Determination of the volumetric shrinkage in jatropha seeds during drying

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ABSTRACT. The objective of this study was to determine the geometric diameter of volumetric shrinkage in Jatropha seeds subjected to drying under six air conditions and to fit different mathematical models to the experimental values. Seeds with an approximate initial moisture content of 0.61 kgw kgdm^{-1} (kilogram of water per kilogram of dry matter) were dried in a convection oven at temperatures of 36, 45, 60, 75, 90, and 105°C until they reached a moisture content of 0.11 ± 0.006 kgw kgdm^{-1}. The experimental data were fitted to five mathematical models used to represent volumetric shrinkage in agricultural products. The models were analyzed using the coefficient of determination, chi-square, mean relative error, mean estimated error, and residual distribution. Geometric diameter data were subjected to regression analysis by adopting a 1% level of significance. It was concluded that moisture content and drying temperature influence volumetric shrinkage in Jatropha seeds, a phenomenon satisfactorily described by the Polynomial equation, where the geometric diameter linearly decreased with a reduction of the moisture content, regardless of the drying conditions.
Keywords: mathematical models, moisture content, Polynomial model.

Determinação da contração volumétrica dos grãos de pinhão-manso durante a secagem

RESUMO. Objetivou-se com o presente trabalho determinar o diâmetro geométrico e ajustar diferentes modelos matemáticos aos valores experimentais da contração volumétrica dos grãos de pinhão-manso submetidos à secagem em seis condições de ar. Os grãos com teor de água inicial de 0,61 kga kgms^{-1} (kg de água por kg de matéria seca), aproximadamente, foram submetidos à secagem em estufa com circulação de ar forçada nas temperaturas de 36, 45, 60, 75, 90 e 105°C, até atingirem o teor de água de 0,11 ± 0,006 kga kgms^{-1}. Aos dados experimentais foram ajustados cinco modelos matemáticos utilizados para representação da contração volumétrica dos produtos agrícolas. Os modelos foram analisados por meio do coeficiente de determinação, teste de qui-quadrado, do erro médio relativo, do erro médio estimado, e da distribuição de resíduos. Os dados do diâmetro geométrico foram submetidos a análise de regressão adotando-se o nível de 1% de significância. Conclui-se que o teor de água e a temperatura de secagem influenciam na contração volumétrica dos grãos de pinhão-manso, sendo este fenômeno satisfatoriamente descrito pela equação Polinomial e, o diâmetro geométrico reduz linearmente com a redução do teor de água, independentemente da condição de secagem.
Palavras-chave: modelos matemáticos, teor de água, modelo Polinomial.

Introduction

With the current oil crisis, research on alternative fuels has intensified, and biodiesel appears to be one possible immediate solution that is capable of being produced using vegetable oils and animal fats (GOLDFARB et al., 2010). Jatropha (*Jatropha curcas* L.) is among those plants with potential for biodiesel production. Indeed, Souza et al. (2009) have found an average concentration of 40.33 ± 1.91% lipids in dry Jatropha seeds.

In addition to its capacity for producing oil with all of the qualities necessary for biodiesel transformation, the geographical distribution of Jatropha is widespread due to its hardiness, resistance to long droughts, pests, and disease, and its adaptability to a wide range of climate conditions (SANTOS et al., 2009). Although Jatropha is a plant that has the ability to adapt to diverse conditions, cultivation practices are essential for healthy plant development and, consequently, to achieve higher productivity.

Among the steps after harvest, drying is of great importance, as it is directly related to the quality of the final product and may result in irreversible damage to the seeds. According to Resende et al. (2005), reducing the moisture content in seeds not

only directly influences their physical properties, but also causes shrinking of the seed, a phenomenon known as volumetric shrinkage. Volumetric shrinkage can be very intensive, depending on the method and drying conditions (KROKIDA; MAROULIS, 1997), which affect the parameters of heat transfer and mass, and should be considered in the establishment of drying models (CORRÊA et al., 2006; RAMOS et al., 2003).

The volumetric shrinkage index determines the relationship between the seed volume, moisture content, and initial volume. This is of utmost importance during the drying process because it is possible to predict the behavior of the reduced volume occupied by the seed mass according to its moisture content reduction.

The development and improvement of equipment used for seed drying is also fundamentally important for drying simulation and to obtain theoretical information about the behavior of each product during water removal (RESENDE et al., 2010). For a simulation based on drying successively thin layers of the product, a mathematical model is used to satisfactorily represent water loss during the drying period (BERBERT et al., 1995; GINER; MASCHERONI, 2002).

According to Midilli et al. (2002), there are three types of models to represent thin layer drying, aimed at describing drying kinetics in agricultural products. These models include a theoretical model, which considers only the internal resistance and the transfer of heat and water between the product and the warm air, and semi-theoretical empirical models, which consider only the internal resistance, temperature, and relative humidity of the drying air.

Models used to represent drying in agricultural products have been developed, in most cases, by neglecting volumetric shrinkage in the product during the dehydration process (BROOKER et al., 1992). According to Ramos et al. (2005), changes in the product with regards to its volumetric shrinkage should also be included in models for the complete description, precision, and analysis of drying phenomena.

Given the prominence of Jatropha cultivation as an oilseed and the lack of theoretical information about the behavior of the seeds of this crop during the drying process, the goal of this study was to fit different mathematical models to experimental volumetric shrinkage data, identify the best model to represent the phenomenon, and determine the geometric diameter in Jatropha seeds submitted to drying under six air conditions.

Material and methods

The experiment was conducted at the Postharvest Laboratory of Plant Products at the Instituto Federal de Educação, Ciência e Tecnologia Goiano – *Campus* Rio Verde (IF Goiano – *Campus* Rio Verde).

Jatropha seeds with a moisture content of 0.61 kgw kgdm^{-1} were used. The seeds were dried in a convection oven under six temperature conditions, 36, 45, 60, 75, 90, and 105°C, and at relative humidities of 31.7, 19.6, 9.4, 4.8, 2.6, and 1.5%, respectively, until reaching a moisture content of 0.11 ± 0.006 kgw kgdm^{-1}, determined at 105 ± 1°C for 24h in three replicates (BRASIL, 2009).

For the drying, 20 Jatropha seeds were individually enclosed in aluminum capsules with dimensions of 60.12 mm in diameter and 41.0 mm in height. The seeds were measured at three positions (length, width, and thickness) at 15-min. intervals with the aid of a digital caliper of 0.01-mm resolution. The moisture content reduction was monitored by weighing three trays with 300 g of the product under the same conditions of seed drying.

To evaluate the drying in Jatropha seeds, the rate of water reduction from the product was determined, according to the following expression described by Corrêa et al. (2001):

$$TRA = \left[\left(Ma_0 - Ma_i \right) / Ms \cdot \left(t_i - t_0 \right) \right] \qquad (1)$$

TRA: water reduction rate, kg kg^{-1} h^{-1};
Ma$_0$: total previous water mass, kg;
Ma$_i$: total current water mass, kg;
Ms: dry matter, kg;
t$_0$: total previous drying time, h;
t$_i$: total current drying time, h.

The volumetric shrinkage index of Jatropha seeds during the drying was determined by the relationship between the seed volume to each moisture content and the initial volume, as shown below:

$$\psi = V / V_o \qquad (2)$$

ψ : volumetric shrinkage index, decimal;
V: volume for each moisture content, mm^3;
V$_o$: initial volume, mm^3.
The volume of each seed, in mm^3, was obtained throughout the drying process, according to the following expression proposed by Mohsenin (1986):

$$V_g = \left[\left(\pi \cdot a \cdot b \cdot c \right) / 6 \right] \qquad (3)$$

V$_g$: seed volume, mm^3;
a: major axis of the seed, mm;
b: medial axis of the seed, mm;
c: minor axis of the seed, mm.

The characteristic dimensions of the orthogonal axes in Jatropha seeds were determined with the seed in its natural resting position, as illustrated in Figure 1.
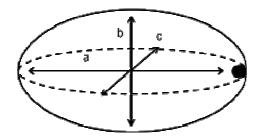

Figure 1. Schematic drawing of a Jatropha seed, considered spheroidal in shape, with its characteristic dimensions.

The geometric diameter of each seed was obtained throughout the drying process, according to the expression proposed by Mohsenin (1986), as follows:

$$D_g = (a \cdot b \cdot c)^{1/3} \tag{4}$$

D_g: geometric diameter, mm.

The experimental data of the unitary volumetric shrinkage index were fitted to the models described by the expressions listed in Table 1.

The mathematical models were fitted using nonlinear regression analysis by the Gauss-Newton method using a statistic software. The models were selected considering the magnitude of the coefficient of determination, the magnitude of the mean relative error, and the estimated error, chi-square, in addition to a verification of the behavior of residual distribution. Mean relative error values of less than 10% were considered as criteria for model selection, according to Mohapatra and Rao (2005).

The mean relative and estimated errors for each model were calculated conforming to the following expressions:

$$P = \left[(100/n) \sum \left(\left| Y - \hat{Y} \right| / Y \right) \right] \tag{10}$$

$$SE = \sqrt{\sum \left(Y - \hat{Y} \right)^2 / GLR} \tag{11}$$

$$\chi^2 = \left[\sum \left(Y - \hat{Y} \right)^2 \Big/ GLR \right] \tag{12}$$

P: mean relative error, %;
n: number of experimental observations;
$\hat{Y}$: the value calculated by the model;
Y: value experimentally observed;
SE: mean estimated error, decimal;
χ^2: chi-square.
GLR: degrees of freedom of the model, the number of observations minus the number of model parameters.

To assess the geometric diameter, the experiment was mounted in a factorial design, with factors such as different temperatures and moisture contents, the data were subjected to analysis of variance and regression adopting the 1% level of significance, with aid application program SISVAR 5.3 according to Ferreira (2008). Apart from this statistical analysis we used the mean relative error test to validate the model employed.

Results and discussion

Figure 2 shows the mean water removal rate in Jatropha seeds submitted to drying under six temperature conditions. The highest value was observed at a drying temperature of 105°C, with a magnitude of 0.1728 kg kg^{-1} h^{-1}. According to Resende et al. (2009), over the course of drying, the water reduction rate decreases because water is strongly bonded, requiring more energy for its evaporation. However, Figure 2 demonstrates that the water reduction rate increased at times, especially at temperatures of 36 and 45°C. This behavior was due to the greater distances between the reading intervals.

Table 1. Models used to simulate the volumetric shrinkage index in agricultural products.

Reference	Model	
Corrêa et al. (2004) - apud Corrêa et al. (2011)	$\psi = 1/\left[a_1 + b \cdot \exp(X) \right]$	(5)
Exponential - Exp.	$\psi = a_1 \cdot \exp(b \cdot X)$	(6)
Linear - Lin.	$\psi = a_1 + a_2 \cdot X$	(7)
Polynomial - Pol.	$\psi = a_1 + a_2 \cdot X + a_3 \cdot X^2$	(8)
Modified Bala and Woods (1984) - Mod. B. and W.	$\psi = 1 - a_1 \left\{ 1 - \exp\left[-a_2 \left(X_0 - X \right) \right] \right\}$	(9)

a_1, a_2, a_3 :parameters dependent on the product; X: moisture content of the product, kgw kgdm^{-1}; X_0: initial moisture content of the product, kgw kgdm^{-1}.
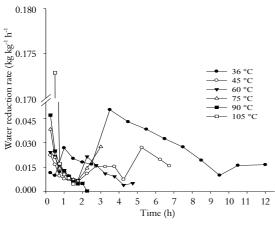
Figure 2. Water reduction rate (kg kg^{-1} h^{-1}) in Jatropha seeds subjected to drying under six air temperatures.

Figure 2 also shows that the period of the water reduction rate in Jatropha seeds was 1.5, 2.25, 3.0, 4.75, 6.75, and 12.0h at drying temperatures of 105, 90, 75, 60, 45, and 36°C, respectively, which corresponds to the time required for the seeds to reach a moisture content of 0.11 ± 0.006 kgw kgdm^{-1}. Therefore, increasing the temperature promoted a reduction of the drying time in Jatropha seeds, whereas higher temperatures also promoted a greater moisture gradient between the seed and drying air, which is an observation that has been made by many researchers of numerous agricultural products (AKPINAR et al., 2003; COSTA et al., 2011; LAHSASNI et al., 2004; MOHAPATRA; RAO, 2005; RESENDE et al., 2008; SIRISOMBOON; KITCHAIYA, 2009; SOUSA et al., 2011; ULLMANN et al., 2010).

Values for the mean estimated relative error (SE), mean relative error (P), and the coefficient of determination (R^2) for models fitted to the volumetric shrinkage index in Jatropha seeds subjected to drying under different temperature conditions are presented in Table 2. Regarding the coefficients of determination (R^2) relative to the adjusted mathematical models, note that only the polynomial and modified Bala and Woods models had values above 96%. Thus, the evaluation of other parameters, such as the mean estimated error, mean relative error, and residual distribution values, is necessary to choose the model that satisfactorily describes the volumetric shrinkage phenomenon.

As observed in the analyzed models, the mean estimated error (SE), which describes the standard deviation value for the estimate, showed little variation between the models and respective temperatures studied. However, the polynomial and modified Bala and Woods models showed the lowest SE values under all of the drying conditions. It is noteworthy that the lower the SE value was, the better the model fitted to the experimental data. Table 2 also shows that the models showed mean relative error values between 0.135 and 0.869%. According to Mohapatra and Rao (2005), mean relative error values below 10% indicate the adequate representation of the phenomenon. Thus, all of the models meet this criterion; however, as in the other evaluations, the polynomial and modified Bala and Woods models stand out. Corrêa et al. (2011) found that the modified Bala and Woods model best represented the volumetric shrinkage rate of red beans during drying.

Table 3 shows the residual distribution data for the models studied. The exponential and linear models showed a biased distribution under all of the conditions studied, and the polynomial model showed more drying conditions with random distribution, the desired behavior for the description of the volumetric shrinkage index in agricultural products.

Table 2. Coefficients of determination, mean relative error, and estimated error for the five models analyzed during volumetric shrinkage in Jatropha seeds under various temperature conditions (°C).

Temperature (°C)		Model				
		Cor.	Exp.	Lin.	Pol.	Mod. B. and W.
	SE	0.005	0.005	0.005	0.005	0.006
36	P	0.477	0.448	0.442	0.457	0.516
	R²	98.4	98.4	98.3	98.5	97.9
	SE	0.006	0.008	0.009	0.007	0.006
45	P	0.553	0.675	0.702	0.583	0.535
	R²	96.7	94.6	94.0	96.5	96.7
	SE	0.004	0.004	0.004	0.004	0.004
60	P	0.331	0.331	0.343	0.320	0.313
	R²	98.9	98.9	98.7	99.1	99.0
	SE	0.010	0.006	0.006	0.004	0.006
75	P	0.845	0.584	0.524	0.347	0.528
	R²	94.3	97.4	97.8	98.9	97.5
	SE	0.010	0.006	0.006	0.005	0.006
90	P	0.869	0.592	0.537	0.248	0.531
	R²	95.1	97.7	98.1	98.7	97.9
	SE	0.009	0.006	0.005	0.002	0.006
105	P	0.665	0.455	0.411	0.135	0.391
	R²	95.9	98.0	98.3	99.8	98.2

Illustrations of residual distribution (random for the polynomial model and biased for the model by Corrêa et al. (2004) apud Corrêa et al. (2011) during the modeling of volumetric shrinkage in Jatropha seeds subjected to drying at a temperature of 105°C are shown in Figure 3.
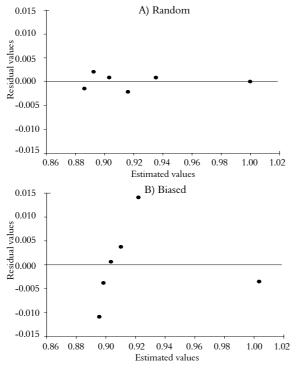

Figure 3. Illustration of the residual distribution during the modeling of volumetric shrinkage in Jatropha seeds: A) Random for the polynomial model; B) Biased for the model of Corrêa et al. (2004) apud Corrêa et al. (2011).

A model is considered random if the residual values found near the horizontal axis are close to zero and do not form defined figures, indicating no bias of the results. If a biased distribution is presented, the model is considered inadequate to represent the phenomenon in question (GONELI et al., 2011).

Table 4 presents the chi-square values obtained for the different models fitted to the experimental values of grain shrinkage of jatropha. It appears that the five models analyzed were found in the range of 99%. However, in general, the polynomial model was the one with the lowest chi-square. The lower the value of chi-square best fit of the model (AKPINAR et al., 2003; GÜNHAN et al., 2005; MIDILLI; KUCUK, 2003).

Data analysis indicates that the polynomial model is adequate for the mathematical description of the unitary volumetric shrinkage phenomenon in Jatropha seeds, demonstrating a high fitted coefficient of determination and reduced mean relative and estimated error values; therefore, the polynomial model was the model that showed the drying conditions with a random residual distribution. Afonso Júnior et al. (2003) have also observed that the polynomial model satisfactorily described volumetric shrinkage in coffee beans during drying.

Table 4 presents the coefficients of the polynomial model fitted to the experimental data of unitary volumetric shrinkage in Jatropha seeds subjected to drying at different temperatures.

As observed in Table 5, the coefficients of the polynomial model do not show a clear trend as a function of drying temperature. Thus, the unitary volumetric shrinkage in Jatropha seeds during drying occurred differently between the evaluated temperatures, as shown in Figure 4.

Table 3. Residual distribution for the models analyzed during volumetric shrinkage in Jatropha seeds under various temperature conditions.

Model	Temperature (°C)					
	36	45	60	75	90	105
Corrêa et al. (2004) apud Corrêa et al. (2011)	T	T	A	T	T	T
Exponential	T	T	T	T	T	T
Linear	T	T	T	T	T	T
Polynomial	T	T	A	A	T	A
Modified Bala and Woods (1984)	T	T	A	T	T	T

Table 4. Chi-square values calculated for the models used in the representation of grain shrinkage of jatropha.

Model	Temperature (°C)					
	36	45	60	75	90	105
Cor.	0.000031	0.000046	0.000019	0.000103	0.000103	0.000090
Exp.	0.000031	0.000074	0.000019	0.000047	0.000047	0.000043
Lin.	0.000034	0.000082	0.000023	0.000038	0.000039	0.000035
Pol.	0.000031	0.000052	0.000017	0.000021	0.000030	0.000004
Mod. B. and W.	0.000041	0.000045	0.000018	0.000044	0.000043	0.000039

Table 5. Coefficients of the polynomial model fitted to volumetric shrinkage in Jatropha seeds under different drying conditions.

Coefficients	Temperature (°C)					
	36	45	60	75	90	105
a_1	0.837^{**}	0.852^{**}	0.838^{**}	0.831^{**}	0.831^{**}	0.848^{**}
a_2	0.188^{**}	0.066^{ns}	0.185^{**}	0.378^{**}	0.356^{**}	0.386^{**}
a_3	0.096^{ns}	0.252^{**}	0.110^{*}	-0.175^{*}	-0.138^{ns}	-0.226^{*}

^{ns}Not significant, *significant at 5% and **significant at 1% probability by the F test.

The unitary volumetric shrinkage values, both experimental and estimated by the polynomial model, are shown in Figure 4. The index of volumetric shrinkage was 0.147, 0.151, 0.145, 0.132, 0.140, and 0.115 at temperatures of 36, 45, 60, 75, 90, and 105°C, respectively. Note that the volumetric shrinkage was lower at higher temperatures (105, 90, and 75°C), especially in the seeds subjected to drying at 105°C. At this temperature, there was a high water reduction rate at the onset of drying, resulting in hardening of the integument that impaired seed shrinkage.

Figure 4. Unitary volumetric shrinkage values in Jatropha seeds, both experimental and estimated by the polynomial model, due to moisture content reduction.

Figure 5 shows the geometric diameter values in Jatropha seeds subjected to drying under different temperature conditions. It was observed that, with drying, the geometric diameter decreased from 12.26, 12.35, 12.27, 12.16, 12.31, and 12.20 mm to 11.77, 1.74, 11.70, 11.54, 11.80, and 11.56 mm, at temperatures of 105, 90, 75, 60, 45, and 36°C, respectively. Thus, different drying conditions did not significantly promote changes in the reduction of the geometric diameter. Sirisomboon and Kitchaiya (2009) have found that geometric diameters in Jatropha seeds were 9.92, 9.37, and 9.97 mm, when subjected to drying at temperatures of 80, 60, and 40°C, respectively. The differences in the geometric diameter values could be related to the seed variety and handling technique adopted during periods of pre- and/or post-harvest.

Table 6 shows the linear models fitted to the experimental data of the geometric diameter in Jatropha seeds as a function of the moisture content. It was observed that the models showed a high degree of significance according to the F test and low values of mean relative error. Thus, they satisfactorily described the behavior of the variable analyzed. These results agree with those found by Razavi et al. (2007), working with pistachios and other nuts.
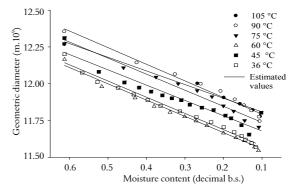

Figure 5. Geometric diameter in Jatropha seeds subjected to various drying conditions.

Table 6. Equations fitted to the geometric diameter values of Jatropha seeds as a function of moisture content.

Temperatura (°C)	Modelo	R² (%)	P (%)
105	$D_g = 0.927Ta + 11.71$	98.0**	0.15
90	$D_g = 1.130Ta + 11.68$	97.7**	0.19
75	$D_g = 1.091Ta + 11.62$	97.4**	0.19
60	$D_g = 1.104Ta + 11.44$	99.1**	0.11
45	$D_g = 1.039Ta + 11.57$	94.6**	0.27
36	$D_g = 1.100Ta + 11.46$	98.4**	0.15

**Significant at 1% probability by the F test.

Conclusion

Based on the results obtained, it can be concluded that the moisture content and drying temperature influence volumetric shrinkage in Jatropha seeds. The phenomenon satisfactorily described by the polynomial equation, where the geometric diameter linearly decreases with the reduction of moisture content, regardless of the drying conditions.

Acknowledgements

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