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# Physical and mechanical properties of lemon (Citrus lemon) seeds

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## ABSTRACT

The objective of this study was to determine some geometric, gravimetric, and mechanical properties of lemon seeds and how these were influenced by moisture content under a range of post-storage processing conditions. The skin, pulp, and seed fractions of lemon fruits were determined. The seed fraction had a value of 1.86% with an average hull/seed ratio of 38.92%. Seed moisture content was varied by drying with hot air from the storage condition (8.28% d.b.) to a minimum moisture condition (1.31% d.b.). The drying kinetics were adequately described using a two-term exponential model, which suggested the presence of two distinct internal seed resistances to moisture transfer related to its composition. Furthermore, with the exception of thickness, all geometric properties evaluated tended to decrease as moisture content decreased, especially at the driest level evaluated. Within these, the most important variations were found in theoretical volume (24.20%) and width (22.28%), for a moisture content change of 84%. Gravimetric and mechanical properties exhibited the same tendency, with the exception of true density, which increased, and bulk density and bulk porosity which did not change significantly. It is noteworthy that rupture energy decreased by approximately 50% as the moisture content was reduced from 8.28 to 4.62%. In conclusion, drying lemon seeds generated smaller and more fragile structures that could benefit subsequent processes such as dehulling or milling, among others, but could also negatively affect seed movement through undesirable breaking. Therefore, knowledge of the properties studied in this work will be useful for the design of manufacturing and storage equipment, as well as handling and process operations.

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## 1. Introduction

Citrus fruits have a significant commercial value in the fresh market and as a raw material for food-processing, 75% of which is traded for fresh consumption (Panwar et al., 2021). Citrus processes generate large amounts of by-products such as peel, pulp, and seeds, which are estimated to account about 50% of the total fruit processed (Putnik et al., 2017). Within this group, >21 million tons of lemons were produced in 2020 (FAOSTAT, 2021). Argentina is the third largest producer of lemon fruits in the world and the country with the highest quantity of fruit destined for

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industrialization, with an annual production of 1.03 million tons of lemon fruits, 71% of which are destined for juice production (USDA, 2021). This fact positions Argentina as the main producer of lemon by-products. Seeds make up 1% of the solid waste generated at the end of juice processing, which is also constituted by a yellow peel with essential oils in the pores, a thick white layer called albedo, and juice sacs. There is a growing interest in the citrus by-products valorization such as seeds, and research has focused on developing techniques to obtain compounds such as oil and protein from these matrices (Yilmaz and Güneşer, 2017; Mahato et al., 2020). Since solid wastes from the juice industry, such as lemon seeds (LS), are produced in large quantities, understanding their physical and mechanical properties is important to assess their productive feasibility, as well as the technoeconomics of seed handling and storage. These properties are directly related to the equipment design used in harvesting, handling, dehulling, separation, drying, aerating, storing, and postprocessing of the seeds (Baümler et al., 2006; Murakonda et al., 2022). In the literature, it has been reported the importance of moisture content in the physical properties of various seeds, and

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Nomenclature			
$A_i$	Dimensionless constant	TSW	Thousand seeds weight, g
$D_b$	Deformation at break, %	$V_{g}$	Theoretical volume, mm <sup>3</sup>
$D_g$	Geometrical diameter, mm	$V_t$	True volume, mm <sup>3</sup>
$E_b$	Rupture energy, J m <sup>-3</sup>	W	Width, mm
k <sub>i</sub>	Kinetic constant, s <sup>-1</sup>	$W_i$	Weight, g
K <sub>ν</sub>	Volume correction factor, mm <sup>3</sup> mm <sup>-3</sup>	X	Dry basis moisture content at time t, kg kg <sup>-1</sup>
L	Length, mm	Xe	Dry basis moisture content at equilibrium, kg kg <sup>-1</sup>
MR	Moisture ratio, % % <sup>-1</sup>	$X_0$	Dry basis moisture content at time $t = 0$ h, kg kg <sup>-1</sup>
$R_{h}$	Rupture force, N	$\mathcal{E}_{h}$	Bulk porosity, m <sup>3</sup> m <sup>-3</sup>
Sg	Projected area, mm <sup>2</sup>	$\tilde{\rho_h}$	Bulk density, kg m <sup>-3</sup>
Т	Thickness, mm	$\rho_t$	True density, kg m <sup>-3</sup>
t	Time, h	$\varphi$	Sphericity, mm mm <sup>-1</sup>

therefore for designing of their handling and processing equipment (Baümler et al., 2006; Aviara et al., 2013; Mirzabe et al., 2021). In conventional oilseeds processing, it is recommended to condition the moisture content of the seeds below 10% to improve not only their storage and handling but also their processing, including for subsequent oil extraction (Rao et al., 2006). Therefore, generating knowledge about the physical and mechanical properties of seeds in relation to their moisture content is a key factor related to the treatment required for LS valorization.

Over the years, the physical and mechanical properties of main oilseeds such as soybean (Barnwal et al., 2012; Pohndorf et al., 2017), corn (Su et al., 2021), and sunflower (Malik and Saini, 2016), as well as alternative oilseeds such as safflower (Baümler et al., 2006), almond (Atteh et al., 2021), and grape (Mirzabe and Hajiahmad, 2021), and those seeds whose potential as oilseed was considered such as ash gourd (Gade et al., 2020), fennel (Ahmadi et al., 2009), and kenaf (Izli, 2015) have been studied. However, scarce research has been conducted on the physical properties of citrus seeds. Recently, some properties of Persian lime seeds, such as mean diameter, projected area, volume, sphericity, thousand-seed weight, true density, bulk density, and bulk porosity at two moisture content, 55.4% and 9.54% d.b., have been reported showing a dependence of these properties on moisture content (Fathollahi et al., 2020). However, to confirm the tendency of these properties with water content variation, a more detailed study involving other moisture conditions is required. Moreover, knowledge of these properties at seed moisture ranges close to storage conditions would be useful for designing equipment and the establishment of subsequent processing conditions.

To date, very few studies related to the evaluation of the physical and mechanical properties of LS have been found. For instance, seed size, thousand seeds weight, and hull ratio were reported for LS at 58% d.b. (Yilmaz and Güneşer, 2017). Thus, the aim of this work was to study the behavior of the physical and mechanical properties of lemon (*Citrus lemon* var. Eureka) seeds at and below the recommended moisture condition to seed storage.

## 2. Materials and methods

## 2.1. Fruit characterization: skin/pulp/seeds (SPS) and seeds/fruit (SF) ratio

In the present study, *Citrus lemon* var. Eureka was characterized. SPS ratio values were calculated from a sample of twenty randomly selected fruits. Each lemon was weighed and its skin and albedo were manually removed from the pulp. The seeds were manually and carefully separated from the lemon pulp over a tray to collect any juice that might escape from the pulp. All fractions (skin, pulp, and seeds) were weighed to calculate the SPS ratio. On the other hand, SF values were obtained from one hundred randomly selected fruits, following the above procedure and registering the weights of seeds and fruits. Seed units per fruit were also calculated from this assay. SPS and SF ratio values were expressed on a wet basis.

## 2.2. Obtaining seeds

The seeds removed from the lemon pulp according to section 2.1 were sanitized using chlorine solution (1.25 ppm) to reduce the microbial load on their surface, since the seeds were not at a safe moisture condition yet. Subsequently, LS were rewashed in distilled water, placed over aluminum foil and dried overnight at room temperature. Then, LS were dried in a convection oven (mean temperature: 55 °C; air velocity over the seeds: 0.2 m/s) for 4.5 h, cooled and stored in glass containers with screw-top glass jars at 4 °C for 48 h in a dark place to attain seed moisture content equilibrium.

#### 2.3. Hull ratio of LS

The total hull content of the seed was determined by manual dehulling of 10 g of LS. The hull was carefully removed with tweezers and separated from the germ and endosperm, both considered as a single part (kernel). The presence of the bran over the hull or the endosperm was considered irrelevant and, therefore, its contribution to the weight of each fraction. The distribution of bran on the hull and endosperm fraction was not mentioned in the literature consulted, understanding that the presence of the bran was also considered negligible by other authors. Hull and kernel fractions were weighed and dried in a vacuum oven at 60 °C and 20 inHg vacuum to constant weight. The hull ratio of LS was calculated as the quotient between the weight of this fraction and the sum of both. Values were obtained by triplicate and expressed on a dry basis.

## 2.4. Drying kinetics and moisture conditioning of LS

A drying curve was carried out in a convection oven (mean temperature: 55 °C; air velocity over seeds: 0.2 m/s). Approximately 5 g of LS were dried and the weight loss was recorded periodically at 20 min time intervals until a constant weight was reached. Values were obtained by triplicate.

Drying seeds with hot air is usually explained using the theoretical model of Fick's second law (Crapiste and Rotstein, 1997). Sharaf-Eldeen et al. (1979) proposed a general solution to the diffusion equation given by a sum of different contributions, each represented by first-order kinetics (Eq. (1)): Agustín Benestante, M.C. Chalapud, E. Baümler et al.

$$MR = \sum_{i=1}^{n} A_i exp(-k_i t) \tag{1}$$

$$MR = \frac{X - X_e}{X_0 - X_e} \tag{2}$$

where X,  $X_0$ , and  $X_e$  represent the seed moisture content on a dry basis at time t (s), at t = 0 s, and in equilibrium with the air relative humidity at the drying temperature, respectively,  $A_i$  is a dimensionless constant, and  $k_i$  is the kinetic constant for each corresponding contribution (i).

Experimental values of moisture content, standardized as MR values using Eq. (2), over time were adjusted to Eq. (1) and plotted using Sigmaplot 2008 software package (Sigmaplot 2008). The fit parameter values were obtained from triplicate experiments.

## 2.5. Effect of seeds moisture content on some properties

Taking into account the drying kinetics obtained, seeds were conditioned in the convection oven (at air conditions similar to those described above). The moisture range content was covered between the initial condition (M1) and the lowest achievable moisture content (M3), with an intermediate value (M2). The moisture content reached by the conditioned seeds was determined by the vacuum oven method (60 °C, 20 inHg vacuum) (IUPAC, 1992). Values were obtained by triplicate. The conditioned seeds were stored in glass containers with screw-top glass jars at 4 °C for 48 h in a dark place to attain seed moisture content equilibrium before use. Prior to the following measurements, the seeds were taken out of the refrigerator and allowed to warm up to room temperature for at least 2 h (Baümler et al., 2006).

#### 2.5.1. Geometrical properties

Length (L), width (W), and thickness (T) of LS were measured with a digital micrometer (IP65 Asimeto, Germany) with an accuracy of 0.001 mm. Geometrical diameter (D<sub>g</sub>) and sphericity ( $\varphi$ ) of seeds were calculated using Eq. (3) and Eq. (4), respectively (Singh and Meghwal, 2020):

$$D_{\rm g} = (LWT)^{1/3}$$
(3)

$$\varphi = \frac{D_g}{L} \tag{4}$$

The projected area  $(S_g)$  and theoretical volume  $(V_g)$  were calculated considering cono-spherical shaped seeds (Jain and Bal, 1997) by using Eq. (5) and Eq. (6), and were calculated for one hundred seeds at each moisture condition.

$$S_{g} = \frac{\pi \sqrt{WTL^{2}}}{(2L - \sqrt{WT})}$$
(5)

$$V_g = \frac{\pi W T L^2}{6(2L - \sqrt{WT})} \tag{6}$$

## 2.5.2. Gravimetric properties

2.5.2.1. Thousand seed weight (TSW). In order to quantify the weight of one thousand seeds, two hundred and fifty LS were weighed on an analytical balance (accuracy  $\pm$  0.0001 g). Then, the value obtained was extrapolated to the weight of one thousand seeds. Values were obtained by triplicate for each moisture content condition.

*2.5.2.2. True density.* The true volume of LS ( $V_t$ ) was determined by the liquid displacement method at room temperature (20 °C). Since water absorption can lead to a change in LS moisture content, toluene was chosen as the anhydrous solvent because of its low

penetration into the matrix (Darfour et al., 2022). The procedure was standardized because the vapor pressure of toluene was higher than of water, and its volatilization could affect the values obtained. First, 1 g of LS was weighed (W1) and kept apart. A 20 mL pycnometer of solids previously weighed (W<sub>2</sub>) was filled with toluene and covered with its lid and plastic wrap, to reduce solvent evaporation. At that moment, time started to run and the pycnometer was placed on an analytical balance (accuracy ± 0.00 01 g) to record its weight at 60 s ( $W_3$ ). Then, the pycnometer was removed from the balance, the weighed seeds were put into the pycnometer with solvent and the procedure was repeated to record the new weight  $(W_4)$ . V<sub>t</sub> was calculated using Eq. (7) and the true density  $(\rho_t)$  as the ratio between  $W_1$  and  $V_t$ . Values were obtained by triplicate and expressed on a dry basis for each moisture content. The density of toluene ( $\rho_{toluene}$ ) was obtained from the data sheet (Anedra) at room temperature.

$$V_t = \frac{(W_3 - W_2) - (W_4 - W_2 - W_1)}{\rho_{toluene}}$$
(7)

Due to the irregular shape of LS and the surface folds, volume correction factor ( $K_V$ ) was calculated using Eq. (8). The mean value and the upper and lower extreme values of each parameter were used to obtain the correction factor, considering a normal distribution with a confidence interval of 95%.

$$K_V = \frac{V_t}{V_g} \tag{8}$$

2.5.2.3. Bulk density. The bulk density of seeds  $(\rho_b)$  is given as the ratio between the mass of seeds occupying a known volume and this volume. This parameter was determined using a Schopper chondrometer modified to contain a bulk volume of 50 mL, similar to that suggested by Lauro et al. (2020). LS were put into the cylinder and then weighed, repeating the experiment six times for each moisture content.

*2.5.2.4. Bulk porosity.* The porosity of the bulk LS ( $\varepsilon_b$ ) was calculated using Eq. (9) (Pradhan et al., 2013):

$$\varepsilon_b = \frac{\rho_t - \rho_b}{\rho_t} \tag{9}$$

The  $\varepsilon_b$  values were determined from the mean value and the upper and lower extreme values of each parameter involved, considering a normal distribution with a confidence interval of 95%.

#### 2.5.3. Mechanical properties

In order to determine rupture force (R<sub>b</sub>), deformation at break (D<sub>b</sub>), and rupture energy (E<sub>b</sub>), LS were subjected to compression until the hull broke. Previously, seeds were further examined visually, discarding those with visible cracks in the hull, and their dimensions were measured as mentioned in Section 2.5.1. A texture analyzer TA Plus (Lloyds Instruments, UK) equipped with a 500 N load cell was used for this test. Force and deformation values were registered by the Nexygen Plus (2009) software (Lloyd Instruments, UK) connected to the testing machine. The crosshead speed was set at 1 mm/min (Baümler et al., 2006). Each seed was positioned horizontally with its length placed perpendicular to the loading direction; then, the seed was compressed between a flat fixed surface and the flat sonde until rupture occurred. This last specific condition was identified as a break in the registered force-deformation curve. By visualizing the curve, the force magnitude and deformation indicated at this breakpoint corresponded to R<sub>b</sub> and the deformation of the seed at break, respectively. D<sub>b</sub> was calculated using Eq. (10) (Mabille et al., 2001), where  $T_i$  and  $T_f$ are the initial and final thickness of the seed, respectively. E<sub>b</sub> was

calculated as the area under the force-deformation curve until break and expressed in terms of the individual theoretical volume of each seed. Values were obtained for fifty seeds at each moisture condition.

$$D_b = \frac{T_f - T_i}{T_i} \tag{10}$$

## 2.6. Statistical analysis

The results obtained were presented as mean values with standard deviations. Significant differences between them were analyzed using one-way ANOVA and LSD Fisher's test ( $\alpha = 0.05$ ) for multiple comparisons. All statistical analyzes were performed using InfoStat statistical analysis software (Di Rienzo et al., 2018). The values obtained in Section 2.1 were analyzed using the Shapiro-Wilks test modified (Rahman and Govindarajulu, 1997), in order to determine the normal distribution of all data.

## 3. Results and discussion

## 3.1. SPS and SF ratio

The SPS and SF ratios are shown in Table 1.

The pulp of the lemon fruit contains mainly the juice in its structure, and as expected, its ratio value was significantly higher (p < 0.05) than the other fractions (skin and seeds). The pulp represents almost three and forty times the amount of skin and seeds, respectively. Despite the fact that fresh fruits have a similar overall anatomy, some variation is to be expected as macroscopic and microscopic structure of the fruits might change due to irrigation, age, maturity, and position in the tree, among other factors (Berk, 2016). However, a normal distribution of their ratios was projected (p > 0.05) since these values were naturally disposed and prior selection should not imply changes of them.

The weights of fresh fruit and seeds per fruit are shown in Table 2. Since lemon weight data did not follow a normal distribution according to the Shapiro-Wilks test (p < 0.05), a histogram of the experimental data was performed to examine its tendency (Fig. 1). Fig. 1 showed a left symmetry of data and a long right tail. Given that fresh fruit was purchased in its original packaging and the industry has an internal and external criteria for fruit trading,

#### Table 1

Skin, pulp, and seeds to fruit ratio.

Parameter	Skin/Fruit	Pulp/Fruit	Seeds/Fruit
Ratio (%)	24.730 ± 6.333 <sup>b</sup>	73.529 ± 6.939 <sup>c</sup>	1.741 ± 1.006 <sup>a</sup>
p-value	0.1364	0.1260	0.4936

The ratio values presented are the mean  $\pm$  standard deviation of the determination (n = 20). Means in the same row carrying different superscripts are significantly (p < 0.05) different, corresponding "a" with the lowest value. Experimental data with p-value < 0.05 do not follow a normal distribution, according to the modified Shapiro-Wilks test.

#### Table 2

Weight of fruit, weight of LS, and seed units per fruit.

Parameter	Weight of fresh	Weight of seeds per	Seeds per fruit
	fruit (g)	fruit (g)	(unit)
Value*	133.132 ± 22.460	2.461 ± 1.261	16.870 ± 7.566
p-value	0.0050	0.0739	0.2089

<sup>\*</sup> The values presented are the mean  $\pm$  standard deviation of the determination (n = 100). Experimental data with p-value < 0.05 do not follow a normal distribution, according to the modified Shapiro-Wilks test.

the absence of a normal distribution may indicate a prior industry selection.

According to the modified Shapiro-Wilks test, the amount of seeds per fruit and their weight followed a normal distribution (p > 0.05) (Table 2). These variations in the seeds are related to botanical, morphological, and environmental parameters. All data were plotted (Fig. 2) and fitted with a linear regression, forcing it to pass through the origin. A good correlation of the experimental data was obtained (coefficient of determination,  $r^2 > 0.97$ ), showing that more than 6 units per gram of seed could be obtained (slope = 6.594 units/g, standard error = 0.112).

Finally, the SF ratio obtained was  $1.864 \pm 0.960\%$  (n = 100), which did not differ from the value obtained with n = 20 (Table 1). Similar values have been reported for lemons and oranges representing 1 and 2% of the total fruit, respectively (Arriola-Guevara et al., 2006; Berk, 2016).

Fig. 3 shows photographs of the hull (Fig. 3a) and the kernel (Fig. 3b) of LS. The bran stuck to both fractions (hull and kernel) can be seen and identified as a brown colored layer. A value of  $38.92 \pm 1.27\%$  was obtained for the hull ratio of LS. Knowledge of this parameter is relevant due to its relationship to some seed treatments such as drying or oil extraction. The hull and kernel ratio has been extensively studied for different seeds over the years, such as rapeseed (Carré et al., 2016), sunflower (De Figueiredo et al., 2011), and cotton (Pahlavani and Abolhasani, 2006), among others. For lemon and grapefruit fresh seeds, it has been reported a hull:kernel mean value of 0.65 and 0.39, which corresponds to a hull ratio of 39.4% and 28.1%, respectively, for seed moisture contents above 49% d.b. approximately (Yilmaz and Güneşer, 2017; Yilmaz et al., 2019).

### 3.2. Drying kinetics

Fig. 4 shows the changes in LS moisture content (expressed as MR using Eq. (2)) over drying time from  $X_0 = 8.56\%$  (d.b.) to  $X_e = 1.47\%$  (d.b.). As can be seen, the drying process of LS followed a typical behavior for convective drying. The non-linear steep drop observed during the first 2 h of drying indicates that the water transfer rate was not constant; it is to say, drying was not controlled externally from the beginning of the process (Crapiste and Rotstein, 1997). This may be directly related to the fact that most of the non-bound water may have been previously removed during overnight and convective drying, leaving the seeds with a moisture content of less than 10%. A MR reduction of almost three times at 3 h of drying with the highest drying rate in the first hour (3.66 kg water (kg seed (d.b.)<sup>-1</sup>) was observed, which indicates the removal of internal moisture of the seed during the drying process (Crapiste and Rotstein, 1997).

The experimental data was fitted using two diffusive models: one-term and two-term exponential models (Eq.1), obtaining the corresponding parameters shown in Table 3. The parameter k represents the effective drying kinetics constant, while the parameter A defines the contribution of the corresponding term to the overall drying process. The one-term exponential model considered the seed as a homogeneous matrix in which water movement would be characterized by a single k value and A would be expected to approach the value of one. However, as previously shown, LS are constituted for two main well-defined fractions —the hull and the kernel—; then, water must go through both until it reaches the outer surface from the inside. Therefore, it is expected that the individual contribution of each fraction could be better represented by the two-term exponential model.

Based on the statistical parameters (p-values,  $r^2$ , and RMSE) shown in Table 3 and as can be observed in Fig. 4, the behavior of the experimental data was better represented by the two-term exponential model. This seems to indicate that both fractions con-



Fig. 1. Histogram of the experimental data of the fresh fruit weight.



Fig. 2. Seeds per fruit and weight of seeds per fruit: dispersion plot of the experimental data and its linear regression fit.

tribute to water transfer in different ways. Furthermore, it became clear that considering the whole seed as a homogeneous matrix would lead to a rough approximation about the drying process of LS. The A value corresponding to this consideration indicates that more than 10% of the initial moisture loss is incorrectly represented by the one-term exponential model.

Although both drying kinetics constant,  $k_1$  and  $k_2$ , were significant and different from each other, the quotient value between them is 14.07, showing that the second term would represent the main resistance to water transfer. It is possible to think that the first term, denoted by  $k_1$  and  $A_1$ , reflects the movement of

water through a structure that does not offer major resistance. On the other hand, after hull drying, there is a joint resistance of the dried hull-kernel to the movement of water from the inner part of the seed, which would be characterized by the parameters  $k_2$  and  $A_2$  of the second term. Moreover, a  $k/k_2$  ratio of 1.48 suggests that the one-term model is mainly driven by the resistance to water transfer in the internal structure of the seed. The fact that the sum of  $A_1$  and  $A_2$  gives a value of 1 confirms the complementarity of both terms involved in the drying process of LS, which could not be adequately represented with the one-term exponential model (A < 1).



Fig. 3. a) Hull and b) kernel of LS.



Fig. 4. Variation in moisture ratio of LS over drying time.

 Table 3

 Parameters of the exponential models obtained for drying kinetics of LS.

Coefficient	Parameter value*	p-value		
One-term exponential model				
A	0.8696 ± 0.0214	< 0.0001		
k (h <sup>-1</sup> )	0.3691 ± 0.0178	< 0.0001		
r <sup>2</sup>	0.9568			
RMSE	0.1127			
Two-term exponential model				
A <sub>1</sub>	0.3210 ± 0.0130	< 0.0001		
$k_1 (h^{-1})$	3.5187 ± 0.2983	< 0.0001		
A <sub>2</sub>	0.6776 ± 0.0118	< 0.0001		
$k_2 (h^{-1})$	0.2500 ± 0.0075	< 0.0001		
r <sup>2</sup>	0.9983			
RMSE	0.0045			

\*The values presented are the mean of determination  $\pm$  standard deviation (n = 3). RMSE: root-mean square deviation.

Some scientific articles have reported similar fitting regressions to represent the drying process of some of the main crops such as sunflower seeds (Smaniotto et al., 2017), rough rice (Cihan et al., 2007), corn (Asemu et al., 2020), and soybean (Rafiei et al., 2009). There, variables and parameters were evaluated and the best mathematical model was selected to accurately describe the drying phenomenon of the studied crops. For instance, Rafiei et al. (2009) emphasized that the particular behavior of the drying curve was associated with a falling-rate period because of moisture diffusion from the inside of the soybeans. Although these authors also used the two-term exponential model, the drying kinetic constant values reported at 50 °C (1.6344 and 0.1332 h<sup>-1</sup> for k<sub>1</sub> and k<sub>2</sub>, respectively) were not similar to those found in the present work for LS. However, the quotient value between both parameters (k<sub>1</sub>/ k<sub>2</sub> = 12.27) was closer to that obtained for LS, showing a comparable relation between both mass transfer periods.

#### 3.3. Effect of seed moisture content on some properties.

With the aim to study the effect of seed moisture on some physical and mechanical LS properties, seeds were dried from their storage condition in order to obtain samples with three different moisture contents (Table 4).

#### 3.3.1. Geometrical properties

Successful seed processing in dehulling, milling, heat conduction, and dryer selection depends on the geometrical properties of the material (Crapiste and Rotstein, 1997; Khodabakhshian et al., 2010). The dimensions of the LS at each moisture condition are shown in Table 5. The length of the seeds showed significant differences between the conditions M1 and M3 (p < 0.05), while the intermediate condition did not differ from the others. Also, no changes in seed width were found between M1 and M2, however, the driest condition M3 reported a significantly lower value (p < 0.05). Therefore, a dependence of length and width of LS with moisture content was observed, suggesting that dehydration leads to a reduction of at least one of their dimensions. This phenomenon has been reported in the literature for some grains and seeds such as ajwain (Singh and Meghwal, 2020), barley (Carvalho et al., 2021), fenugreek (Altuntas et al., 2005), melon (Mansouri et al., 2017), fennel (Ahmadi et al., 2009), jamun (Bajpai et al., 2020), nigella (Singh et al., 2015), kenaf (Izli, 2015), and Lathyrus (Kenghe et al., 2013). However, no significant changes in seed thickness were observed (p > 0.05), which could be attributed to the deformed shapes observed in LS. Moreover, the measurement

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Moisture content of LS conditioned by drying.

Nomenclature	M1	M2	M3
Moisture content (d.b.) (%)	8.28 ± 0.26 <sup>c</sup>	$4.62 \pm 0.25^{b}$	1.31 ± 0.10 <sup>a</sup>

\*The values presented are the mean of determination  $\pm$  standard deviation (n = 3). Means in the same row carrying different superscripts are significantly (p < 0.05) different, corresponding "a" with the lowest value.

#### Table 5

Geometrical, gravimetric, and mechanical properties of LS at the different moisture content.

Moisture condition	M1	M2	M3
Geometrical properties			
Length (mm)	11.815 ± 1.442 <sup>b</sup>	11.776 ± 1.771 <sup>ab</sup>	11.359 ± 1.502 a
Width (mm)	4.944 ± 0.729 <sup>b</sup>	4.831 ± 0.832 <sup>b</sup>	3.997 ± 0.762 <sup>a</sup>
Thickness (mm)	3.929 ± 0.704 <sup>a</sup>	3.852 ± 0.858 <sup>a</sup>	4.062 ± 1.050 <sup>a</sup>
Geometrical diameter, D <sub>g</sub> (mm)	$6.089 \pm 0.650^{b}$	$5.977 \pm 0.762^{b}$	5.615 ± 0.733 <sup>a</sup>
Projected area, Sg (mm <sup>2</sup> )	100.853 ± 20.586 <sup>b</sup>	97.958 ± 25.021 <sup>b</sup>	86.832 ± 21.056 <sup>a</sup>
Theoretical volume, Vg (mm <sup>3</sup> )	75.496 ± 23.790 <sup>b</sup>	72.235 ± 29.230 <sup>b</sup>	59.470 ± 22.413 <sup>a</sup>
Sphericity, $\phi$ (mm mm <sup>-1</sup> )	$0.519 \pm 0.053^{b}$	$0.512 \pm 0.059$ <sup>ab</sup>	$0.498 \pm 0.061$ <sup>a</sup>
Gravimetric properties			
Thousand seed weight, TSW (g)	71.171 ± 2.308 <sup>b</sup>	70.701 ± 0.878 <sup>b</sup>	64.757 ± 1.372 <sup>a</sup>
True volume, V <sub>t</sub> (mm <sup>3</sup> )	86.575 ± 1.332 <sup>b</sup>	85.043 ± 2.418 <sup>b</sup>	75.209 ± 1.593 <sup>a</sup>
Volume correction factor, K <sub>v</sub> (mm <sup>3</sup> mm <sup>-3</sup> )	$0.872 \pm 0.021^{b}$	$0.849 \pm 0.008^{b}$	0.790 ± 0.018 <sup>a</sup>
True density, $\rho_t$ (g mL <sup>-1</sup> )	0.822 ± 0.013 <sup>a</sup>	$0.832 \pm 0.024$ <sup>ab</sup>	$0.861 \pm 0.018^{b}$
Bulk density, $\rho_b$ (g mL <sup>-1</sup> )	0.523 ± 0.009 <sup>a</sup>	0.532 ± 0.009 <sup>a</sup>	0.525 ± 0.017 <sup>a</sup>
Bulk porosity, $\varepsilon_b$ (mm <sup>3</sup> mm <sup>-3</sup> )	0.364 ± 0.013 <sup>a</sup>	0.359 ± 0.034 <sup>a</sup>	0.389 ± 0.032 <sup>a</sup>
Mechanical properties			
Rupture force, R <sub>b</sub> (N)	48.033 ± 22.876 <sup>b</sup>	35.022 ± 18.508 <sup>a</sup>	31.091 ± 12.754 <sup>a</sup>
Deformation at the break, D <sub>b</sub> (%)	$28.029 \pm 12.475^{b}$	19.958 ± 7.222 <sup>a</sup>	20.458 ± 8.535 <sup>a</sup>
Rupture energy, $E_b$ (J m <sup>-3</sup> )	$426.976 \pm 370.760^{\rm b}$	201.141 ± 162.129 <sup>a</sup>	212.274 ± 180.856 <sup>a</sup>

\*The values presented are the mean of determination  $\pm$  standard deviation. Means in the same row carrying different superscripts are significantly (p < 0.05) different, corresponding "a" with the lowest value.

technique used did not allow the quantification of considerable changes, and therefore significant differences, due to the presence of superficial folds, as shown in Fig. 5.

During seed classification, the size of the screen holes must be evaluated according to the diameter of the seed, so this is an important parameter in the engineering design of the separation or sieving equipment. In addition, seed sphericity is an important property to characterize their tendency to roll, while surface area and other physical parameters are required to design hoppers and screens, among others, concerning size and inclination (Darfour et al., 2022). The geometrical parameters calculated with Eq. (3) to Eq. (6) and their moisture content dependence are also presented in Table 5. The geometrical parameters showed a decreasing trend as seed moisture reduces (p < 0.05). It was observed that the  $D_g$ ,  $S_g$ , and  $V_g$  did not differ significantly between the moisture conditions M1 and M2, while a decrease was observed at the lowest moisture condition (M3) (p < 0.05). Regarding  $\varphi$ , the intermediate moisture condition M2 did not differ from the others, although there were significant differences in this parameter between the extreme moisture conditions M1 and M3



Fig. 5. Appearance of LS before being subjected to the hot air drying process.

(p < 0.05). The reduction in the D<sub>g</sub>, S<sub>g</sub>, V<sub>g</sub> and  $\varphi$  of LS with decreasing moisture content is consistent with previous research on different seeds. Directly proportional differences in these parameters with moisture content have been published for ajwain (Singh and Meghwal, 2020), fenugreek (Meghwal and Goswami, 2012), melon seeds (Mansouri et al., 2017), nigella (Singh et al., 2015), black pepper (Meghwal and Goswami, 2011), and jamun (Bajpai et al., 2020). Similar behavior has been reported for surface and volume for kenaf (Izli, 2015), *Lathyrus* (Kenghe et al., 2013), black pepper (Meghwal and Goswami, 2011), and fenugreek (Altuntaş et al., 2005; Meghwal and Goswami, 2012). As already mentioned, water loss leads to a reduction of some seed dimensions, resulting in changes of the geometrical parameters.

#### 3.3.2. Gravimetric properties

The gravimetric properties of seeds are important parameters to be considered not only during classification and loading, as well as when designing drying, transportation, and storage equipment (Darfour et al., 2022). TSW,  $\rho_t$ ,  $\rho_b$  and  $\varepsilon_b$  at different moisture content are shown in Table 5. Not only TSW but also V<sub>t</sub> tended to decrease with a reduction in seed moisture, showing a significant decrease at the driest moisture condition (M3) (p < 0.05); however, no significant variations were observed between seeds at M1 and M2 (p > 0.05). Although there is not enough experimental data to demonstrate a linear dependence as reported by most authors, a loss in the weight of LS due to dehydration was observed. This behavior was also reported for some grains and seeds as barley (Carvalho et al., 2021), pomegranate (Dak et al., 2014), jamun (Bajpai et al., 2020), kenaf (Izli, 2015), quinoa (Altuntas et al., 2018), melon (Mansouri et al., 2017), millet (Jain and Bal, 1997; Balasubramanian and Viswanathan, 2010), fennel (Ahmadi et al., 2009), fenugreek (Altuntaş et al., 2005), and Lathyrus (Kenghe et al., 2013). As previously stated, this trend is associated with a reduction in the geometrical properties of the seeds as moisture content decreases, implying that condition M3 produced a smaller matrix than conditions M1 and M2 and therefore a reduction in its true volume.

The  $K_v$  of LS is a relevant parameter since it is not possible to describe the irregular shapes of the seed due to its multiple folds (Fig. 5) in the theoretical volume calculation. This factor was significantly lower for M3 seeds than for the other moisture conditions (M1 and M2) (p < 0.05). Seed dehydration generates more brittle and turgid structures, pronouncing the superficial roughness and

introducing larger differences between the experimental data  $(V_t)$  and the theoretical values  $(V_g)$ .

As expected,  $\rho_t$  increased as the moisture content decreased due to the reduction in V<sub>t</sub> and its inverse dependence, obtaining significant differences between the extreme seed moisture conditions (M1 and M3) (p < 0.05). Similar tendency was reported for multiple seeds, such as kenaf (Izli 2015), fennel (Ahmadi et al., 2009), millet (Jain and Bal 1997; Balasubramanian and Viswanathan, 2010), jamun (Bajpai et al., 2020), Amaranthus (Ilori and Akinyele, 2016), fenugreek (Altuntas et al., 2005), and barley (Carvalho et al., 2021). The values previously obtained show that the changes in V<sub>t</sub> were relatively higher than those corresponding to their mass, which is reflected in the value of the density obtained. Considering that  $\rho_b$  takes into account the void interstices of the bed,  $\rho_t$  was expected to be higher than  $\rho_b$ , as it was confirmed with  $\rho_t/\rho_b$  ratios higher than 1.55. However, no significant differences in  $\rho_b$  or  $\epsilon_b$  values were observed with variation in seed moisture content (p > 0.05). Although the bed volume was expected to compact and reduce as the volume of the individual seeds decreased, the superficial rugosity of the seeds may have prevented changes in the spatial conformation of the bed and therefore in  $\rho_{\rm b}$  and  $\varepsilon_{\rm b}$ . No significant differences were also reported for  $\rho_{\rm b}$  at different moisture content for pomegranate and bottle gourd, sponge gourd, garden pea, and radish seeds, respectively (Dak et al., 2014; Mishra et al., 2019).

#### 3.3.3. Mechanical properties

For some of the operations mentioned before, such as dehulling, milling, and handling of seeds, the knowledge of breaking parameters is required prior to defining suitable processing conditions. The R<sub>b</sub>, D<sub>b</sub>, and E<sub>b</sub> values for each moisture content studied are shown in Table 5, presenting significantly higher values for M1 (p < 0.05). The reduction of LS moisture content produced a decreasing effect in R<sub>b</sub>. According to some authors, R<sub>b</sub> decreased with increasing moisture content in soybean (Tavakoli et al., 2009), sorghum (Rodrigues et al., 2019), fennel (Ahmadi et al., 2009), and safflower (Baümler et al., 2006). In this study, the opposite structural behavior found could be related to the different nature of LS, since these seeds are located inside of a fruit at a high relative humidity and they are not exposed to the external environment like intensive crops are. Furthermore, the moisture content range established in the present work was relatively lower than those considered in the aforementioned studies. This specific working condition leads to the interpretation that the differences found between the trends might be related to the dissimilar moisture contents studied. Also, as mentioned above, reducing the moisture content generates more brittle structures that are prone to breaking, which causes a depletion of R<sub>b</sub>.

Moreover, D<sub>b</sub>, and E<sub>b</sub> of LS also decreased with moisture content reduction, as it is shown in Table 5 (p < 0.05). Similar trends in D<sub>b</sub> were reported for safflower (Baümler et al., 2006), fennel (Ahmadi et al., 2009), soybean (Tavakoli et al., 2009), and walnut (Altuntaş and Erkol, 2011). The brittle structures of LS obtained after their dehydration are more susceptible to breaking under less relevant deformations, resulting in matrices with lower compression resistance and, therefore, requiring a reduced amount of energy to break them. However, the absence of statistical differences for R<sub>b</sub>, D<sub>b</sub>, and E<sub>b</sub> values of LS obtained for the moisture content of M2 and M3 indicated that these may be mechanically similar.

## 4. Conclusions

Lemon seed conditioning process by air-drying was adequately represented by a two-term exponential model. The determined parameters of the drying kinetics indicated a differential contribution of the kernel and the hull as the drying process progressed. Furthermore, this revealed the higher resistance of the dried hull in conjunction with the inner part to water transfer.

The variation in moisture content proved to be a key factor related to the physical and mechanical properties of LS. The reduction of the moisture condition generated smaller matrices, reflected in the values for length, width, projected surface, and theoretical and true volume at the driest condition. In the same way, the reduction in the size of the seeds was shown indirectly on the values obtained for true density and sphericity under the conditions mentioned. Otherwise, the unchanged bulk density and porosity with variations in seed moisture content suggests that the bed has not been compacted, which allows inferring that the surface folds of the seed hull could be playing an important role in these parameters. Regarding the mechanical properties, more fragile structures were generated with seed dehydration, as reflected in a reduction in the rupture force, deformation, and energy with moisture content.

The driest condition generated substantial changes in the morphology and structure of the lemon seeds. Dehydration beyond the safe moisture condition could have positive implications, producing smaller and more fragile structures for their further treatment such as handling, dehulling, or crushing of seeds, among other unit operations. However, extensive seed drying could also generate fines when they are moved, causing mass losses.

#### **Declaration of Competing Interest**

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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