Escuela Agrícola Panamericana, Zamorano

Food Science and Technology Department

Food Science and Technology Major

Graduation Research Project

Effect of Milling Methods on Flow Properties of Whole Pearl Millet Flours

Presented by

Berta Guadalupe del Cid Alvarez

Advisers

Raúl Espinal, PhD.

Carlos Campabadal, PhD.

Kaliramesh Siliveru, PhD.

Honduras, november 2023

Authorities

SERGIO ANDRÉS RODRÍGUEZ ROYO

President

ANA M. MAIER ACOSTA

Vice President and Academic Dean

ADELA M. ACOSTA MARCHETTI

Head of Food Science and Technology Department

HUGO ZAVALA MEMBREÑO

General Secretary

Content

List of Tables

List of Figures

List of Annexes

Abstract

In recent years, there has been interest in the use of pearl millet as flour for human consumption due to its nutritional quality and gluten-free nature. However, studies on the particle level that relates to its processing and handling are limited. The effect of different milling methods (roller mill, pin mill, and hammer mill) on the particle size distribution and flowability of pearl millet flour was evaluated. The physicochemical properties (moisture, water activity, ash content, and damaged starch), particle size distribution, and shape characteristics were investigated and correlated with flow properties. Flowability was measured in terms of bulk, dynamic, and friction flow properties using the FT4 powder rheometer. Pin mill and hammer mill exhibited the highest bulk density (537.83 and 555.26 kg/m^3) and tapped density (641.71 and 670.13 kg/m^3). No differences were found for true density, compressibility index, and Hausner ratio among the three different milling methods. However, when the flour was subjected to an applied normal stress, the compressibility percentage was significantly higher for pin mill and hammer mill. Roller mill had the best permeability at different applied normal stresses and lower energy consumption during the stability test at different blade velocities. Pin mill and hammer mill flours are more susceptible to different air velocities, presenting the best aerated energy (573.33mJ and 544.34 mJ), aeration ratio (2.25 and 2.34), and normalized aeration sensitivity (0.070 and 0.068). The three milling methods showed the same behavior for wall friction angle at different applied pressures. The results of this study showed the importance of milling methods to achieve better flow behavior of pearl millet flour during processing and storage.

Key words: Density, flowability, particle shape, particle size, physicochemical properties.

Resumen

En los últimos años, ha surgido interés por el uso del mijo perla como harina para el consumo humano debido a su calidad nutricional y a que no contiene gluten. Sin embargo, los estudios a nivel de partícula relacionados con su procesado y manipulación son limitados. En el presente estudio se evaluó el efecto de distintos métodos de molienda (molino de rodillos, molino de púas y molino de martillos) sobre la distribución granulométrica y la fluidez de la harina de mijo perla. Se investigaron las propiedades fisicoquímicas (humedad, actividad del agua, contenido de cenizas y almidón dañado), la distribución del tamaño de las partículas y las características de forma; correlacionándolas con las propiedades de fluidez. La fluidez se midió en términos de propiedades de flujo a granel, dinámico y de fricción utilizando el reómetro de polvo FT4. Los valores de densidad aparente (537,83 y 555,26 kg/m^3) y densidad compactada (641,71 y 670,13 kg/m^3) fueron mayores utilizando molino de púas y molino de martillos. No se encontraron diferencias para la densidad real, el índice de compresibilidad y la relación de Hausner entre los tres métodos de molienda. Sin embargo, cuando la harina se sometió a una tensión normal, el porcentaje de compresibilidad fue significativamente mayor en el molino de púas y en el molino de martillos. El molino de rodillos presentó la mejor permeabilidad a diferentes tensiones normales aplicadas y un menor consumo de energía durante la prueba de estabilidad a diferentes velocidades de las cuchillas. La harina obtenida en el molino de púas y molino de martillos fue más susceptible a diferentes velocidades del aire, presentando la mejor energía aireada (573,33mJ y 544,34 mJ), relación de aireación (2,25 y 2,34) y sensibilidad normalizada a la aireación (0,070 y 0,068). Los tres métodos de molienda mostraron el mismo comportamiento para el ángulo de fricción de la pared a diferentes presiones aplicadas. Los resultados de este estudio mostraron la importancia de los métodos de molienda para conseguir un mejor comportamiento de flujo de la harina de mijo perla durante su procesamiento y almacenamiento.

Palabras Claves: Densidad, distribución de partículas, fluidez, forma de partículas, propiedades fisicoquímicas.

Introduction

Pearl millet (*Pennisetum glaucum*) is commonly cultivated in cities of Africa and Asia for food, feed, and forages (Rani et al., 2018). India is the largest producer of various types of millets, commonly referred to as coarse grains. Pearl millet is considered a food security crop due to its ability to thrive under adverse agricultural conditions (Prasanthi & Sireesha, 2022). This grain is rich in various nutrients and non-nutrients, such as phenols. It is high in energy, low in starch, high in fiber (1.2%, mostly insoluble), has a low glycemic index (55), and is gluten-free. Additionally, its protein content usually ranges from 8% to 19%, and its energy is greater than that of sorghum and nearly equal to that of brown rice because the lipid content is generally higher (3 to 6%) (Vanisha S et al., 2011).

In the United States, it has historically been grown mainly for forage and livestock grazing. However, due to its nutritional quality, since 2007 research has focused on its potential use as a grain for human consumption. Pearl millet breeding for food grain has been extensively carried out under the International Sorghum and Millet (INTSORMIL) Collaborative Research Support Program funded by the United States Aid for International Development (USAID). The research efforts for breeding, crop production, and use have been focused at Kansas State University, USDA-ARS, Georgia, and the University of Nebraska-Lincoln (Gulia et al., 2007).

The milling process is primarily carried out to maximize the separation of endosperm, bran, and germ, as well as to reduce the particle size of the grains, thereby facilitating the production of fine flour. Milling of pearl millet is difficult because of its small kernel with a firmly embedded germ along with hard endosperm (Rani et al., 2018). The most common milling methods are hammer mill, roller mill, and pin mill.

The roller mill flow for pearl millet consists of four breaks (BK1, BK2, BK3, and BK4) and four reduction steps (M1, M2, M3, and M4), in each of which the grain is subjected to different pressure forces, aiding in the fracturing of the grain, and achieving separation of the endosperm, bran, and germ; the shafts of the mills used in this method of milling works at 1,760 rpm. In the hammer mill, grinding is performed at 3,450 rpm of the rotor. Finally, in the case of pin mill, the rotor operates at

14,000 rpm. The flour obtained from the three milling methods pass through a series of sieves to achieve physical separation of the flour from the rest of the grain (Acar et al., 2020).

Predicting the behavior of powders during transportation processes is important as the flow properties tend to change, leading to poor quality products. The use of the powder rheometer FT4 aids in understanding the flow properties of flours, generating quick, repeatable, and sensitive measurements with a high degree of automation (Freeman Technology, 2016). The Freeman FT4 powder rheometer consists of blades, pistons, and shear heads that could be rotated and simultaneously moved transversely down into a powder bed while axial force and rotational force are measured (Freeman, 2007).

At present, similar studies exist only on other grains such as sorghum, rice and teff flours. However, there are no studies on flow properties of pearl millet flour. Characterization of these properties could help as a basis for the design of processing and handling equipment and optimization of pearl millet milling process. For this reason, the present research has the following objectives:

Characterize physicochemical properties of whole pearl millet flour.

Evaluate the bulk flow properties of the flour.

Evaluate the dynamic flow properties of the flour.

Assess the friction properties of the flour.

Materials and Methods

Study Location

The study was conducted in the Grain and Particle Processing and Safety Research Laboratory at Kansas State University. Grain milling was performed in the Milling Laboratory.

The first phase of this project consisted of grain milling using three different types of mills (roller, pin, and hammer mills). The second phase included the analysis of physicochemical properties (water activity, moisture content, damage starch, ash content and particle size distribution and shape characteristics), bulk flow properties (bulk density, tapped density, true density, compressibility index, hausner ratio, compressibility and, permeability), dynamic flow properties (aeration, stability), and friction properties (wall friction angle).

Sample Preparation

The pearl millet grains (5 kg) were obtained from Organic Sphere, India. Upon receiving the grains, samples of 400 g were separated for each repetition.

Milling

Three types of mills were used in this study: roller mill (Model 915, Ross Machine and Mill Supply, Oklahoma City, OK), pin mill (Model UT-03, Bauermeister, Inc., Memphis, TN), and hammer mill (Model 22115, Bliss Industries, Ponca City, OK) that is equipped with 24 hammers set with a 0.40 mm gap between hammer and screen). Approximately 400 grams of pearl millet were ground in triplicates.

For the roller mill, the grains were allowed to pass between a combination of four breaks and reduction rolls with clearances of 0.46, 0.20, 0.10 and 0.05 mm successively. After roller milling, the separated bran was milled using a pin mill and powdered bran was added back to the roller milled flour fractions to make it a whole grain flour. Samples were milled in triplicate for each milling method to obtain whole pearl millet flour.

Moisture Content of Grains

The moisture content of the grains was determined by drying 10 g of the samples in a circulating air oven at 105 °C for 24 hours (American Society for Agricultural Engineers [ASAE], 1994). The resulting moisture content was 13%.

Moisture Content of Flour

The moisture content of pearl millet flour was determined following the methodology established by the American Association of Cereal Chemists (AACC) standard methodologies, method 44-15.02 (American Association of Cereal Chemists [AACC], 1999).

Initially, the moisture dishes were dried at 130 °C, then cooled in a desiccator. Each sample was then homogenized, and 2 to 3 g were taken in moisture dishes in triplicates. The dishes were placed inside the hot air oven using lids were under the dishes. They were heated for exactly 60 minutes at 130 °C. After the time had elapsed, the dishes were removed from the oven and transferred to the desiccator with their lids closed. Once room temperature was reached (30-40 min normally), the dishes were weighed, and the weight loss was determined as moisture (Equation 1).

$$Moisture\ content\ \% = \frac{\text{moisture loss in grams}}{\text{original weight of sample}} * 100 \qquad [1]$$

Ash Content

The ash content was determined following the methodology established by the American Association of cereal chemists (AACC), method 08-01.01 (American Association of Cereal Chemists [AACC], 2020). Methodology described below:

3 ± 0.005 g of well-mixed sample was weighed into a completely dry ashing crucible.

The crucibles were shifted to muffle furnace set for 550 °C for 24 hours. It was incinerated until light gray ash or constant weight was obtained. It was then cooled in the desiccator and weighed shortly after reaching room temperature. The ash content was determined using the Equation 2.

$$Ash\ content\ \% = \frac{weight\ of\ residue}{sample\ weight} * 100 \qquad [2]$$

Water Activity

An Aqualab vapor sorption analyzer (VSA) (Decagon Devices, Inc., Pullman, WA) was used to obtain the water activity of the flour (Decagon Devices, 2016).

Particle Characterization and Size Distribution

Particle size distribution and shape characterization of pearl millet flours were carried out using the Malvern Morphologi G4 SE (Malvern Instruments, Malvern, UK, 2023). A standard sample of $7mm^3$ was used for each replicate, and from this, 10,000 particles were imaged at 10x magnification. The PSD was generated on a number basis with focus on diameter fractions D_{10}, D_{50}, and D_{90}. The subscript in each fraction represents the percentage of particles with diameters less than the specified value. Particle shape characteristics, including circularity, elongation, convexity, and aspect ratio, were also measured using the same equipment.

Particle Size Distribution

The particle size distribution was determined using an Alpine jet sieve (Hosokawa Micron, E200 LS). For this, a sample of 100 g per repetition of pearl millet flour was placed using the different sieves in ascending order, as described below: 25 µm, 53 µm, 75 µm, 90 µm, 125 µm, 150 µm, 180 µm, 212 µm, and 250 µm (Doddabematti Prakash et al., 2023).

Bulk Density

To measure the bulk density, a graduated cylinder was used. The weight of the sample was taken after filled the cylinder with each sample. Following the Winchester Bushel method (United States Departmment of Agriculture [USDA], 2016), (Equation 3).

$$Bulk\ density\ (kg/m^3) = \frac{Sample\ weight}{Volume} \qquad [3]$$

Tapped Density

Tapped density or compacted bulk density is the ratio of the mass to the volume of the sample after the flour has been tapped a fixed number of times. The Auto tap density analyzer (Quantachrome Instruments, Boynton Beach, FL) was used to measure the tapped density (Equation 4). Tapped density is an indicator of how the grains or flour will compress during storage or transportation. To measure it, a known volume cylinder was filled with each sample, and then the cylinder was tapped 750 times (260 taps per minute) (Doddabematti Prakash et al., 2023).

$$Tapped\ density\ (kg/m^3) = \frac{Sample\ weight}{Tapped\ volume} \qquad [4]$$

Compressibility Index (CI) and Hausner Ratio (HR)

Compressibility index and Hausner ratio are the flowability indicators that measures the propensity of a powder to be compressed, reflecting the relative degree of interarticulate interactions (Bian, 2014). These two parameters were determined using the bulk density and tapped density, as shown in Equations 5 and 6.

$$Compressibility\ index\ (\%) = \frac{tapped\ density - bulk\ density}{tapped\ density} * 100 \qquad [5]$$

$$HR = \frac{tapped\ density}{bulk\ density} \qquad [6]$$

True Density

The true density values were obtained using the Automatic Density Analyzer, ULTRAPYC 1200e equipment, according to the procedure described by (Choudhury & Gautam, 1999). Density values for each sample were taken in triplicates and presented as the mean ± SD.

Damage Starch

Damage starch was measured using the SDmatic TM (Chopin Technologies, Villeneuve la Garenne, France) with the reference of the approve method AACC 76.33.01 (American Association of Cereal Chemists [AACC], 2007). The percentage of damage starch was recorded.

Flow Property Determination

A FT4 powder rheometer, (Freeman Technologies, Tewkesbury, Gloucestershire, UK) was used to quantify the flow properties of pearl millet flour. Descriptions of this equipment and the methodology of its use can be found in (Bian et al., 2015; Divya & Ganesh, 2019). Flow properties were measured following standard methodology for the use of the FT4 powder rheometer, including dynamic flow (stability and variable flow rate, and aeration), shear (wall friction), and bulk (permeability and compressibility) flow tests (Barretto et al., 2022). The FT4 powder rheometer system consists of a vertical glass sample container and a rotating blade (Leturia et al., 2014).

Stability and Variable Flow Rate

The parameters taken for the stability and flow rate variable include specific energy (SE), flow rate index (FRI), basic flow energy (BFE), and stability index (SI) (Divya & Ganesh, 2019).

SI is a measure of the powders tendency to change its form due to flow (Equation 7). Stability test involved running seven test cycles (from test 1 to 7) of the blade moving downward at 100mm/s tip speed across the vessel (Barretto et al., 2022). According to Leturia et al. (2014), SE is the energy per gram needed to displace conditioned powder during upwards testing. It represents a measure of how the powder will flow in a low stress environment, which is created by an upward movement of the blade in a clockwise direction. FRI is the factor by which the flow energy changes when the blade tip speed is reduced by a factor of 10. It evaluates the sensitivity of the powder to flow rate. The program begins by subjecting the powder to a standard flow rate of 100mm/s (Test 8), followed by measuring at 70 (Test 9), 40 (Test 10), and 10m/s (Test 11) blade speeds. From the flow energy, the flow rate index was calculated using the Equation 8 (Freeman, 2007). BFE corresponds to the stabilized flow energy (Test 7), which represents the energy needed to displace a conditioned powder sample during downward testing of the blade through the sample (Barretto et al., 2022).

$$SI = \frac{Total\ energy\ consumed\ at\ test\ 7}{Total\ energy\ consumed\ at\ test\ 1} \qquad [7]$$

$$FRI = \frac{Flow\ energy\ at\ test\ 11\ (10mm/s)}{Flow\ energy\ at\ test\ 8\ (100mm/s)} \qquad [8]$$

Aeration

The aeration test involved introducing an air flow at different velocities from the base of the container, to measure its effect on the flow properties of the flour. The flow energy was measured at different velocities (0-10 mm/s). An 85 ml sample was placed in a 50 mm cylindrical vessel fitted with a porous plate and a mass flow controller. From this test, three parameters were measured: aeration energy (AE) is the flowability energy at maximal air velocity (10 mm/s); aeration ratio (AR) designates the factor by which the basic flowability is reduced when aerating the powder bet at maximal air velocity of 10 mm/s (Equation 9); and the normalized aeration sensitivity (NAS) is a measure of powder sensitivity to aeration, specially at small air velocities. It is defined as the maximal difference in normalized test energy between the two consecutive points of the test, divided by the change in air velocity. NAE was calculated as the ratio between total energy at investigated air velocity and total energy in the absence of air flowing (Gnagne et al., 2017).

$$AR = \frac{Flowability\ energy\ at\ 0 mm/s\ (air\ velocity)}{Flowability\ energy\ at\ 10\ mm/s\ (air\ velocity)} \qquad [9]$$

Compressibility

Compressibility refers to the change in density of a material when subjected to an applied normal stress and is associated with the mechanical compaction that occurs during handling, storage, and transportation (Bian et al., 2015). Compressibility tests were performed using a vented piston assembly that applied increasing normal stress to the sample. In this study, nine normal stresses were applied, ranging from 0.5 kPa to 15 kPa. The equipment used recorded the conditioned bulk density (CBD) and the change in volume after compression (CPS). Finally, the compressibility index (CI) was calculated using the Equation 10:

$$CI\ (\%) = \frac{Density\ after\ compression}{Conditioned\ bulk\ density} * 100 \qquad [10]$$

Permeability

Compression of flours reduces their porosity and ability to aerate. As flours are subjected to pressure during storage or transportation, it is important to understand their permeability to develop effective discharge strategies (Bian et al., 2015). The resistance to the airflow through the flour was quantified using a vented piston assembly of the FT4 powder rheometer. As the piston contacted the flour, the air pressure drop in response to the applied normal stress was recorded. The normal stress was applied from 1 to 15 kPa. Similarly, air was introduced to the powder at 2mm/s during the permeability test (Babu et al., 2018; Barretto et al., 2022).

Wall Friction

This property measures the ability of powders to flow (steady-state flow) as a function of the surface material of the containing vessel. A wall friction head attached to the FT4 powder rheometer imposed vertical and rotational stresses on the samples (Bian et al., 2015). In this test, the powder rheometer measured the frictional force acting between the powder and the surface material (Barretto et al., 2022). The torque required to maintain this rotational momentum was measured as the shear stress. The wall friction angle (φ), was calculated from the relationship between normal stress ($\sigma\omega$) and shear stress ($\tau\omega$), using the Equation 11:

$$\varphi = tan^{-1}\left(\frac{\tau\omega}{\sigma\omega}\right) \qquad [11]$$

Data Analysis

The experimental data were analyzed using Statistical Analysis Software (SAS) 9.3 (SAS Institute, Cary, NC). Three milling methods were studied in triplicate, resulting in a total of 9 experimental units. A completely randomized design (CRD) was used; only compressibility, permeability, and wall friction were studied using a completely randomized design in a factorial arrangement, at different applied normal stresses. Analysis of variance, Duncan means separation and LSMEANS were used to estimate significant differences ($p < 0.05$) between the treatments. Pearson´s correlation procedure was also done to determine the degree of association between measured variables.

Results and Discussion

The effect of milling on proximate composition of whole pearl millet flour is presented in Table 1. The flour obtained from the three different milling methods showed a moisture percentage below the maximum recommended limit for food intended for human consumption (13%) (Food and Agriculture Organization of the United Nations [FAO] & World Health Organization [WHO], 2019). However, roller milling (10.35%), and pin milling (10.22%), exhibited statistically higher moisture content than hammer milling (8.72%); in a study conducted by Suma and Urooj (2011), it was reported that pearl millet flour had a moisture percentage ranging from 9.6 to 10.1, pulverizing the grains in pin mill to obtain whole flour. Tortoe et al. (2019) also mention that pearl millet flour using hammer mill presented a moisture content of 9.2 to 10.4; similar to the results obtained in the present study. Furthermore, in the CODEX ALIMENTARIUS (2019) standard, it is mentioned that whole pearl millet flour must have a maximum moisture content of 13%, a value higher than those obtained in this study.

Similarly, results were obtained for water activity statistically higher for roller mill and pin mill (0.46 and 0.40, respectively). These values are below those reported by Tortoe et al. (2019) (0.595-0.567), who also mentioned that moisture and water activity are important parameters that greatly influence the shelf life and quality of flours.

The differences obtained for water activity and moisture content are attributed to the milling time, as hammer milling requires less time, the flour is less time in contact with the environment compared to the other milling methods evaluated (roller mill and pin mill). Due to the characteristics of the flour (low moisture and water activity), it is highly hygroscopic, so it easily absorbs moisture from the environment and the longer the exposure time, the more moisture it absorbs. Another factor that influenced was the heat generated in the milling process; hammer mill generates more heat, thus producing flour with lower moisture content compared to roller mill and pin mill. High moisture and water activity values promote biochemical reactions and support the growth of microorganisms, which can lead to product spoilage and subsequent loss of quality. Based on the above, despite of the differences found, flours were shelf stable as the water activity is below 0.6 and moisture is below safe storage level (13%) (MEPBA et al., 2009; Suma P & Urooj, 2015).

Table 1

Physicochemical analysis values (mean ± SD) of pearl millet flour using different milling methods.

Test variable	Milling Method			
	Roller mill	Pin mill	Hammer mill	CV (%)
Moisture (% wet basis)	10.35 ± 0.52^a	10.22 ± 0.79^a	8.72 ± 0.15^b	5.64
Water activity	0.46 ± 0.03^a	0.40 ± 0.04^a	0.32 ± 0.003^b	6.81
Ash (%)	1.25 ± 0.07^a	1.26 ± 0.05^a	1.35 ± 0.05^a	4.44
Damaged starch (%)	7.30 ± 0.59^a	7.01 ± 0.38^a	7.28 ± 0.07^a	5.63

Note. Mean values with different small case letters in the same row differ significantly among different milling methods ($p<0.05$). SD: Standard Deviation, CV: Coefficient of Variation.

Ash represents the non-combustible fraction of the sample, i.e., minerals. The ash content for roller milling, pin milling, and hammer milling was 1.25%, 1.26%, and 1.35%, respectively (Table 1). Statistically, there were no significant differences. The values found were similar like those reported by Tortoe et al. (2019) (0.9 to 1.5%) and Balasubramanian et al. (2014)(1.14 to 1.35%).

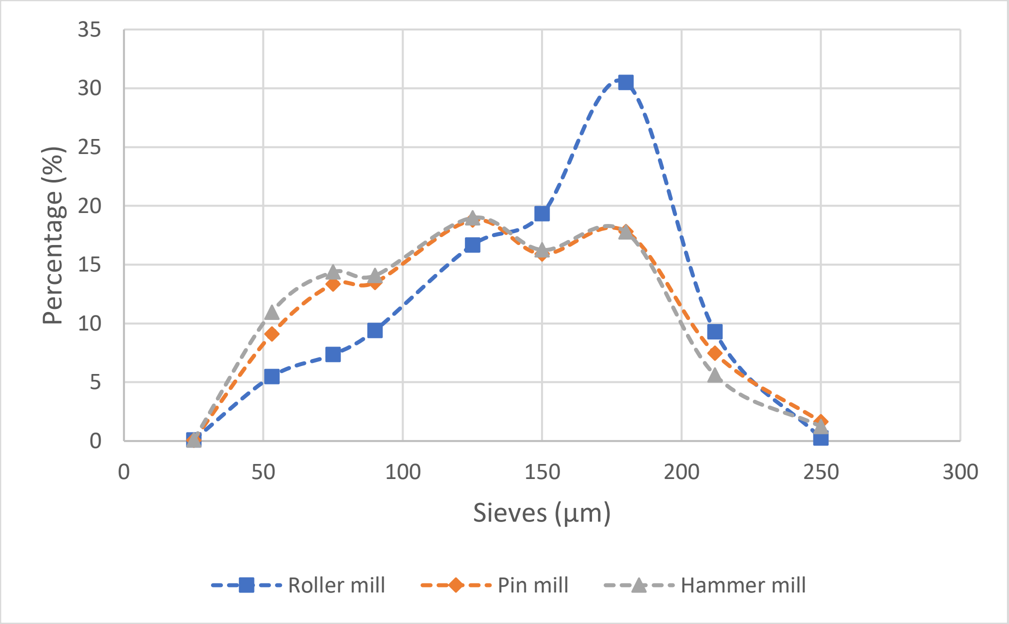
Regarding the percentage of starch damage, as shown in Table 1, no significant differences were found among the three different types of milling evaluated. In a study conducted by Doddabematti Prakash et al. (2023), for proso millet, they found that pin mill generated a higher percentage of damage starch; however, in the present study no significant differences were found between the different milling methods evaluated. On the other hand, Vidya et al. (2012) reported an average starch damage of 5.3% for pearl millet flour, a value that remains lower than what was found in the present study (7.01 to 7.3%). Pulivarthi et al. (2022) mentioned that as the fraction of smaller-sized particles increases, a higher percentage of starch damage is expected. This is attributed to greater mechanical damage or force applied during the process, as well as the heat generated by impact/shear force during particle size reduction. However, as can be seen in Figure 1, despite roller milling had a lower fraction of smaller-sized particles, no significant differences were found in the percentage of starch damage compared to the other treatments.

Particle Size Distribution and Shape Characteristics

The Particle Size Distribution (PSD) of pearl millet flours obtained from different milling methods is showed in Table 2 and Figure 1. PSD shows that pin mill and hammer mill produced a trimodal distribution of particles while the roller mill produced a unimodal particle distribution. The reason is that for roller mill, the grain was processed step by step allowing gradual reduction of particle size, while for the pin mill and hammer mill, the grains were just ground in one single run which does not allow for a gradual reduction of particle size. Therefore, particle size reduction is not controlled (Doddabematti Prakash et al., 2023). According to Food and Drug Admministration [FDA] (1998), not less than 98% of the flour should pass through a cloth having openings not larger than those of woven wire cloth designated as "212 µm". Roller milling and hammer milling met this parameter, as both had a particle percentage below 212 µm of 98.08%. On the other hand, pin milling had a percentage of 95.97%.

Figure 1

Particle size distribution of pearl millet flour.



The hammer mill and pin mill exhibited smaller particle sizes compared to the roller mill. As can be observed in Table 2, 10 percent of the particles observed for the pin mill and hammer mill had diameters smaller than 33.32 µm and 41.37 µm, respectively. Likewise, the roller mill showed 50 percent of particles with diameters smaller than 159.87 µm, a value significantly higher than that observed for the pin mill and hammer mill at the same percentage of particles (103.04 and 139.30 µm, respectively). However, no significant differences were found in the diameter when observing 90 percent of the particles for the flour obtained from the different milling methods. In a study on teff flour, conducted by Barretto et al. (2022), found no significant differences between the particle size at 10% and 50% of the particles, using roller mills, pin mills, and hammer mills. Only for 90% found that roller and hammer mill had the highest values of the particle size.

In terms of the shape characteristics, circularity exhibited significant differences, while convexity, elongation, and aspect ratio showed no significant differences among the flour particles from the three mills. The circularity values for pearl millet flour ranged from 0.72 to 0.76. Olson (2011) mentions that the further away from a perfectly round, smooth circle a particle becomes, the lower circularity value. Circularity is a dimensionless value. As can be observed in Table 2, pin mill (0.76) and hammer mill 0.74) presented significantly rounder particles compared to roller mill (0.72). Zhang and Ghadiri (2002) mention that larger particles tend to be more rounded and circular due to the forced attrition they experience at the edges during the milling process. However, there is a percentage of particles that tend to chip and exhibit a more irregular shape. Attrition force makes large particles dominating the distribution rounder compared to finer particles.

The convexity of a particle is a measure of the overall roundness of the particle. A rounded particle should not contain too many concave corners, and a particle with many concave corners is not considered rounded (Mora & Kwan, 2000). Convexity is a two-dimensional measure of the roughness of the particle's edge, which relates the real perimeter of the particle to the convex hull perimeter. This results in convexity values close to 1 when the particles have a smooth surface, regardless of their shape, and values close to 0 when the particles have rough or pointed edges (Barretto et al., 2022). As it can be observed in Table 2, the convexity values of the three types of flour ranged from 0.95 to 0.96, indicating that the particles have very smooth edges. The reason for this is that the particles subjected to attrition experience chipping in the edges and corners making them rounder and more circular (Siliveru et al., 2016; Zhang & Ghadiri, 2002).

On the other hand, as showed in Table 2, no significant differences were observed in elongation and aspect ratio. Merkus (2009) mentions that aspect ratio is the ratio of the maximum to minimum Feret diameter. Elongation and aspect ratio are two shape descriptors that are related, and it is defined by the formula $1 - aspect\ ratio$. Therefore, when no significant differences are observed in one parameter, the same result is expected for the other parameter. The closer the aspect ratio value is to 1, the lower the elongation value (closer to 0), indicating that the particles are less elongated.

The observed differences in shape descriptors and particle size distribution are due to the type of milling applied. Hammer mills reduce particle size through impact. They consist of a series of hammers hinged on a central shaft which is enclosed within a rigid metal case (Barretto et al., 2022). Roller mills, on the other hand, utilize the principle of compression for particle size reduction. Metal rollers are mounted horizontally with an adjustable gap (Aulton & Taylor, 2018; Vukmirovic et al., 2016). Finally, pin mills perform size reduction by continuous impact and shearing of the pins, also have a rotor-stator configuration with a faster tip speed compared to hammer mills (Barretto et al., 2022; Chen et al., 2020).

Table 2

PSD and shape characteristics (mean ± SD) of pearl millet flour using different milling methods.

	Milling Method			
	Roller mill	Pin mill	Hammer mill	CV (%)
PSD				
D_{10}(µm)	62.32 ± 8.22^{a}	33.32 ± 0.85^{b}	41.37 ± 1.85^{b}	10.71
D_{50}(µm)	159.87 ± 7.10^{a}	103.04 ± 4.13^{c}	139.30 ± 0.62^{b}	3.55
D_{90}(µm)	246.73 ± 8.58^{a}	242.10 ± 4.84^{a}	222.87 ± 19.11^{a}	5.23
Shape characteristics				
Circularity (Ci)	0.72 ± 0.03^{b}	0.76 ± 0.01^{a}	0.74 ± 0.01^{ab}	2.35
Convexity (Co)	0.95 ± 0.02^{a}	0.96 ± 0.00^{a}	0.96 ± 0.01^{a}	1.15
Elongation (E)	0.32 ± 0.01^{a}	0.31 ± 0.00^{a}	0.32 ± 0.01^{a}	2.23
Aspect Ratio (AR)	0.68 ± 0.01^{a}	0.69 ± 0.00^{a}	0.68 ± 0.01^{a}	1.03

Note. Mean values with different small case letters in the same row differ significantly among different milling methods ($p<0.05$). SD: Standard Deviation, PSD: Particle Size Distribution, CV: Coefficient of Variation.

Bulk Flow Properties

The bulk flow properties of pearl millet flour using three different milling methods are presented in Table 3. Pulivarthi et al. (2022) mentioned that bulk density provides a better understanding of packaging and designing various conveying equipment. On the other hand, tapped density can be used to measure the compaction of flour in the absence of consolidation pressure. Hammer-milled and pin-milled pearl millet flour showed statistically higher values of bulk density (555.26 kg/m^3 and 537.83 kg/m^3) and tapped density (670.13 kg/m^3 and 641.71 kg/m^3) compared to the flour obtained from roller milled (505.25 kg/m^3 and 604.46 kg/m^3). In a study conducted by Doddabematti Prakash et al. (2023), in proso millet flour, found the highest values of aerated bulk density and tapped density for hammer mill (615 and 690 kg/m^3). In another study on teff flour conducted by Barretto et al. (2022), similar results were reported, with the highest values for bulk density (548 kg/m^3) and tapped density (804.33 kg/m^3) obtained using the hammer mill, which are like the results obtained in this study.

The relationship between aerated and tapped densities explains the degree of powder cohesiveness. Additionally, bulk density tends to be lower than tapped density for cohesive powders. Conversely, free-flowing powders have a lower tapped density than aerated bulk density, as weaker cohesion allows for relative particle displacement, fitting more closely and creating smaller voids. The kinetic energy of individual particles in free-flowing powder during aeration is greater and faster than of simultaneously reorganizing particles during tapping (Barretto et al., 2022).

True density is the density of the solid part of solids without voids in particles and intergranular spaces. By measuring true density, the porosity of bulk samples could be calculated. The porosity of a sample indicates the potential permeability and aeration through the bulk solids (Bian, 2014). According to the results obtained, no significant differences were found in the true density of the flour obtained under the three different milling methods.

Moreover, no significant differences were found among the values of compressibility index (CI) and Hausner ratio (HR) for the flour obtained from the three different milling methods. Powder bridge strength and stability are explained by CI, while interparticulate friction is measured by HR. Poor flow behavior is exhibited by higher CI or higher HR (Barretto et al., 2022). The classification of powder flow performance based on CI and HR is shown in Table 4 (Singh & Kumar, 2012). Based on this classification, roller milling, pin milling, and hammer milling fall into the category of fair flow ($16 \leq$ CI ≤ 20 and $1.19 \leq$ HR ≤ 1.25). However, Jan et al. (2018) mention that although HR and CI are useful indicators of general flowability of powders for characterization purposes, should not be used for practical applications, because the flour is exposed to aeration and compaction within processing units and storage bins.

Table 3

Bulk flow properties (mean ± SD) of pearl millet flour using different milling methods.

Bulk flow properties	Milling Method			
	Roller mill	Pin mill	Hammer mill	CV (%)
Aerated bulk density (kg/m³)	505.25 ± 17.8^b	537.83 ± 4.67^a	555.26 ± 7.20^a	2.14
Tapped bulk density (kg/m³)	604.46 ± 9.33^b	641.71 ± 24.84^a	670.13 ± 6.91^a	2.48
True density (kg/m³)	$1{,}449.02 \pm 3.20^a$	$1{,}446.70 \pm 4.00^a$	$1{,}444.85 \pm 1.73^a$	0.22
Compressibility index (%)	16.43 ± 1.67^a	16.12 ± 2.62^a	17.14 ± 0.24^a	10.87
Hausner Ratio (HR)	1.20 ± 0.03^a	1.19 ± 0.03^a	1.21 ± 0.01^a	2.08

Note: Mean values with different small case letters in the same row differ significantly among different milling methods ($p<0.05$). SD: Standard Deviation, CV: Coefficient of Variation.

Table 4

Classification of flowability (adapted from Singh and Kumar 2012).

Flow character	Compressibility index	Hausner Ratio
Excellent flow	≤ 10	1.00-1.11
Good flow	11-15	1.12-1.18
Fair flow	16-20	1.19-1.25
Passable flow	21-25	1.26-1.34
Poor flow	26-31	1.35-1.45
Very poor flow	32-37	1.46-1.59
Very, very poor flow	> 38	> 1.60

The change in volume after compression was also obtained from the powder rheometer FT4 for all flours. This process involved applying a normal stress to the powder using a vented piston, which forced the particles to be closely packed. This test increased the bulk density as the same number of particles was packed into a smaller volume (Barretto et al., 2022). In Figure 2, the compressibility percentage of the flours is shown versus the applied normal stress. As can be observed, all the flours follow the same upward trend with the increasing applied normal stress from 0.5 to 15 kPa. However, pin milled, and hammer milled exhibit a more similar trend compared to roller milled, showing a higher compressibility percentage, this can also be observed in Annex A . Barretto et al. (2022), mention that higher compressibility percentages indicate a greater percentage change in volume for a given applied normal stress. According to this, the flour obtained from pin milled and hammer milled is more compressible than roller milled when subjected to the same applied normal stress. The compressibility of pearl millet flour, using different milling methods, is comparable to that of wheat flour, as reported by (Siliveru et al., 2017). However, in a study conducted by Barretto et al. (2022) on teff flour, the best compressibility values were found using the roller and pin mill. The compressibility percentage is also related to the size and shape of the particles. As mentioned by Hlosta et al. (2016), generally, cohesive flours with smaller particle sizes (less than 30 µm) tend to have a higher compressibility percentage.

Figure 2

Compressibility of pearl millet flour using different milling methods.

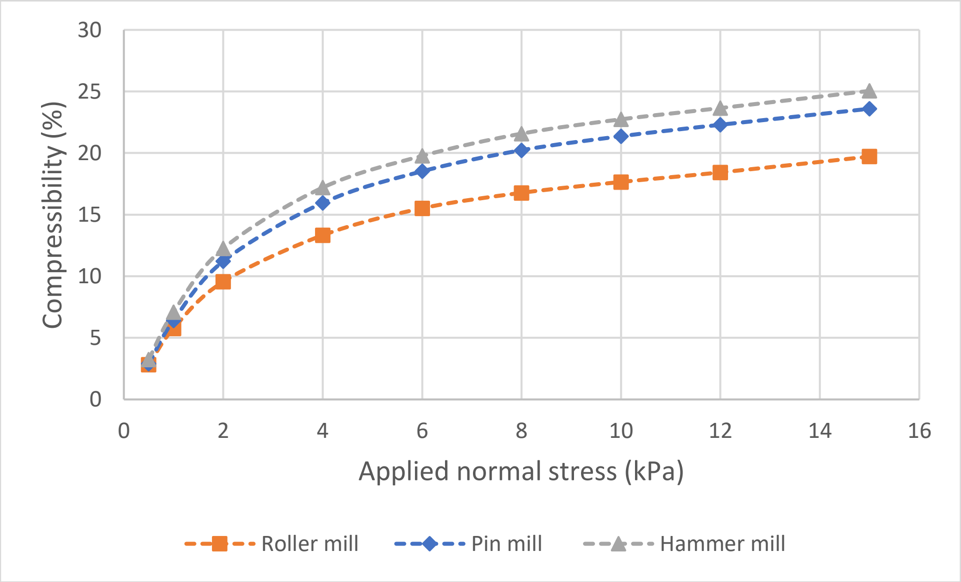
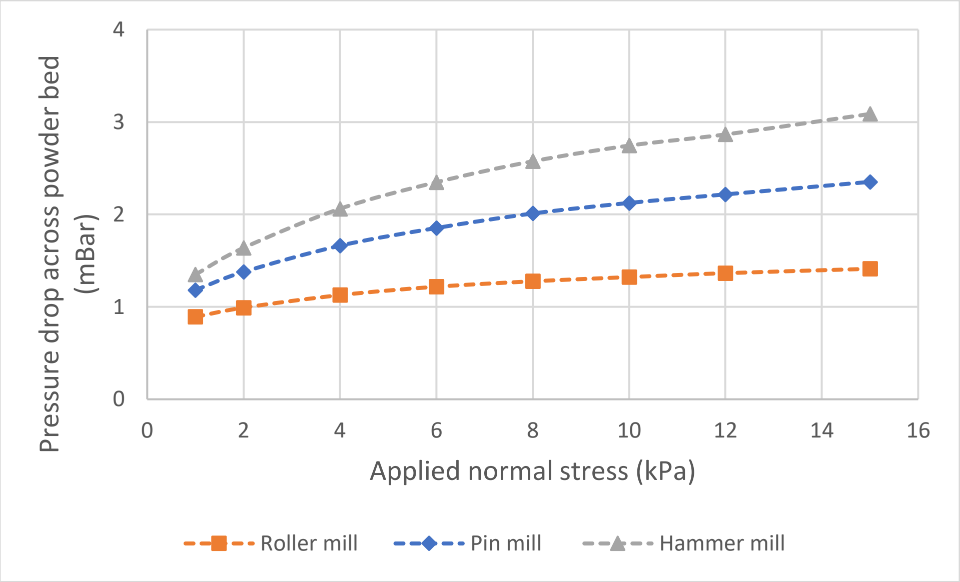


Figure 3 shows the pressure drop across powder bed to achieve a constant air velocity of 2 mm/s while the powder was consolidated at increasing normal stresses. As can be observed, it tends to increase with increasing applied normal stress, being more significant the change in pin mill and hammer mill. This indicates that permeability is influenced by the shape of the particles and particle size distribution (Fu et al., 2012). Roller milled generates the lowest pressure drop, making it the most permeable sample. On the other hand, hammer-milled pearl millet flour required the highest pressure drop (3.09 mBar at 15 kPa) to maintain the air flow adjustment. The behavior of pressure drop across powder bed can also be observed in Annex B. The results obtained are like those reported by Barretto et al. (2022) who found that flour obtained using roller mill presented the highest permeability. However, they also reported that pin mill generated lower permeability, which differs from the findings of the present study, where the flour obtained through the hammer mill demonstrated lower permeability.

Figure 3

Pressure drops across powder bed of pearl millet flour using different milling.



Dynamic Flow Properties

Table 5 presents the dynamic flow properties for pearl millet flour obtained through the three different milling methods, which were measured using the powder rheometer FT4. Subsequently, Figure 4 illustrates the flow energy measurements over 7 identical tests at a tip speed of 100 mm/s, followed by 4 successive tests at different speeds (100, 70, 40, and 10 mm/s). As observed in Figure 4 and Annex C, the flour obtained from the three different milling methods was quite stable in terms of changes (agglomeration, segregation, or breakage) during the first 7 tests. Likewise, Annex D shows that the roller mill had the lowest total energy consumption as the blade speed was reduced. In a study conducted on teff flour by Barretto et al. (2022), it was found that roller mill exhibited better stability, followed by pin mill, and hammer mill at different blade velocities, similar to the results obtained in the present study. Stability index for roller mill, pin mill, and hammer mill were 0.99, 1.02, and 1.02, respectively, and these values did not show significant differences among them. Furthermore, these values fall within the normal range for flours (0.9-1.1), while any value above or below this range would be categorized as unstable (Bian et al., 2015).

Table 5

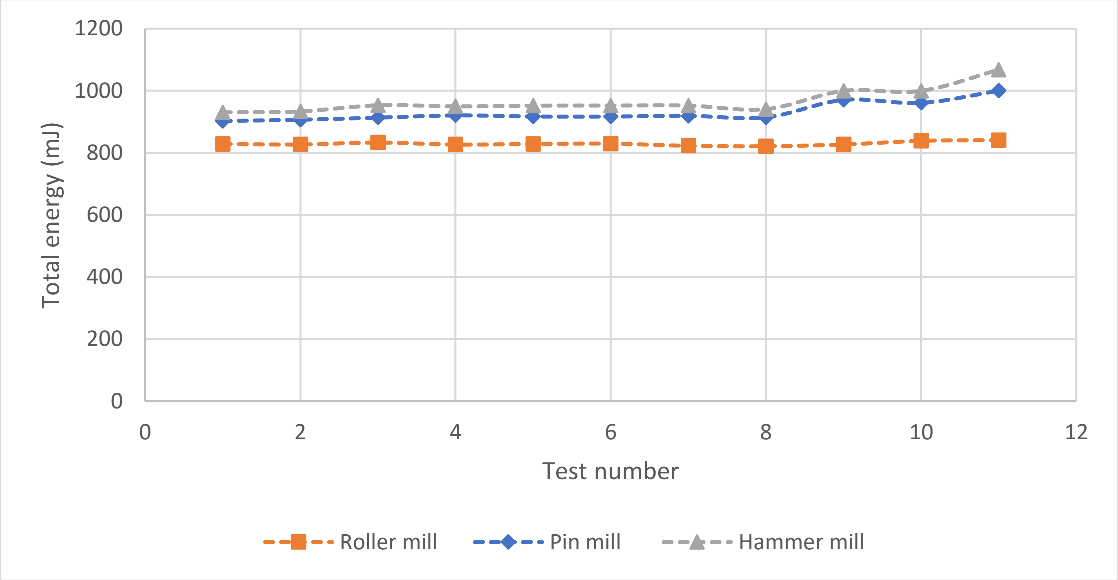
Dynamic flow properties (mean ± SD) of pearl millet flour using different milling methods.

Dynamic flow properties	Milling Method			
	Roller mill	Pin mill	Hammer mill	CV (%)
Basic flow energy (mJ)	822.64 ± 5.39^{b}	919.39 ± 27.13^{a}	952.29 ± 23.63^{a}	2.34
Stability index	0.99 ± 0.02^{a}	1.02 ± 0.02^{a}	1.02 ± 0.01^{a}	1.78
Flow rate index	1.02 ± 0.004^{b}	1.09 ± 0.04^{a}	1.13 ± 0.01^{a}	2.03
Specific energy (mJ/g)	7.30 ± 1.57^{a}	7.46 ± 1.05^{a}	8.41 ± 1.58^{a}	18.42
Aerated energy (mJ) at 10 mm/s	633.51 ± 55.79^{a}	573.33 ± 38.48^{ab}	544.34 ± 27.49^{b}	7.23
Aeration ratio (BFE/energy at 10 mm/s)	2.03 ± 0.06^{b}	2.25 ± 0.09^{a}	2.34 ± 0.07^{a}	3.48
Normalized aeration sensitivity (s/mm)	0.063 ± 0.004^{b}	0.068 ± 0.002^{ab}	0.070 ± 0.002^{a}	4.50

Note. Mean values with different small case letters in the same row differ significantly among different milling methods ($p<0.05$). SD: Standard Deviation, CV: Coefficient of Variation.

Figure 4

Energy consumption during dynamic flow of roller-milled, pin-milled, and hammer-milled flours.



On the other hand, no significant differences were found in the BFE values between pin mill and hammer mill (Table 5). However, roller mill was statistically different, with the lowest value observed (822.64 mJ). In a study conducted on teff flour by Barretto et al. (2022), the lowest BFE was found for roller mill, which was 835.43 mJ at a moisture content of 9.93%. In another study on two varieties of wheat by Bian et al. (2015), value of 680.70 mJ was found for hard wheat at a moisture content of 14.5%, and 713 mJ for soft wheat flour at a moisture content of 11.4%, which is much lower than the values found for pearl millet in this study.

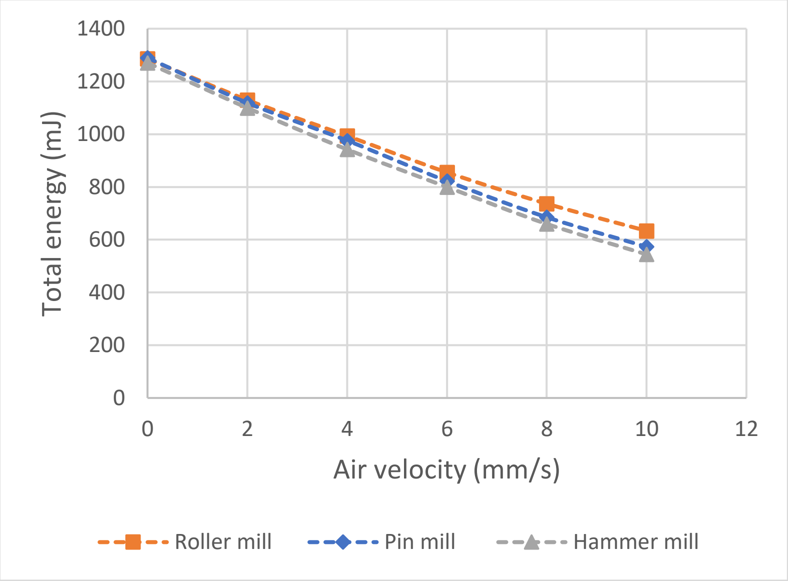
FRI of the roller milled was statistically different from that of pin milled and hammer milled flours, this are show in Table 5. The sensitivity of the flour to different blade tip speeds was measured from test 8 to test 11. This can be observed in Figure 4 and Annex D, where it is also evident that as the blade speed decreases, more energy is required to displace the conditioned powder sample. Cohesive flours require more flow energy at lower flow rates because the air can escape. On the other hand, non-cohesive powders are less sensitive to changes in blade flow rate (Freeman, 2007). Leturia et al. (2014) mention that for an ideal non-cohesive powder, the flow energy would be virtually independent of the flow rate (FRI≈1). Based on the results obtained, roller milled showed the statistically lowest FRI value, indicating that it produces the least cohesive and therefore more fluid flour compared to pin and hammer milled.

Unlike BFE, which is dependent from bulk compressibility, powder consolidation level, and density, SE is mostly influenced by intrinsic particle properties such as cohesion, particle size and shape distributions, and surface roughness. For flours, SE values below 5 mJ/g indicate low cohesiveness (Gnagne et al., 2017). Mitra et al. (2017) mention that SE values between 5 and 10 mJ/g indicate moderately cohesive flour. Additionally, a higher BFE value represents lower flowability, while a higher SE value represents higher cohesiveness. Under these parameters, although no significant differences in SE were found among the treatments, all of them fall within the range of moderately cohesive flours, with roller milled flour showing the lowest SE value (7.30 mJ/g).

The Figure 5 and Annex E shows that as aeration through the powder bed increases, the flowability of pearl millet flour improves, resulting in lower energy consumption as the air velocity increases from 0 to 10 mm/s. The air helps reduce interparticulate cohesion forces and induces flow; however, the extent of energy reduction required for flow also depends on physical properties such as particle shape, texture, and density (Bian et al., 2015). As observed in Figure 5 and Annex E, the flour obtained from all three milling methods exhibited similar behavior as the airflow increased, with hammer milled flour showing the lowest energy consumption at 10 mm/s (544.34mJ). Similar results were reported by Barretto et al. (2022) on teff flour.

Figure 5

Change in flow energy with respect to increasing air velocity for roller-milled, pin-milled, and hammer-milled flours.



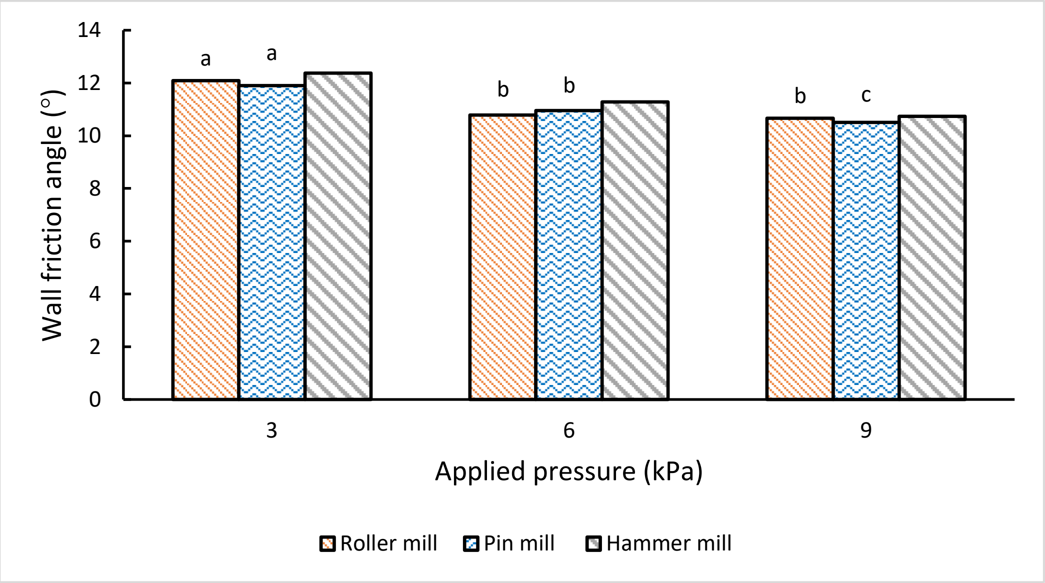
As shown in Table 5, the aerated ratio for hammer milled and pin milled flours had statistically higher values compared to roller milled flour. However, all the flours exhibited moderate sensitivity to aeration. As mentioned by Irie et al. (2021), values of aeration ratio between 2 and 6 are considered to have moderate sensitivity to aeration, while values above 10 indicate high sensitivity to aeration. Moreover, higher aeration ratios and lower aerated energy correspond to lower powder cohesion. Following this rule, hammer milled, and pin milled flours were the most fluidizable. Additionally, Gnagne et al. (2017) mentioned that the irregular shape of particles disrupts air flow, leading to a higher pressure differential across the particles. The higher the normalized aeration sensitivity (NAS), the more the powder is prone to aeration. According with this, in Table 5 its observed that pin milled and hammer milled flours had statistically higher NAS values. Furthermore, Barretto et al. (2022) mention that, in general, less cohesive powders have lower aerated energy (AE) and higher aerated ratio (AR) and NAS. Therefore, pin milled and hammer milled flours exhibit less cohesion than roller milled flour.

Friction Properties

Wall friction is an important property for understanding the flow behavior of powders, especially in hoppers or transfer conduits (Barretto et al., 2022). This parameter measures the resistance of a powder against the material of the container wall, and it is highly influenced by the type of material in contact with the powder (Divya & Ganesh, 2019). Figure 6 shows the wall friction angle of pearl millet flour using different milling methods and tested against stainless steel walls. The ratio of the normal and shear stress forms a wall yield locus, which when plotted, creates the wall friction angle (Freeman Technology, 2014). In Figure 6, it can be observed that there was no significant difference in the wall friction angle among the three evaluated milling methods. This means that all three treatments showed the same resistance between the flour and stainless-steel surface at different applied pressures (3, 6, and 9 kPa). However, it can be observed that roller mill and pin mill had a significantly higher wall friction angle at 3 kPa, while the wall friction angle for hammer mill did not show significant differences with increasing pressure.

Figure 6

Wall friction angle of pearl millet flour using different milling methods tested against stainless steel wall.



Note. Bar with different capital letters differ significantly among different milling methods (p<0.05) at the same applied pressure; and bar with different small case letters differ significantly among different applied pressure in the same treatment (p<0.05).

Correlation Between Physical Properties and Flow Properties

Table 6 shows the coefficient of Pearson correlation between selected physical properties and flow properties of pearl millet flour. Results revealed that D_{10} moderately affected bulk density, BFE, SI, FRI, AR, and NAS. Meanwhile, D_{90} moderately affected bulk density, tapped density, BFE, and FRI. On the other hand, convexity moderately influenced only true density. D_{50}, circularity, elongation and aspect radio didn't affect any flow properties. In terms of proximate composition, moisture content moderately affected tapped density, true density, BFE, and FRI. Water activity moderately influenced bulk density, true density, AR and NAS, and strongly influenced tapped density, BFE, and FRI. Significant influenced was detected between ash content and SE. Finally, damage starch didn't affect any flow properties.

Table 6

Coefficient of Pearson correlation ($p<0.05$) between selected proximate and physical properties and flow properties of pearl millet flours.

Property	Bulk density	Tapped density	True density	BFE	SI	FRI	SE	AE	AR	NAS
D_{10}	-0.67*	-0.64	0.49	-0.8*	-0.71*	-0.73*	-0.01	0.66	-0.74*	-0.71*
D_{50}	-0.42	-0.39	0.34	-0.58	-0.52	-0.45	0.08	0.38	-0.48	-0.43
D_{90}	-0.73*	-0.71*	0.21	-0.68*	-0.37	-0.67*	-0.52	0.38	-0.43	-0.46
Ci	0.35	0.41	-0.55	0.61	0.6	0.6	-0.24	-0.51	0.55	0.52
Co	0.25	0.35	-0.67*	0.54	0.63	0.5	-0.32	-0.49	0.42	0.46
E	-0.29	-0.18	-0.25	-0.26	0.04	-0.16	-0.25	-0.11	-0.16	-0.04
AR_2	0.29	0.18	0.25	0.26	-0.04	0.16	0.25	0.11	0.16	0.04
MC	-0.57	-0.79*	0.74*	-0.70*	-0.38	-0.79*	-0.03	0.39	-0.45	-0.59
WA	-0.79*	-0.92*	0.71*	-0.89*	-0.53	-0.94*	-0.14	0.58	-0.67*	-0.74*
Ash	0.53	0.45	-0.05	0.49	0.32	0.4	0.84*	-0.2	0.45	0.19
DS	0.24	0.22	0.23	-0.07	-0.54	0.07	0.06	0.14	-0.12	0.02

Nota. *Pearson's values (positive or negative) are found to be significant ($p<0.05$). BFE: Basic Flow Energy, SI: Stability Index, FRI: Flow Rate Index, SE: Specific Energy, AE: Aerated Energy, AR: Aeration Ratio, NAS: Normalized Aeration Sensitivity, D_{10}: particle size below which 10% of the sample lies, D_{50}: particle size at which 50%of the sample is larger and 50% is smaller, D_{90}: particle size below which 90% of the sample lies, Ci: Circularity, Co: Convexity, E: Elongation, AR_2: Aspect Ratio, MC: Moisture Content, WA: Water Activity, Ash: Ash Content, DS: Damage Starch.

Conclusions

Hammer mill presented the lowest moisture content and water activity, and no significant differences were found for ash content and damage starch. On the other hand, roller mill presented larger particle sizes, while the shape characteristics were similar among the three milling methods evaluated.

The flour from the pin mill and hammer mill had higher aerated bulk density, tapped density and compressibility percentage; roller mill had the best permeability, and no differences were found for true density among the milling methods. According to the compressibility index and Hausner ratio, all flours fall into the category of fair flow.

Roller mill had lower energy consumption at different blade velocities, with the lowest values observed for basic flow energy and flow rate index. Stability index and specific energy did not present significant differences among the milling methods evaluated. Pin mill and hammer mill flours are more susceptible to different air velocities, presenting the best aeration characteristics, such as lower aerated energy, and higher aeration ratio and normalized aeration sensitivity.

The three milling methods showed the same wall friction angle behavior at different applied pressures. Roller mill and pin mill had a significantly higher wall friction angle at 3 kPa, while the wall friction angle for hammer mill did not show significant differences with increasing pressure.

Recommendations

Determine the shelf life of flours, as well as the ideal flour storage conditions.

Measure flow properties periodically to understand how the flow behavior of flours changes with time and storage conditions.

Evaluate the effect of different milling methods in the production of bakery products.

Conduct sensory evaluation tests on food products made with whole pearl millet flours obtained from different milling methods.

References

Acar, O., Izydorczyk, M. S., Kletke, J., Yazici, M. A., Ozdemir, B., Cakmak, I., & Koksel, H. (2020). Effects of roller and hammer milling on the yield and physicochemical properties of fibre-rich fractions from biofortified and non-biofortified hull-less barley. *Journal of Cereal Science*, *92*(1–2), 102907. https://doi.org/10.1016/j.jcs.2020.102907

American Association of Cereal Chemists (AACC) (1999). *Approved Methods of Analysis, 11th Edition:* Method 44-15.02 Moisture-Air oven methods. St. Paul, MN. Cereals & grains association. https://www.cerealsgrains.org/resources/Methods/Pages/44Moisture.aspx

American Association of Cereal Chemists (AACC) (2007). *Approved Methods of Analysis, 11th Edition:* Method AACC 76.33.01 Damaged Starch. St. Paul, MN. Cereals & grains association. https://www.cerealsgrains.org/resources/Methods/Pages/76Starch.aspx

American Association of Cereal Chemists (AACC) (2020). *Approved Methods of Analysis, 11th Edition:* Method 08-01.01 Ash-Basic Method. St. Paul, MN. Cereals & grains association. https://www.cerealsgrains.org/resources/Methods/Pages/08TotalAsh.aspx

American Society for Agricultural Engineers (ASAE) (1994). *S352.3-moisture measurement-ungrounded grains and seeds*. St. Joseph, MI. ASAE. http://asae.org/standards/

Aulton, M. E., & Taylor, K. M. (2018). The desing and manufacture of medicines. *Elsevier*, *1*, 128–189. https://www.scribd.com/document/486111135/Michael-E-Aulton-Kevin-M-G-Taylor-Aulton-s-Pharmaceutics-The-Design-and-Manufacture-of-Medicines-Elsevier-2017-pdf

Babu, K. S., Siliveru, K., Amamcharla, J. K., Vadlani, P. V., & Ambrose, R. P. K. (2018). Influence of protein content and storage temperature on the particle morphology and flowability characteristics of milk protein concentrate powders. *Journal of Dairy Science*, *101*(8), 7013–7026. https://doi.org/10.3168/jds.2018-14405

Balasubramanian, S., Sharma, R., Kaur, J., & Bhardwaj, N. (2014). Characterization of modified pearl millet (Pennisetum typhoides) starch. *Journal of Food Science and Technology*, *51*(2), 294–300. https://doi.org/10.1007/s13197-011-0490-1

Barretto, R., Buenavista, R. M., Pandiselvam, R., & Siliveru, K. (2022). Influence of milling methods on the flow properties of ivory teff flour. *Journal of Texture Studies*, *53*(6), 820–833. https://doi.org/10.1111/jtxs.12630

Bian, Q. (2014). *Bulk flow properties of wheat* [Thesis]. Kansas State University, Kansas, Estados Unidos. https://krex.k-state.edu/bitstream/handle/2097/18679/QiBian2014.pdf?sequence=6&isAllowed=y

Bian, Q., Sittipod, S., Garg, A., & Ambrose, R. K. (2015). Bulk flow properties of hard and soft wheat flours. *Journal of Cereal Science*, *63*(3), 88–94. https://doi.org/10.1016/j.jcs.2015.03.010

Chen, X., Wang, L. G., & Ooi, J. Y. (2020). A DEM-PBM multiscale coupling approach for the prediction of an impact pin mill. *Powder Technology*, *366*(9), 408–419. https://doi.org/10.1016/j.powtec.2020.02.065

Choudhury, G. S., & Gautam, A. (1999). Screw Configuration Effects on Macroscopic Characteristics of Extradates Produced by Twin-screw Extrusion of Rice Flour. *Journal of Food Science*, *64*(3), 479–487. https://doi.org/10.1111/j.1365-2621.1999.tb15067.x

Codex Alimentarius (2019). *Standard for pearl millet flour* (CXS 170-1989). United States of America. Food and Agriculture Organization of the United States and World Health Organization. https://www.fao.org/fao-who-codexalimentarius/sh-proxy/en/?lnk=1&url=https%253A%252F%252Fworkspace.fao.org%252Fsites%252Fcodex%252FStandards%252FCXS%2B170-1989%252FCXS_170e.pdf

Decagon Devices. (2016). *AquaLab Vapor Sorption Analyzer (VSA) Manual. Decagon Devices, Washington, USA.* https://www.labcell.com/media/140409/vsa%20manual%20web.pdf

Divya, S., & Ganesh, G. N.K. (2019). Characterization of Powder Flowability Using FT4 – Powder Rheometer. *Journal of Pharmaceutical Sciences and Research*, *11*(1), 25–29. https://www.jpsr.pharmainfo.in/Documents/Volumes/vol11Issue01/jpsr11011906.pdf

Doddabematti Prakash, S., Nkurikiye, E., Rajpurohit, B., Li, Y., & Siliveru, K. (2023). Significance of different milling methods on white proso millet flour physicochemical, rheological, and baking properties. *Journal of Texture Studies*, *54*(1), 92–104. https://doi.org/10.1111/jtxs.12717

Food and Agriculture Organization of the United Nations, & World Health Organization (2019). Codex Alimentarius Commission – Procedural Manual twenty-seventh edition. *Joint FAO/WHO Food Standards Programme*, 254. https://www.fao.org/3/ca2329en/ca2329en.pdf

Food and Drug Admministration (FDA) (1998). *21 CFR Part 137.105-Flour*. United States. Code of Federal Regulations. https://www.ecfr.gov/current/title-21/chapter-I/subchapter-B/part-137/subpart-B/section-137.105

Freeman, R. (2007). Measuring the flow properties of consolidated, conditioned and aerated powders — A comparative study using a powder rheometer and a rotational shear cell. *Powder Technology*, *174*(1-2), 25–33. https://doi.org/10.1016/j.powtec.2006.10.016

Freeman Technology. (2014). *FT4 powder rheometer- shear testing- shear cell*. https://www.freemantech.co.uk/powder-testing/ft4-powder-rheometer-powder-flow-tester/shear-testing

Freeman Technology. (2016). *Measuring and understanding the flow properties of powders with the FT4 Powder Rheometer*. https://www.freemantech.co.uk/uploads/downloads/ft4-powder-rheometer-brochure_en.pdf

Fu, X., Huck, D., Makein, L., Armstrong, B., Willen, U., & Freeman, T. (2012). Effect of particle shape and size on flow properties of lactose powders. *Particuology*, *10*(2), 203–208. https://doi.org/10.1016/j.partic.2011.11.003

Gnagne, E. H., Petit, J., Gaiani, C., Scher, J., & Amani, G. N. (2017). Characterisation of flow properties of foutou and foufou flours, staple foods in West Africa, using the FT4 powder rheometer. *Journal of Food Measurement and Characterization*, *11*(3), 1128–1136. https://doi.org/10.1007/s11694-017-9489-2

Gulia, S. K., Wilson, J. P., Carter, J., & Singh, B. P. (2007). Progress in Grain Pearl Millet Research and Market Development. *Issues in New Crops and New Uses*, 197–203. https://www.researchgate.net/publication/237323258_Progress_in_Grain_Pearl_Millet_Research_and_Market_Development

Hlosta, J., Zurovec, D., Jezerska, L., Zegzulkaj, I., & Necas, J. (Eds.) (2016). *Effect of particle shape and size on the compressibility and bulk properties of powders in powder metallurgy*. : Vol. *159*. https://www.confer.cz/metal/2016/read/1976-effect-of-particle-shape-and-size-on-the-compressibility-and-bulk-properties-of-powders-in-powder-metallurgy.pdf

Irie, K. R., Petit, J., Gnagne, E. H., Kouadio, O. K., Gaiani, C., Scher, J., & Amani, G. N.'G. (2021). Effect of particle size on flow behaviour and physical properties of semi-ripe plantain (AA B Musa spp) powders. *International Journal of Food Science & Technology*, *56*(1), 205–214. https://doi.org/10.1111/ijfs.14620

Jan, S., Karde, V., Ghoroi, C., & Saxena, D. C. (2018). Effect of particle and surface properties on flowability of rice flours. *Food Bioscience*, *23*(2), 38–44. https://doi.org/10.1016/j.fbio.2018.03.001

Leturia, M., Benali, M., Lagarde, S., Ronga, I., & Saleh, K. (2014). Characterization of flow properties of cohesive powders: A comparative study of traditional and new testing methods. *Powder Technology*, *253*, 406–423. https://doi.org/10.1016/j.powtec.2013.11.045

Malvern Instruments, Malvern, UK. (2023). *Morphologi 4 Range, Automated imaging for advanced particle characterization*. https://www.malvernpanalytical.com/en/assets/MRK2324-02_MORPHOLOGI4_RANGE_BR_SPCTRS_A4_Infopedia_tcm50-54693.pdf

MEPBA, H. D., EBOH, L., EKO, C. B., & UKPABI, U. J. (2009). Composition and pasting properties of starch from two cocoyam cultivars. *Journal of Food Quality*, *32*(4), 522–537. https://doi.org/10.1111/j.1745-4557.2009.00257.x

Merkus, H. G. (2009). Particle size measurements, Fundametals, practice and quality. *Springer Acience+Business Media*. https://books.google.es/books?hl=es&lr=&id=lLx4GzA-7AUC&oi=fnd&pg=PA2&dq=ISO+9276-6+for+analysis+OF+FLOURS&ots=VcH-6P603e&sig=mGS7EAbUnv8dm1E2HummvFAFYLY#v=onepage&q=ASPECT&f=false

Mitra, H., Pushpadass, H. A., Franklin, M. E. E., Ambrose, R. K., Ghoroi, C., & Battula, S. N. (2017). Influence of moisture content on the flow properties of basundi mix. *Powder Technology*, *312*, 133–143. https://doi.org/10.1016/j.powtec.2017.02.039

Mora, C. F., & Kwan, A.K.H. (2000). Sphericity, shape factor, and convexity measurement of coarse aggregate for concrete using digital image processing. *Cement and Concrete Research*, *30*(3), 351–358. https://doi.org/10.1016/S0008-8846(99)00259-8

Olson, E. (2011). *Particle Shape Factors and Their Use in Image Analysis – Part 1: Theory*. Malvern instruments. https://particletechlabs.com/ptl-press/particle-shape-factors-and-their-use-in-image-analysis-part-1-theory/

Prasanthi, K., & Sireesha, G. (2022). Individuals' Knowledge, Attitude and Practices onMillets. *Int J Food Nutr Sci*, *11*, 21–27. https://www.researchgate.net/publication/360719758_Individuals'_Knowledge_Attitude_and_Practices_on_Millets

Pulivarthi, M. K., Selladurai, M., Nkurikiye, E., Li, Y., & Siliveru, K. (2022). Significance of milling methods on brown teff flour, dough, and bread properties. *Journal of Texture Studies*, *53*(4), 478–489. https://doi.org/10.1111/jtxs.12669

Rani, S., Singh, R., Sehrawat, R., Kaur, B. P., & Upadhyay, A. (2018). Pearl millet processing: a review. *Nutrition & Food Science*, *48*(1), 30–44. https://doi.org/10.1108/NFS-04-2017-0070

Siliveru, K., Ambrose, R. K., & Vadlani, P. V. (2017). Significance of composition and particle size on the shear flow properties of wheat flour. *Journal of the Science of Food and Agriculture*, *97*(8), 2300–2306. https://doi.org/10.1002/jsfa.8038

Siliveru, K., Kwek, J. W., Lau, G. M. L., & Ambrose, R. P. K. (2016). Image Analysis Approach to Understand the Differences in Flour Particle Surface and Shape Characteristics. *Cereal Chemistry Journal*, *93*(3), 234–241. https://doi.org/10.1094/CCHEM-05-15-0108-R

Singh, I., & Kumar, P. (2012). Preformulation studies for direct compression suitability of cefuroxime axetil and paracetamol: a graphical representation using SeDeM Diagram. *Acta Poloniae Pharmaceutica - Drug Research*, *69*(1), 87–93. https://www.ptfarm.pl/pub/File/Acta_Poloniae/2012/1/087.pdf

Suma, P. F., & Urooj, A. (2011). Nutrients, antinutrients & bioaccessible mineral content (invitro) of pearl millet as influenced by milling. *Journal of Food Science and Technology*, *51*(4), 756–761. https://doi.org/10.1007/s13197-011-0541-7

Suma P, F., & Urooj, A. (2015). Isolation and Characterization of Starch from Pearl Millet (Pennisetum typhoidium) Flours. *International Journal of Food Properties*, *18*(12), 2675–2687. https://doi.org/10.1080/10942912.2014.981640

Tortoe, C., Akonor, P., Hagan, L., Kanton, R., Asungre, P., & Ansoba, E. (2019). Assessing the suitability of flours from five pearl millet (Pennisetum americanum) varieties for bread production. *International Food Research Journal*, *26*(1), 329–336. http://www.ifrj.upm.edu.my/26%20(01)%202019/(37).pdf

United States Departmment of Agriculture. (2016). *Inspecting Grain Practical Procedures for Grain Handlers*. USDA. https://www.ams.usda.gov/sites/default/files/media/PracticalProceduresBook2017.pdf

Vanisha S, N., Dhaduk, J. J., Sareen, N., Shadu, T., & Desai, R. (2011). Potential Functional Implications of Pearl millet (Pennisetum glaucum) in Health and Disease. *Journal of Applied Pharmaceutical Science*, *01*(10), 62–67. https://japsonline.com/admin/php/uploads/299_pdf.pdf

Vidya, S., Ravi, R., & Bhattacharya, S. (2012). Effect of Thermal Treatment on Selected Cereals and Millets Flour Doughs and Their Baking Quality. *Food Bioprocess Technology*, *6*(5), 1218–1227.

Vukmirovic, D., Levic, J., Fistes, A., Colovic, R., Brlek, T., Colovic, D., & Djuragic, O. (2016). Influence of grinding method and grinding intensity of corn on mill energy consumption and pellet quality. *Hemijska Industrija*, *70*(1), 67–72. https://doi.org/10.2298/HEMIND141114012V

Zhang, Z., & Ghadiri, M. (2002). Impact attrition of particulate solids. Part 2: Experimental work. *Chemical Engineering Science*, *57*(17), 3671–3686. https://doi.org/10.1016/S0009-2509(02)00241-5

Annexes

Annex A

Compressibility percentage (mean ± SD) evaluating the three treatments at different pressures.

Applied normal stress (KPa)	Milling Method		
	Roller mill	Pin mill	Hammer mill
0.5	2.82 ± 0.29^{Ag}	2.90 ± 0.77^{Ah}	3.25 ± 0.61^{Ah}
1	5.77 ± 0.53^{Af}	6.42 ± 1.36^{Ag}	7.10 ± 0.10^{Ag}
2	9.56 ± 0.65^{Be}	11.22 ± 1.64^{ABf}	12.26 ± 1.15^{Af}
4	13.33 ± 0.69^{Bd}	15.96 ± 1.68^{Ae}	17.20 ± 1.14^{Ae}
6	15.52 ± 0.46^{Bc}	18.52 ± 1.62^{Ad}	19.77 ± 1.10^{Ad}
8	16.77 ± 0.68^{Bbc}	20.23 ± 1.78^{Acd}	21.58 ± 1.10^{Acd}
10	17.65 ± 0.61^{Bb}	21.37 ± 1.55^{Abc}	22.74 ± 1.04^{Abc}
12	18.43 ± 0.55^{Bab}	22.30 ± 1.73^{Aab}	23.65 ± 1.03^{Aab}
15	19.71 ± 0.39^{Ba}	23.60 ± 1.69^{Aa}	25.05 ± 1.01^{Aa}
CV (%)		7.35	

Note: Mean values with different capital letters in the same row differ significantly among different milling methods at the same applied normal stress and mean values with different small case letters in the same column differ significantly between the different pressures evaluated in each treatment ($p<0.05$). SD: Standard Deviation, CV: Coefficient of Variation.

Annex B

Pressure drops (mBar) evaluating the three treatments at different pressures (mean ± SD).

Applied normal stress (KPa)	Milling Method		
	Roller mill	Pin mill	Hammer mill
1	0.89 ± 0.44^{Aa}	1.18 ± 0.42^{Ad}	1.35 ± 0.29^{Ae}
2	0.99 ± 0.43^{Ba}	1.38 ± 0.40^{Acd}	1.64 ± 0.27^{Ade}
4	1.13 ± 0.44^{Ba}	1.66 ± 0.37^{ABbcd}	2.06 ± 0.25^{Acd}
6	1.22 ± 0.45^{Ba}	1.85 ± 0.36^{Aabc}	2.35 ± 0.23^{Abc}
8	1.28 ± 0.44^{Ba}	2.01 ± 0.32^{Aab}	2.57 ± 0.22^{Aabc}
10	1.32 ± 0.45^{Ca}	2.12 ± 0.32^{Bab}	2.74 ± 0.22^{Aab}
12	1.36 ± 0.45^{Ca}	2.22 ± 0.31^{Bab}	2.87 ± 0.21^{Aab}
15	1.41 ± 0.45^{Ca}	2.35 ± 0.28^{Ba}	3.09 ± 0.21^{Aa}
CV (%)		19.79	

Note: Mean values with different capital letters in the same row differ significantly among different milling methods at the same applied normal stress and mean values with different small case letters in the same column differ significantly between the different pressures evaluated in each treatment ($p<0.05$). SD: Standard Deviation, CV: Coefficient of Variation.

Annex C

Energy consumption (mJ) during stability test evaluating the three treatments at blade sped of 100 mm/s (mean ± SD).

Test 1 to 7 @ 100 mm/s	Milling Method		
	Roller mill	Pin mill	Hammer mill
1	828.50 ± 15.49^{Ba}	902.76 ± 39.22^{Aa}	930.47 ± 21.34^{Aa}
2	826.98 ± 12.50^{Ba}	906.65 ± 31.68^{Aa}	933.59 ± 20.30^{Aa}
3	833.14 ± 14.27^{Ba}	913.41 ± 29.87^{Aa}	953.22 ± 26.23^{Aa}
4	826.70 ± 7.84^{Ba}	920.56 ± 37.23^{Aa}	949.68 ± 26.86^{Aa}
5	828.56 ± 6.98^{Ba}	917.06 ± 32.32^{Aa}	952.00 ± 23.54^{Aa}
6	829.67 ± 7.40^{Ba}	916.94 ± 33.89^{Aa}	952.34 ± 26.08^{Aa}
7	822.63 ± 5.39^{Ba}	919.39 ± 27.14^{Aa}	952.29 ± 23.63^{Aa}
CV (%)		2.74	

Note: Mean values with different capital letters in the same row differ significantly among different milling methods at the same test and mean values with different small case letters in the same column differ significantly between the different test evaluated in each treatment ($p<0.05$). SD: Standard Deviation, CV: Coefficient of Variation.

Annex D

Energy consumption (mJ) during stability test evaluating the three treatments at different blade velocities (mean ± SD).

Blade speed	Milling Method		
	Roller mill	Pin mill	Hammer mill
100 mm/s	821.22 ± 2.30^{Ba}	914.21 ± 28.41^{Ab}	940.75 ± 21.40^{Ac}
70 mm/s	826.66 ± 4.25^{Ba}	960.76 ± 41.68^{Aab}	999.52 ± 35.78^{Ab}
40 mm/s	838.69 ± 4.14^{Ba}	970.51 ± 48.04^{Aa}	999.75 ± 32.59^{Ab}
10 mm/s	840.66 ± 5.07^{Ca}	999.91 ± 62.88^{Ba}	1067.52 ± 28.96^{Aa}
CV (%)		3.47	

Note: Mean values with different capital letters in the same row differ significantly among different milling methods at the same blade speed and mean values with different small case letters in the same column differ significantly between the different blade velocities evaluated in each treatment ($p<0.05$). SD: Standard Deviation, CV: Coefficient of Variation.

Annex E

Energy consumption (mJ) during aeration test evaluating the three treatments at different air velocities.

Air Velocity (mm/s)	Milling Method		
	Roller mill	Pin mill	Hammer mill
0	1286.05 ± 84.05^{Aa}	1289.42 ± 47.05^{Aa}	1270.56 ± 39.60^{Aa}
2	1129.69 ± 86.80^{Ab}	1118.77 ± 42.66^{Ab}	1099.76 ± 24.76^{Ab}
4	993.58 ± 90.28^{Ac}	975.85 ± 46.80^{Ac}	942.32 ± 34.60^{Ac}
6	855.24 ± 73.53^{Ad}	822.19 ± 33.56^{Ad}	799.72 ± 27.93^{Ad}
8	736.53 ± 62.99^{Ae}	685.06 ± 31.94^{Ae}	659.77 ± 28.96^{Ae}
10	633.51 ± 55.79^{Af}	573.33 ± 38.48^{ABf}	544.33 ± 27.49^{Bf}
CV (%)		5.83	

Note: Mean values with different capital letters in the same row differ significantly among different milling methods at the same air velocity and mean values with different small case letters in the same column differ significantly between the different air velocities evaluated in each treatment ($p<0.05$). SD: Standard Deviation, CV: Coefficient of Variation.