulometry curves. The generalized size reduction model (Eq. 1) was used in order to determine the milling constant and index (n). The non-linear regression of data was completed by using the computer program STATGRAPHICS Centurión XVI.I. The non-normal distribution model Rosin Rammler Bennet was used (Equation 2) to determine the values of characteristic diameter (D_{63}) and homogeneity index (n).

Determination of the particle size of the flours. The particle size of the of each point of the experimental design were measured by the laser diffraction (SLS, Static Light Scattering) method on a Mastersizer 2000 with a hydro 2000 UM as the dispersing unit (Malvern Instruments, Worcester Shire, United Kingdom). The speed of the flow pump was 1800 rpm, the refractive index (IR) of the dispersing phase was 1.330 (water) and the starch was 1.535 with an absorption value of 0.001. The equipment operates by performing 10 sweeps on each sample and provides the particle size distribution in terms of volumetric fraction as well as the measurements of central tendency and homogeneity of the sample. The volumetric diameters ($D_{4/3}$) were used to determine the percentiles of the distribution size which was calculated as the ratio of differences between the diameters (D_{10} , D_{50} and D_{90}), where 10%, 50% and 90% represent the particle volume with diameters of less than or equal to D_{10} , D_{50} and D_{90} respectively.

Determination of water absorption and solubility index. The water absorption index (WAI) was determined using the Roa method (2014), with some modifications. Two grams of ground flours were dispersed in 30mL of distilled water and heated at $30 \pm 1^\circ\text{C}$ for 30 minutes. The content was then centrifuged at 1000g for 10 minutes. The sediment was weighed and the supernatant decanted and dried to determine the content of solids. The water solubility index (WSI) was determined by evaporating the supernatant overnight and obtaining the content of the dissolved solids. The average and standard deviation obtained from duplicate are reported.

Experimental design and statistical analysis. The Doehlert design behaves according to the polynomial function of the second degree in terms of coded variables:

$$Y = a_0 + \sum_{i=1}^n a_i x_i + \sum_{i=1}^n a_{ii} x_i^2 + \sum_{i=1}^{n-1} \sum_{j=2}^n a_{ij} x_i x_j \quad \text{equation 3}$$

Whereby a_0 , a_i and a_{ii} represent the coefficients corresponding to the terms constant, linear and quadratic. The interaction between the factors studied is given by the a_{ij} coefficient. A linear coding was applied assigning the coded values 1 and -1 to the upper and lower ends of the established experimental range for each of the variables. The coded factors were X1 for time, X2 for speed. Using an ANOVA, verification was made regarding which of the process variables had significant effects ($p < 0.05$) on the responses. The proportion of the variance detailed by the proposed model was calculated by determining the coefficient R^2 . In addition, the adjustment of the model was evaluated by using the lack of fit test², which is significant for values less than $p < 0.05$. For the analysis of the experimental design, as well as to perform each of the non-linear regressions for the modeling of the granulometry curves, the statistical program Statgraphics Plus ® version 5.1 (Statistical graphics Corporation, Princeton, New Jersey, United States of America) was used. Moreover, to compare the properties of WAI and WSI of the flours, an ANOVA and a Tukey Test were also performed using the Graphpad 5 program (Graphpad Software, San Diego, United States of American).

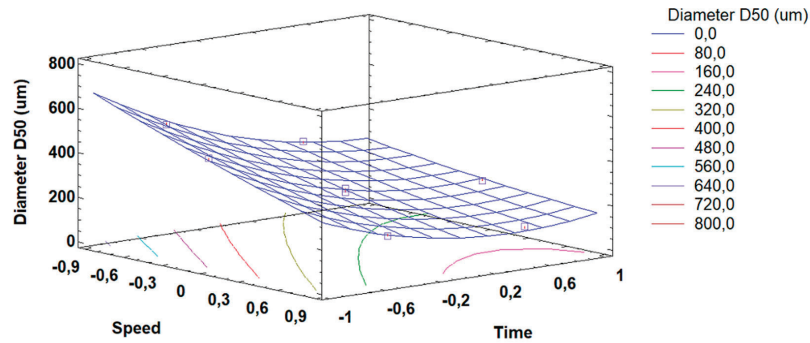
RESULTS AND DISCUSSION

To develop an efficient milling in a planetary ball mill, some parameters were established such as: the size of balls, the amount of grains, the rotation speed of the mill, the operating time, the efficient ratio of ball mass/mass of the grain, and the effects of speed. Preliminary tests were carried out in order to determine the ratio R1:5 (ball mass/amaranth mass). The mass of balls was fixed/set at 800g that corresponded to 1/3 of the volume of the milling jar and the speed and milling time were set at 350rpm for thirty (30) minutes respectively, this condition was recommended by the manufacturer to carry out the milling process.

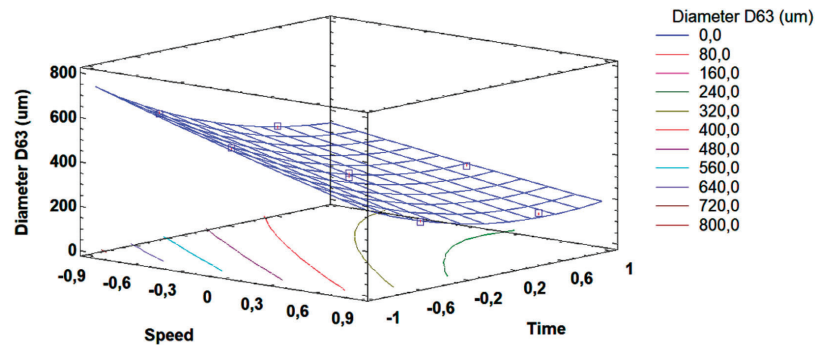
Determination of the granularity and the characteristic diameters (D_{50} , D_{63} and D_{80}). It was carried out the study of the Dolerth model in order to determine the effect of the speed and operating time on the different responses. Table 1 shows the experimental velocity and time values as well as the coded values for each variable. It is important to highlight that the Dolerth model is a hexagon figure inscribed inside a circle, which provides it with symmetry from the central point to the extreme outer points of the design. A reference is made in figure 1 regarding the estimated response surface of diameter D_{50} to different combinations of speed and milling time. The shape of the response surface was similar for each particle diameter (D_{50} , D_{63} and D_{80}).

The correlation of the quadratic model (Eq. 3) was 99% indicating that the response surface can be used to predict different values within the experienced range. The equation modeling of the response surface is shown below figure 1. Where shows a very significant effect of the speed and milling time in each characteristic diameter (D_{50} , D_{63} and D_{80}), where it also shows a quadratic effect of the operation time on the particle size, meaning that after a critical time t_c (values coded between 0 and 0.2) an increase in particle diameters may occur, which would be related to the phenomenon

$$\text{Diameter } D_{50} = 253,433 - 130,658 \cdot \text{speed} - 132,967 \cdot \text{time} + 15,4676 \cdot \text{speed}^2 + 69,2841 \cdot \text{speed} \cdot \text{time} + 90,6667 \cdot \text{time}^2$$



$$\text{Diameter } D_{63} = 328,333 - 146,276 \cdot \text{speed} - 139,683 \cdot \text{time} + 8,36716 \cdot \text{speed}^2 + 55,3695 \cdot \text{speed} \cdot \text{time} + 79,2667 \cdot \text{time}^2$$



$$\text{Diameter } D_{80} = 404,731 - 162,287 \cdot \text{speed} - 146,547 \cdot \text{time} + 1,89398 \cdot \text{speed}^2 + 40,9883 \cdot \text{speed} \cdot \text{time} + 68,4157 \cdot \text{time}^2$$

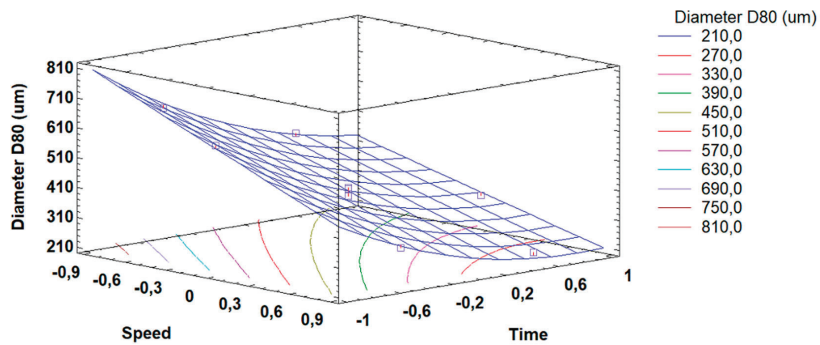


Figure 1. Effect of the process variables on the response surface of the particle diameters. The surface model adjusted the experimental data with a correlation ($R^2 > 99\%$)

Table 1. Real and coded operational conditions of the experimental design.

Sample	Speed	Factor	Time	Factor	Characteristic Diameter (μm)		
	(Rpm)	(x1)	(min)	(x2)	D ₅₀	D ₆₃	D ₈₀
1	250	-0.87	12	-0.5	486	563.6	643.6
2	250	-0.87	26	0.5	315.7	398.6	483.9
3	350	0	5	-1	488.4	558.6	630.9
4*	350	0	19	0	259	336.1	414.7
4*	350	0	19	0	259.1	334.9	412.2
4*	350	0	19	0	242.2	314	387.2
5	350	0	33	1	199.8	256.6	315.3
6	450	0.87	26	0.5	199.7	262.3	327.1
7	450	0.87	33	1	149.4	193.2	238.4

*Central design point in triplicate.

of surface agglomeration of dust by caking, similar to reported by other authors (Suresh *et al.* 2016; Kowalski *et al.* 2016). It is worth highlighting the significant effect in the interaction between the speed and the time, which is only visible in particles of smaller than D_{50} diameters.

Monitoring the distribution of granularity using the Rosin-Ramler-Bennet model (RRB). The figure 2 shows the displacement of the distribution towards smaller size values with the increase of the milling time, in the same way, we can observe the change of shapes in the distribution of peaks (5 min) and “flattened” forms (450rpm - 26min), which indicates that the curves do not follow a normal behavior, therefore, it would not be appropriate to use the normal model. All the points of the experimental design were modeled by the RRB equation (Eq. 2), which adjusted satisfactorily, allowing us to determine two (2) characteristic parameters: the homogeneity index (n) related to the dispersion of the sample, and the diameter D_{63} (characteristic diameter).

Figure 3 presents the adjustment of the particle distribution by the RRB model in flours obtained at constant speed of 250rpm and variable times from 12 upto 26 minutes. It is possible to observe a better data adjustment as the milling time increase. The same effect was observed when the rotation speed was increased from 250rpm to 450rpm generating an improved adjustment of the experimental values. In general, the RRB equation was useful to model the granularity of flours when they do not follow a normal behavior; in addition, the RRB model provides specific information regarding homogeneity, as well as that of a characteristic D_{63} dimension. De la Cruz *et al.* (2015) modeled the granularity of the coffee husk when it undergoes a process of size reduction, concluding that as the milling time increases, the correlation of the RRB model adjusts better.

Method II. Evolution of granularity as a function of the milling energy - Holme model. The milling energy range was increased to a speed of 450rpm and 6.5kJ/g in order to achieve smaller particle sizes. Figure 4 shows the granularity behavior

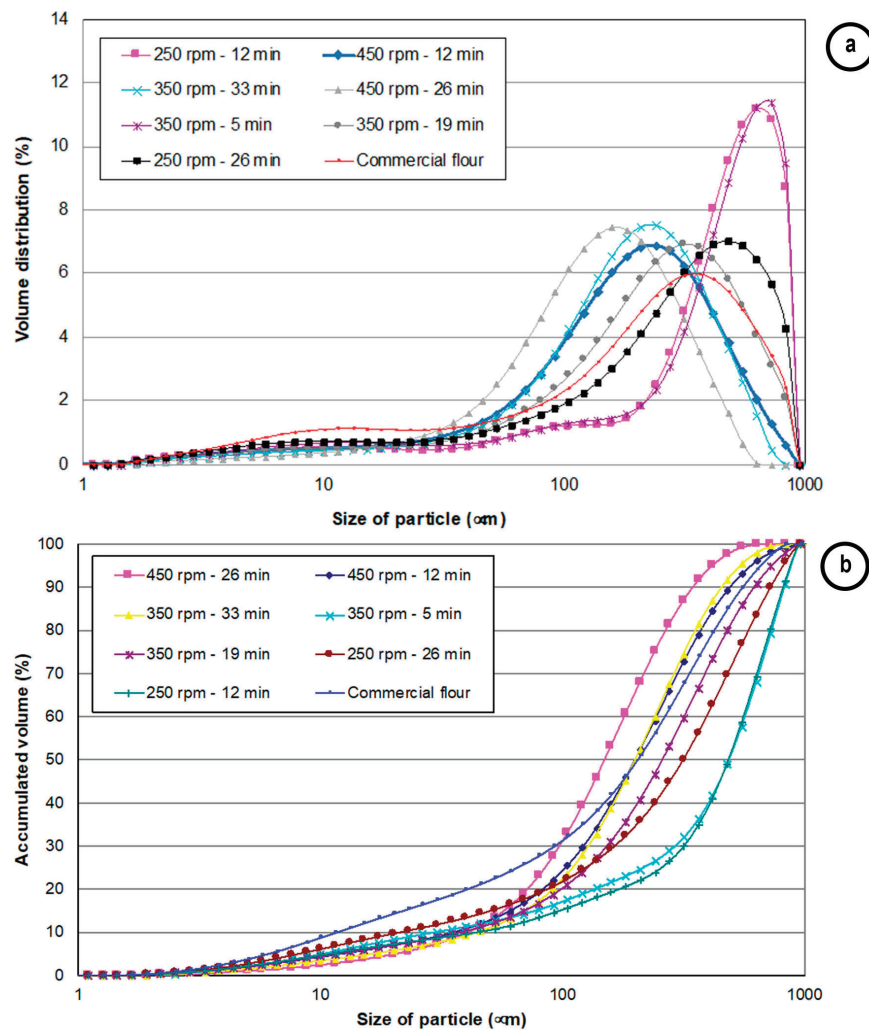


Figure 2. Distribution of sizes of flours determined by Laser Diffraction (SLS), a) frequency distribution; b) cumulative distribution.

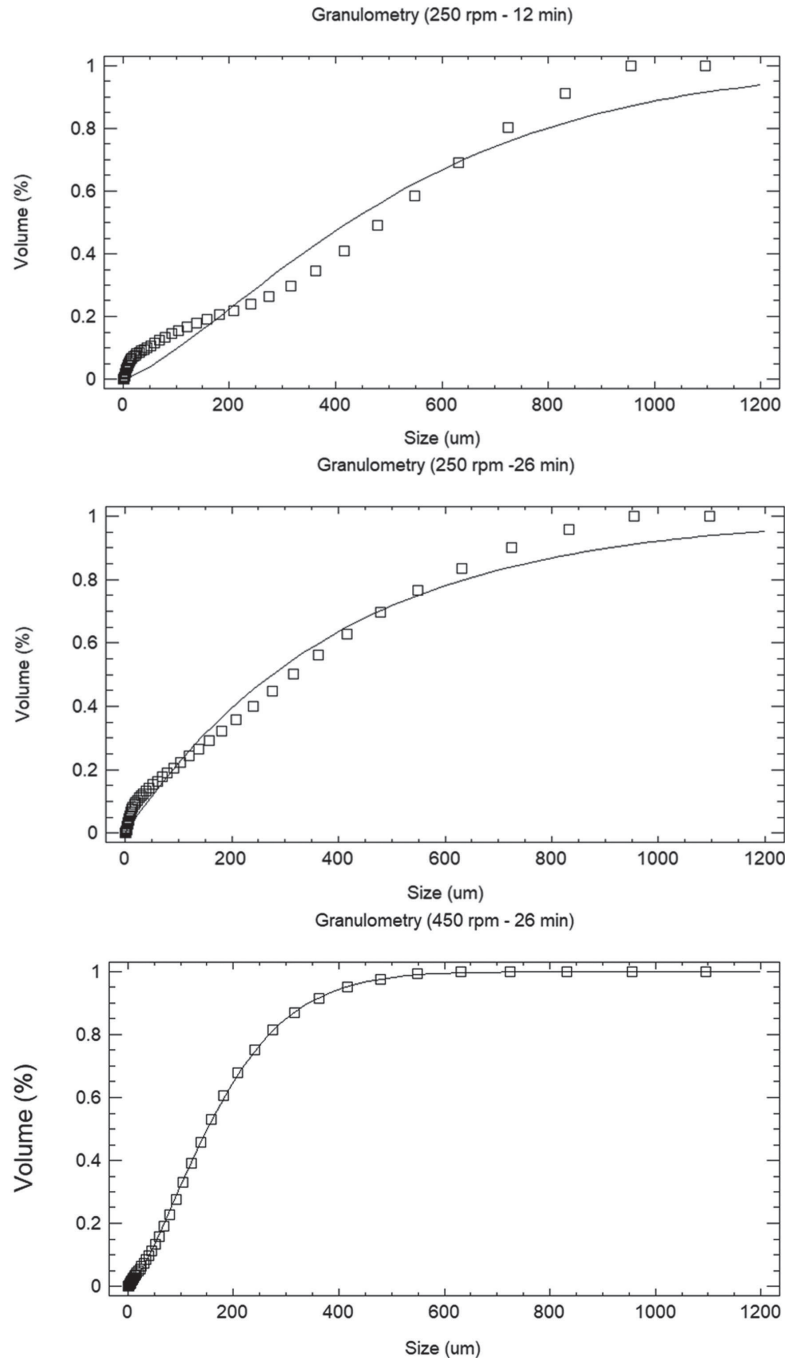


Figure 3. Evolution of the granularity modeled by the Rosin-Ramler-Bennet equation.

in relation to the milling energy. The correlation between the experimental values and those predicted by the Holmes equation is shown in (Equation. 1).

The figure 4 shows the correlation of the different regions where the index (n_1) acquires different values (from 1 to 4). The process comprises two stages: the first part is called grinding and is the initial size reduction until reaching a grain size of $250\mu\text{m}$; the second is milling and in this stage the particle size is reduced at the expense of greater energy expenditure. Figure 4 shows that at the beginning

of the process of size reduction, the value of the index (n_1) is equal to 1, which indicates that the energy needed to carry out the process are independent from the initial size of the particles. In other words, this stage of the process can be modeled conveniently by the Kick equation. If energy is still being delivered to the system, the index value (n_1) increases to 1.5, indicating that this part of the process can be modeled by the Bond equation, which establishes a linear relationship between the energy needed to carry out the spraying and the square root of the particle size.

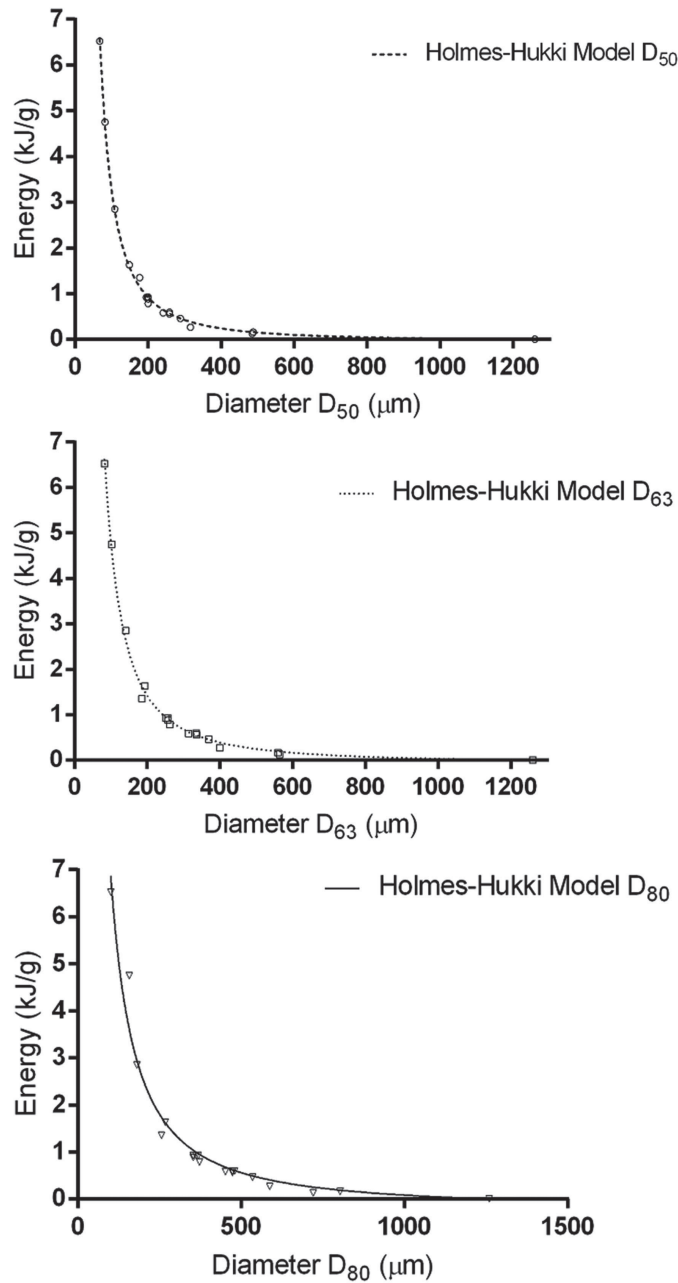


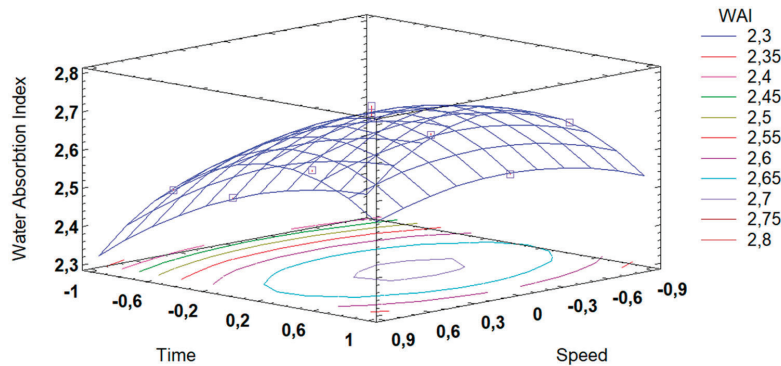
Figure 4. Relationship between the milling energy and the granularity of the flours.

When the value of n_1 is equal to 2 there is a fine grinding process, which is satisfactorily described by the classic Rittinger equation; this model describes how the energy consumed in the process is proportional to the new surface created. Therefore, the process of reducing the size of the amaranth grain can be modeled in real working conditions by using any of the previous models keeping in mind the range of the size that is being studied (Ivanova, 2016; Sanchez *et al.* 2008). The milling process managed to reduce the diameter (D_{50}) to 68 µm, and the index (n_1) takes of 2.7. The difference of the value ($n_1 = 2$) means that the surplus energy is lost in elastic deformations of particles before breaking, friction between particles, and dissipation of energy in the form of noise,

vibrations and heat, among others (Rhodes, 2008). The proposed generalized model (Eq. 1) provided a very satisfactory fit ($r^2 = 0.991$) for the relationship between energy and size.

Changes in the hydration properties (IAA) and solubility (ISA) of the flours. It can be seen in figure 5 that the change in the milling speed and time affect the absorption and solubility properties of the flours. These changes are related not only to the decrease in particle size, but also to the change in the crystalline of the starch. The change in crystalline is due to the disruption of the structure of the starch granules, which is affected by the forces of impact and friction in the planetary mill (Roa *et al.* 2014).

$$\text{WAI} = 2,69773 - 0,00560046 * \text{speed} + 0,0974 * \text{time} - 0,0832882 * \text{speed}^2 + 0,0112009 * \text{speed} * \text{time} - 0,193683 * \text{time}^2$$



$$\text{WSI} = 9,22737 + 4,70144 * \text{speed} + 3,55343 * \text{time} - 0,728443 * \text{speed}^2 + 2,34342 * \text{speed} * \text{time} - 1,70507 * \text{time}^2$$

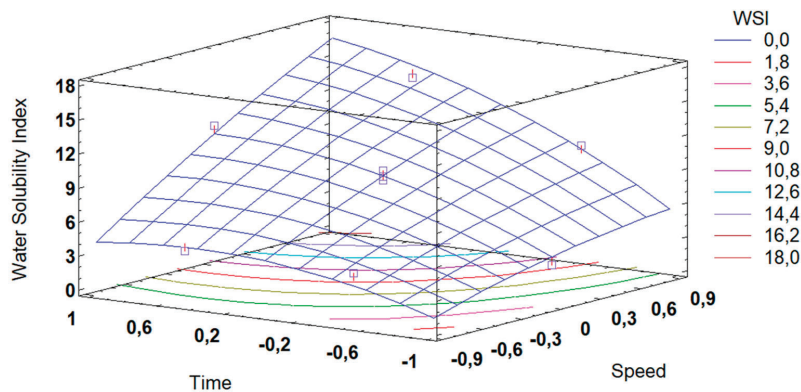


Figure 5. Changes in the hydration properties (IAA) and solubility (ISA) of the flours

The disruption of the grain can be determined by using the X-ray technique, where it is possible to observe how the diffraction peaks disappear as the treatment increases (Ghorbannezhad *et al.* 2016; Dharmaraj *et al.* 2015). Another technique to verify this phenomenon is the (DSC) scanning differential calorimeter. By using this analysis, it's possible to determine how the enthalpy of gelatinization is reduced as the grinding treatment increases (Nawaz *et al.* 2016).

Huang *et al.* (2007) and Zhang *et al.* (2010) obtained similar results when they subjected cassava starch and rice to a process of intense milling, respectively. They found that the disruption of the crystalline zones of the starch granule affects the IAA and ISA properties. Other authors found similar results in the variation of the IAA and ISA indices when they subjected quinoa flour to an extrusion process (Zhang *et al.* 2010; Cerón *et al.* 2016). In summary, it is noted that the ISA index is more sensitive to changes in the process variables (linear effect), compared to the IAA index (quadratic effect). This type of effect is also observed when the flours are subjected to strong shearing and compression processes (Roa *et al.* 2017; Kowalski *et al.* 2016; Tovar *et al.* 2017).

Obtained flour with low granularity and modified hydration properties can be studied using the Holmes and Rosin-Ramler-Bennet models. The indicators generated by these models have an

important physical sense, as the results can be well correlated with each other, offering useful scalable information at an industrial level. Thanks to the advent of high impact mills, flours with modified hydration and solubility properties may be obtained, which can be used in the manufacture of instant foods or as raw materials in nutraceutical products with interesting health benefits.

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