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# Desorption isotherms of *Lactuca sativa* seeds

Juliana S. Zeymer[1], Paulo C. Corrêa[1], Gabriel H. H. de Oliveira[2], Fernanda M. Baptestini[3] & Rita C. P. Freitas[1]

[1] Universidade Federal de Viçosa/Departamento de Engenharia Agrícola. Viçosa, MG. E-mail: juliana.zeymer@ufv.br; copace@ufv.br; rcrispfreitas@gmail.com
[2] Instituto Federal do Sudeste de Minas Gerais/Campus Manhuaçu. Manhuaçu, MG. E-mail: gabriel.oliveira@ifsudestemg.edu.br (Corresponding author)
[3] Universidade Federal do Espírito Santo/Departamento de Engenharia Rural. Alegre, ES. E-mail: fbaptestini@yahoo.com.br

**Key words:**
mathematical modeling
hygroscopic equilibrium
relative humidity
temperature

**A B S T R A C T**
*Lactuca sativa* seeds are highly sensitive to climate conditions; thus, they should be stored securely to maintain their qualitative and quantitative characteristics. Studies on hygroscopicity aim to decrease possible changes in agricultural products under specific environmental conditions. Accordingly, this study aims to develop an appropriate mathematical model to represent the desorption isotherms of *Lactuca sativa* seeds. The hygroscopic equilibrium was achieved using a static-gravimetric method at temperatures of 10, 20, 30, 40 and 50 °C and water activity in the range 0.11-0.96. Six mathematical models were fitted to the experimental data of the equilibrium moisture content of *Lactuca sativa* seeds. The best model was chosen based on the determination coefficient ($R^2$), magnitude of mean relative error (MRE), standard deviation of the estimate (SDE), and analysis of residue distribution. The modified Oswin model best represented the hygroscopicity of the *Lactuca sativa* seeds, with values of 8.02% and 0.55 for the MRE and SDE, respectively; moreover, the residual values were randomly distributed. The shape of the isotherms of the *Lactuca sativa* seeds estimated using the modified Oswin model is sigmoidal, which is characteristic of a type II curve.

**Palavras-chave:**
modelagem matemática
equilíbrio higroscópico
umidade relativa
temperatura

## Isotermas de dessorção de sementes de *Lactuca sativa*

**R E S U M O**
Sementes de *Lactuca sativa* são altamente sensíveis às condições edafoclimáticas; portanto, faz-se necessário armazená-las de forma segura e racional, a fim de manter suas características qualitativas e quantitativas. Estudos sobre a higroscopicidade têm a finalidade de amenizar possíveis alterações nos produtos agrícolas em determinadas condições climáticas. Dito isto, objetivou-se, neste trabalho, determinar o modelo matemático mais adequado para predizer as isotermas de dessorção de sementes de *Lactuca sativa*. O equilíbrio higroscópico foi alcançado por meio do método estático-gravimétrico, nas temperaturas de 10, 20, 30, 40 e 50 °C e atividade de água entre 0,11 e 0,96. Seis modelos matemáticos foram ajustados aos dados experimentais do teor de água de equilíbrio das sementes de *Lactuca sativa*. O modelo mais adequado foi escolhido considerando-se o coeficiente de determinação ($R^2$), magnitude do erro médio relativo (MRE), desvio padrão da estimativa (SE) e análise de distribuição de resíduos. O modelo de Oswin Modificado foi o que melhor representou a higroscopicidade de sementes de *Lactuca sativa*, apresentando valores de 8,02% e 0,55 de MRE e SE, respectivamente, e distribuição aleatória dos resíduos. As curvas isotérmicas de sementes de *Lactuca sativa* estimadas pelo modelo Oswin Modificado apresentaram formato sigmoidal, característica de curva do tipo II.

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## Introduction

*Lactuca sativa* is one of the most consumed verdure in Brazil and the third most consumed vegetable with respect to the volume of production, second only to watermelon and tomato (ABCSEM, 2015). According to ABCSEM (2015), *Lactuca sativa* returns annually an average of R$ 8 billion in retail, with a production capacity of more than 1.5 million tons per year.

*Lactuca sativa* seeds are highly sensitive to climate conditions; thus, they are stored securely to maintain their qualitative and quantitative characteristics until their effective use (Villela et al., 2010). Studies on hygroscopicity aim to minimize possible changes in agricultural products once they acquire the ability to exchange gas, including water in vapor form with the surrounding environment. These changes may occur because of the gain or loss of water, which are phenomena known as adsorption and desorption, respectively, based on the hygroscopic properties of the products and air (Brooker et al., 1992; Texeira Neto & Quast, 1993; Prado et al., 1999).

The characteristic curves of the equilibrium moisture content are referred to as sorption isotherms, which correlate the equilibrium moisture content of a product with relative humidity (Resende et al., 2006). Several studies have been conducted on hygroscopic behavior of various agricultural products, wherein different mathematical models were developed to express the equilibrium moisture content as a function of temperature and relative humidity. The studies include the following: *Phaseolus vulgaris* (Resende et al., 2006), *Ricinus communis* (Goneli et al., 2008), *Coffea arabica* L. (Corrêa et al., 2010), *Zea mays* L. (Oliveira et al., 2010), and *Brassica rapa* subsp. Rapa (Sousa et al., 2013).

With the help of studies conducted on hygroscopicity of *Lactuca sativa* seeds and the need to store the seeds appropriately, this study aimed to fit different mathematical models to the experimental data and obtain desorption isotherms of the seeds.

## Material and Methods

The current study was conducted in the Laboratory of Physical Properties and Quality of Agricultural Products at Federal University of Viçosa, Viçosa, MG.

*Lactuca sativa* seeds were purchased at the local market of Zona da Mata, MG. The mean initial moisture content of the seeds was 9.51% (d.b.), which was determined gravimetrically using a forced-air oven at 105 ± 1 °C for 24 h (BRASIL, 2009).

The static-gravimetric method was used to obtain hygroscopic equilibrium of the *Lactuca sativa* seeds by desorption (BRASIL, 2009). The experimental design was completely randomized with five levels of temperature (10, 20, 30, 40, and 50 °C), six levels of water activity (0.3, 0.4, 0.5, 0.6, 0.7, and 0.8 decimal d.b.), and four replicates. The water activity was determined using saturated salt solutions (Table 1), prepared using the methodology described by Dhingra & Sinclair (1995).

Samples of *Lactuca sativa* seeds of mass 20 g with three replicates were placed in aluminum crucibles. The samples were then stored in desiccators with their respective salt solutions at every relative humidity value. The desiccators were packed in a camera type B.O.D. (model 347 CD/brand Fanem), which was adjusted based on the specified temperatures.

Table 1. Values of water activity with respect to saturated salt solutions at temperatures of 10, 20, 30, 40, and 50 °C

| Saline solution | Temperature (°C) | | | | |
|---|---|---|---|---|---|
| | 10 | 20 | 30 | 40 | 50 |
| LiCl | 0.13 | 0.11 | 0.11 | 0.12 | 0.11 |
| $CaCl_2$ | 0.40 | 0.35 | - | - | - |
| $Ca(NO_3)_2$ | 0.59 | 0.55 | | | |
| $NH_4Cl$ | - | - | - | - | - |
| NaCl | 0.76 | 0.76 | 0.76 | 0.75 | 0.75 |
| KBr | - | 0.84 | - | - | - |
| $K_2S04$ | - | - | - | 0.96 | - |
| $MgCl_2$ | - | - | 0.32 | - | - |
| $KNO_2$ | - | - | 0.48 | - | - |
| $KNO_3$ | 0.96 | 0.93 | 0.91 | - | - |
| $MgCl_2$ x $6H_20$ | - | - | - | 0.32 | 0.31 |
| $Na_2Cr_20_7$ | - | - | - | 0.50 | 0.46 |

During the desorption process, the samples were weighed periodically at intervals of 24 h on an analytical balance (model AY220/brand Marte) until a constant weight was obtained. As a criterion to indicate the end of the experiment, a variation of less than 0.01 g or equal to this values, in three consecutive weight measurements, was adopted. If the readings repeat, the samples are considered to have reached the equilibrium moisture, where the partial pressure of water vapor of the product is equal to the partial pressure of ambient air steam inside the desiccator.

At the end of the sorption process, the equilibrium moisture content of the *Lactuca sativa* seeds was determined using gravimetric method, wherein the seeds are placed in an oven at 105 ± 1 °C for 24 h in two replicates (BRASIL, 2009).

Mathematical models conventionally used to predict the hygroscopicity of agricultural products were fitted to the experimental data of the equilibrium moisture content (Table 2).

Nonlinear regression was performed on a set of mathematical models using the Gauss–Newton method with

Table 2. Mathematical models used to represent sorption isotherms

| Model | Equation | |
|---|---|---|
| Chung-Pfost | $M = a - b\ln[-(T + c)\ln(A_w)]$ | (1) |
| Copace | $M = \exp[a - (bT) + (cA_w)]$ | (2) |
| Henderson | $M = \left[\frac{\log(1 - A_w)}{-a(T + 273.16)}\right]^{\frac{1}{b}}$ | (3) |
| Modified Henderson | $M = \left[\frac{\ln(1 - A_w)}{-a(T + b)}\right]^{\frac{1}{c}}$ | (4) |
| Modified Halsey | $M = \left[\frac{\exp(a - bT)}{-\ln(A_w)}\right]^{\frac{1}{c}}$ | (5) |
| Modified Oswin | $M = (a + bT)\left[\frac{A_w}{1 - A_w}\right]^{\frac{1}{c}}$ | (6) |

M - Equilibrium moisture content, % d.b.; Aw - Water activity, decimal; T - Temperature, °C; a, b, and c - Coefficients that depend on the product the help of Statistica software 7.0® (Statsoft, 2004). The best model was chosen based on the determination coefficient ($R^2$), magnitude of mean relative error (MRE), standard deviation of the estimate (SDE), and residual plots. For a good mathematical fit, MRE must be less than 10%, $R^2$ near unity, SDE close to zero, and residues randomly distributed (Resende et al., 2006; Lima et al., 2008) (Eqs. 7 and 8).

$$SDE = \sqrt{\frac{\sum_{i=1}^{n}\left(Y_i - \hat{Y}_i\right)^2}{DF}} \tag{7}$$

$$MRE = \frac{100}{n}\sum_{i=1}^{n}\frac{\left|Y_i - \hat{Y}_i\right|}{Y_i} \tag{8}$$

where:

$Y_i$ - is the observed value, % d.b.;

$\hat{Y}_i$ - is the value estimated by the model, % d.b.;

n - refers to the number of observed data; and,

DF - denotes the degree of freedom of the model (the number of model parameters subtracted from the number of observed data).

## Results and Discussion

Except for the Henderson model, other models showed high values for the coefficient of determination (> 0.96), thereby satisfactorily representing the phenomenon under study (Madamba et al., 1996; Resende et al., 2006; Faria et al., 2012) (Table 3). However, for a more detailed analysis, other statistical parameters were used to supplement the selection of the best model.

According to Draper & Smith (1998), the ability of a model to adequately describe a physical process is inversely proportional to SDE values. As such, analyzing Table 3, the modified Oswin, Chung Pfost, modified Henderson, and Copace models showed lower values of SDE as compared to the others. The MRE values were below 10% for the modified Oswin, modified Henderson, and Copace models, thereby adequately representing the phenomenon studied (Henao et al., 2009; Rosa et al., 2010; Corrêa et al., 2016).

Table 3. Model parameters of hygroscopic equilibrium of lettuce seeds obtained by desorption, with its respective determination coefficients ($R^2$), standard deviation of the estimate (SDE), mean relative error (MRE), and residual plot

| Model | Parameters* | $R^2$ | SDE (decimal) | MRE (%) | Residual plot |
|---|---|---|---|---|---|
| Chung-Pfost | a = 19.6633<br>b = 3.8084<br>c = 10.6186 | 0.9787 | 0.75 | 10.39 | Biased |
| Copace | a = 1.2707<br>b = 0.0121<br>c = 1.9622 | 0.97.56 | 0.82 | 8.13 | Random |
| Henderson | a = 0.0001<br>b = 1.6023 | 0.9219 | 1.35 | 17.58 | Random |
| Modified Henderson | a = 0.0004<br>b = 25.6494<br>c = 1.7147 | 0.9776 | 0.76 | 9.58 | Random |
| Modified Halsey | a = 5.8293<br>b = 0.0338<br>c = 2.7345 | 0.9702 | 0.91 | 13.23 | Biased |
| Modified Oswin | a = 9.5790<br>b = -0.0887<br>c = 3.2309 | 0.9888 | 0.55 | 8.02 | Random |

* Significant at 0.01 probability by "t" test

Finally, the distribution of the residues should be considered to select the best model, which is the difference between the experimentally observed values and the values estimated using the model. Corrêa et al. (2014) considered a model as random if the residual values meet the next horizontal band at approximately zero, and when no defined or forming geometrical figures are observed, indicating unbiased results.

Figure 1 shows that the trend exhibited by the residues of the modified Oswin, modified Henderson, and Copace models to describe the equilibrium moisture contents of the *Lactuca sativa* seeds are random, showing no tendency of specific points.

Thus, in addition to presenting lower magnitudes of MRE and SDE, the modified Oswin, modified Henderson, and Copace models exhibited higher determination coefficient ($R^2$) and random distribution of residual values. The modified Oswin model best correlated the estimated and experimentally observed values as compared to the modified Henderson and Copace models.

Accordingly, the modified Oswin model was chosen to represent the experimental data of the equilibrium moisture content of the *Lactuca sativa* seeds. Figure 2 shows the desorption isotherms of the lettuce seeds estimated using the modified Oswin model and the experimental values of the equilibrium moisture content.

For a constant water activity, the equilibrium moisture content of the *Lactuca sativa* seeds decreased with increase in the temperature; this trend was also reported by previous studies (Caetano et al., 2012; Smaniotto et al., 2012; Sousa et al., 2013) (Figure 2). According to Mazza & LeMaguer (1980), this tendency is due to a reduction in the number of active sorption sites required to bind water owing to physical and chemical changes in the product induced by temperature.

Figure 2 shows that the temperature effect is most pronounced in water activities above 0.60. According to Alves et al. (2015), at water activities below 0.60, water becomes increasingly linked to the food substrate with moderate forces, reaching high-intensity forces at very low water activities. Thus, the temperature required to remove the moisture when water activity is less than 0.60 becomes less effective, as the binding force between water molecules and the product constituents is high, requiring an increase in the temperature to observe its effect.

The shape of the desorption isotherms of the *Lactuca sativa* seeds estimated using the modified Oswin model is sigmoidal, which is characteristic of type II curves (Brunauer et al., 1940). The same authors classified the isotherms of food by the ability of the pores to adsorb gases through forces such as van der Waals.

The sigmoidal isotherms are generally observed for most agricultural products, particularly vegetable seeds, as reported

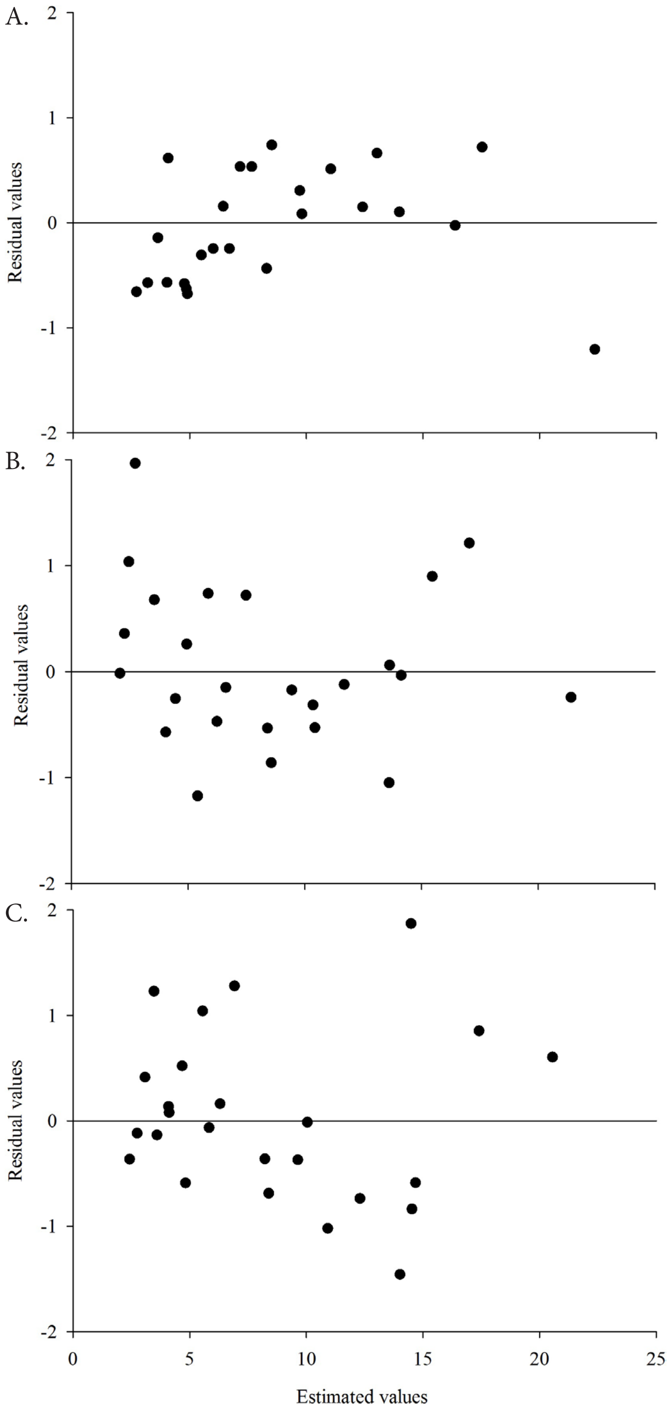

Figure 1. Residual plots of desorption process of lettuce seeds using mathematical models: modified Oswin (A), modified Henderson (B), and Copace (C)

in previous studies: *Abelmoschus esculentus* (Goneli et al., 2010), *Capsicum* L. (Ferreira et al., 2011; Rodovalho et al., 2015; Silva et al., 2015), and *Beta vulgaris* (Corrêa et al., 2016).

The shape of the isotherms depends on the physical structure and chemical composition of the product. *Lactuca sativa* seeds are small with low mass and irregular shape; in general, most vegetable-crop seeds have similar characteristics, which explain the similarity of these results with those observed in other vegetable crops (Silva et al., 2015).

Fabra et al. (2009) stated that the typical shape of an isothermal curve reflects the way in which water binds the product layering system. Thus, the weak interactions between the water molecules

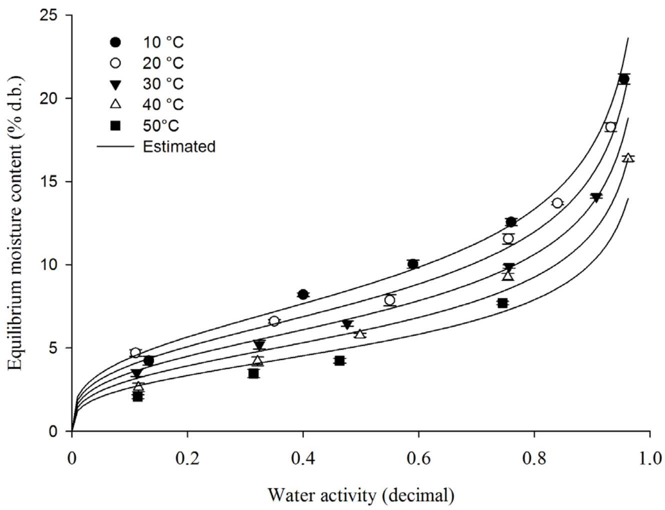

Figure 2. Observed and estimated values of equilibrium moisture content obtained by desorption of *Lactuca sativa* seeds using modified Oswin model

and surface of the product generate high water activity, thereby making the product unstable. When the interactions are strong, the water activity is low and the product is more stable, reducing the chances of deterioration during storage.

## Conclusions

1. The modified Oswin model was chosen to represent the desorption isotherms of the *Lactuca sativa* seeds, with values 8.02% and 0.55 for the MRE and SDE, respectively.

2. For a constant water activity, increased temperature promotes the reduction in equilibrium moisture content of the *Lactuca sativa* seeds.

3. The shape of the desorption isotherms estimated using the modified Oswin model is sigmoidal, which is characteristic of type II curves.

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