CIÊNCIA e NATURA

ISSN 2179-460X
OPEN ACCESS

Ci. e Nat., Santa Maria, v. 46, e73679, 2024 • https://doi.org/10.5902/2179460X73679
Submitted: 31/12/2022 • Approved: 22/11/2023 • Published: 10/04/2024

Chemistry

Drying study and phytochemical profile of the aqueous extract of the scaly leaf bulb of *Allium cepa* L (var.roxa)

Estudo da secagem e perfil fitoquímico do extrato aquoso do bulbo foliar escamoso de *Allium cepa* L (var.roxa)

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ABSTRACT

The objective of this study was to evaluate the phytochemical profile, predict drying kinetics, and quantify the content of total phenolics and flavonoids present in the aqueous extract of the bulb of scamiform leaves of *Allium cepa* L. The plant material was collected in the municipality of São Luís (MA). For the preparation of the aqueous extract, the cold maceration process was used using distilled water as a solvent. For the study of the drying kinetics, 10 g of the material *in natura* was submitted to a convective air oven at 45 °C/4h. The statistical parameters were determined by nonlinear regression using the Statistica 10.0 software, using the Quasi-Newton method. To determine the total phenolic content, the Folin-Ciocalteu methodology was used. And for the total flavonoids, the aluminum complexation assay was used. The determination of phenolics and total flavonoids revealed significant quantities in the extract, with values of 106.54 mg EAT g^{-1} and 26.36 mg EAT g^{-1}, respectively. According to the prediction of the statistical data, it was verified that the mathematical methods of Midilli-Kucuk and Verma proved to be adequate to describe the drying process of *Allium cepa* L. Through the results obtained, the mathematical model was determined for the drying kinetics of the squamous leaf bulb *Allium cepa* L, in addition to attesting the presence of secondary metabolites, thus making the product fit for biologically active activities.

Keywords: Aqueous extract; Mathematical models; Phytochemical profile

RESUMO

Este trabalho teve como objetivo avaliar o perfil fitoquímico e a predição da cinética de secagem, e quantificar o conteúdo dos fenólicos e flavonoides totais presente no extrato aquoso do bulbo de folhas escamiformes de *Allium cepa* L. O material vegetal foi coletado no munícipio de São Luís (MA). Para o preparo do extrato aquoso, foi empregado o processo de maceração a frio utilizando-se como solvente água destilada. Para o estudo da cinética de secagem, submeteu-se 10 g do material *in natura* a estufa de ar convectivo a 45 °C/4h. Os parâmetros estatísticos foram determinados por regressão não linear através do Software Statistica 10.0, pelo método Quase-Newton. Para determinação do conteúdo fenólico total empregou-se a metodologia de Folin-Ciocalteu. Para os flavonoides totais, utilizou-se o ensaio de complexação com alumínio. A determinação de fenólicos e flavonoides totais, apresentaram quantitativos significativos de 106,54 mg EAT g^{-1} e 26,36 mg EAT g^{-1} respectivamente. De acordo com predição dos dados estatísticos, verificou-se que métodos matemáticos Midilli-Kucuk e Verma mostraram-se adequados para descrever o processo de secagem de *Allium cepa* L. Por meio dos resultados obtidos, determinou-se o modelo matemático condizente para cinética de secagem do bulbo de folha escamosa *Allium cepa* L, além de atestar a presença de metabólitos secundários, tornando assim o produto apto para atividades biologicamente ativas.

Palavras-chave: Extrato aquoso; Modelos matemáticos; Perfil fitoquímico

1 INTRODUCTION

The interest in natural products and their herbal and medicinal actions is something that presents a well-defined historical context in terms of contributions to humanity. In this sense, they are used as raw material in various industrial sectors such as food, cosmetics and pharmaceuticals. Their use is recurrent due to their low cost and their excellent therapeutic properties derived from their active ingredients (Carneiro, 2019; Leite, 2019).

It is notorious that there is an extensive punctiform in relation to studies in the area of natural products that have in their laboratory routines of isolation, purification and elucidation of their structural composition, a series of biotechnological activities, with the intention of designating through the phytochemical study of extracts based on plant materials, the substances responsible for conferring the ability to present themselves as bioactive (Soares *et al.*, 2016).

Plant extracts usually come from plants that are known as spices and for their aromaticity, being used as anti-inflammatories and analgesics in the form of teas and infusions. Among these plants, the species *Allium cepa* L (red onion) stands out, which contains a significant content of total phenolics in its composition, with emphasis on quercetins and anthocyanins both for the purple and yellow varieties (Pereira, 2021).

In view of the mentioned secondary metabolites, an operation considered essential is precisely the drying kinetics, to maintain their production through metabolic processes carried out by enzymes, since they require a demand for water to carry out their activities. Then some mathematical models are used, with the purpose of simulating and evaluating the loss of water by the thin tissue during a certain time to assess a certain degree of confidence in relation to the process. This operation is fundamental in accordance with the needs of pharmaceutical industries of herbal medicines, as it does not have a space or structure to use plants *in natura* (Garcia *et al*., 2019). Thus, the present study aimed to evaluate the study of drying kinetics and phytochemical profile of the scaly leaf bulb of the aqueous extract of *Allium cepa* L.

2 METHODOLOGY

2.1 Plant material

The collections of the plant material used in this research were carried out during the period from February to July 2022. The scaly leaf bulbs of *Allium cepa* L. were obtained in the municipality of São Luís, Maranhão, Brazil (-2.549085,-44, 266149), identified by the Herbarium of the Federal University of Maranhão. After collection, the samples were transported to the Laboratory for Research and Application of Essential Oils (LOEPAV/UFMA).

In the laboratory, the plant material was analyzed and the visual aspects of the undamaged bulbs were selected. The bulbs were cut manually, with caution in standardizing the cuts. The cuts were made in a radial manner in pieces of a maximum of 4 cm in length and 1 cm in width. The samples were subjected to initial moisture content determination and drying in a FANEM 520 convective air oven at 45°C/24h during the kinetic study.

2.2 Drying

To carry out drying, a digital convective air drying oven FANEM 520 was used, standard air speed at 1 m/s. Drying was carried out on alternate days using a temperature of 45 °C and relative humidity monitored through a digital thermo-hygrometer (model INS-28 Intrusul).

A mass of 10 g of samples was used on aluminum-coated plates measuring 90 x 15 mm, the mass being monitored throughout the process by discontinuous weighing on an analytical scale Shimadzu AUY220 and the weighing process following intervals of 30 min in 30 min during the period of 4 h, until the end of the process. Weighings were carried out until mass variations were insignificant. Drying was completed when there was no mass variation of 0.0100 g between five successive weighings (Guterres *et al*., 2022). To determine the moisture ratio (RU) during the drying of red onion skins (*Allium cepa* L) for the different drying temperatures, Equation 1 was used (Guterres *et al*., 2022,; Cavalcante *et al.*, 2020):

$$RU_{(adm)}=\frac{Ubs\text{-}\ U_e}{Ubs_{inicial}\text{-}Ue} \tag{1}$$

Where; RU $_{(adm)}$ = moisture ratio, (dimensionless); $_{initial}$ Ubs =initial water content (bs); Ue=equilibrium water content (bs); Ubs=water content at time t (bs).

2.3 Mathematical modeling for drying prediction

The RU values obtained for each drying air temperature were analyzed using six different empirical and semi-empirical equations and non-linear regression, as shown in Table 1.

To adjust the mathematical models to the experimental data, a non-linear regression analysis was performed using the QuasiNewton method , using the Statistica 10.0 software.

Table 1 – The respective mathematical models to predict the drying kinetics

Model	Equation	Reference
Newton	RU=exp(-kt)	(Fogaça *et al*., 2021)
Page	RU=exp(- kt ^{n})	(Barros *et al*., 2020a)
Page Modified	RU=exp[(- kt)^n]	(Santos *et al*., 2021)
Herderson & Pabis	RU=a.exp(-kt)	(Sousa *et al*., 2020)
Logarithmic	RU=a.exp(- kt) + c	(Sousa *et al*., 2021)
Two Terms	RU=a.exp(-k $_0$ t) + b.exp (-k $_1$ t)	(Barros *et al*., 2020b)
Two Exponential Terms	RU=a.exp(- kt) + (1-a).exp (-kat)	(Martim , 2019)
Herderson & Pabis Modified	RU=a.exp(- kt) + b.exp(-gt) + c.exp (- ht)	(Karathanos *et al.*, 1999)
worm	RU=a.exp(- kt) + (1-a).exp (- gt)	(Erbay & Icier, 2010)
Midilli-Kucuk	RU=a.exp(- kt^n) + bt	(Morais *et al*., 2013)

Source: Guterres (2022)

The criteria used to determine the best fit of the models to the experimental data was the coefficient of determination (R^2) and the mean squared deviation (DQM) by Equation 2.

$$DQM=\sqrt{\frac{\Sigma(RU_{exp}-RU_{pre})^2}{N}} \quad (2)$$

2.4 Obtaining the aqueous extract

For the preparation of the aqueous extract, 200 g of the scaly leaf bulb were used in distilled water, following the proportion 1:10, by the method of cold maceration for 24 h at room temperature. At the end of 24 hours, the material was filtered and dried in an oven to eliminate residual solvent. Subsequently, stored for the necessary tests (Harborne, 1998).

Figure 1 – Aqueous Extract of *Allium cepa*

Source: Authors (2022)

2.5 Phytochemical Prospecting

The aqueous extract obtained was subjected to chemical tests based on the methodology presented by Matos (2009). The tests carried out to identify alkaloids, steroids, phenolics, flavonoids, glycosides, cardiac glycosides, saponins and tannins.

2.5.1 Steroids (Salkowsk test)

About 100 mg of dry extract was dissolved in 2 mL of chloroform. Sulfuric acid was carefully added to form a lower layer. A reddish brown color at the interface indicated the presence of a ringsteroid.

2.5.2 Alkaloids (Mayer's test)

1.36 mg of mercury chloride were dissolved in 60 mL and 5 mg of potassium iodide dissolved in 10 mL of distilled water, respectively. These two solvents were mixed and diluted to 100 mL using distilled water. A few drops of the previously prepared reagent were added to 1 mL of the acidic aqueous solution of the samples. The formation of white or pale precipitation showed the presence of alkaloids.

2.5.3 Flavonoids

In a test tube containing 0.5 mL of alcoholic extract from the samples, 5 to 10 drops of diluted HCl were added and a small amount of Zn or Mg were added to the solution, which was then boiled for a few minutes. The appearance of a reddish pink or dark brown color indicated the presence of flavonoids.

2.5.4 Glycosides

A small amount of alcoholic extract from samples was dissolved in 1 mL of water and then aqueous sodium hydroxide was added. The formation of a yellow color indicated the presence of carbohydrates.

2.5.5 Cardiac glycosides [Keller killiani test]

About 100 mg of extract was dissolved in 1 mL of glacial acetic acid containing one drop of ferric chloride solution and 1 mL of concentrated sulfuric acid was added. A brown ring obtained at the interface indicated the presence of an oxy sugar characteristic of cardenolides.

2.5.6 Saponins

One drop of baking soda was added to a test tube containing about 50 mL of an aqueous extract of the sample. The mixture was shaken vigorously and held for 3 min. A honey-foam comb was formed and showed the presence of saponins.

2.5.7 Phenols [Deronic Chloride Test]

For 1 mL of alcoholic sample solution, 2 mL of distilled water was added followed by a few drops of 10% aqueous ferric chloride solution. The formation of a blue or green color indicated the presence of phenols.

2.5.8 Tannins [Lead Acetate Test]

In a test tube containing about 5 mL of an aqueous extract, a few drops of 1% lead acetate solution were added. The formation of a yellow or red precipitate indicated the presence of tannins.

2.6 Total Phenolics

The phenol content was determined for the aqueous extract by the Folin-Ciocalteu spectrophotometric method (Lugasi *et al*., 1998; Oliveira *et al*., 2009).

The aqueous extract was diluted in ethanol to obtain solutions with a concentration of 10 mg mL^{-1}. To an aliquot of 0.1 mL of each solution were added 7 mL of distilled water, 800 μL of 10% Folin-Ciocalteu reagent and 2.0 mL of 20% sodium carbonate. After 2 hours, the reading was performed in a UV-Vis spectrophotometer, at a wavelength of 760 nm. As a reference, a standard curve of tannic acid was obtained, which provided the straight line equation for converting the measured absorbance into equivalent milligrams of tannic acid per gram of extract (mg EAT g^{-1}).

2.7 Total Flavonoids

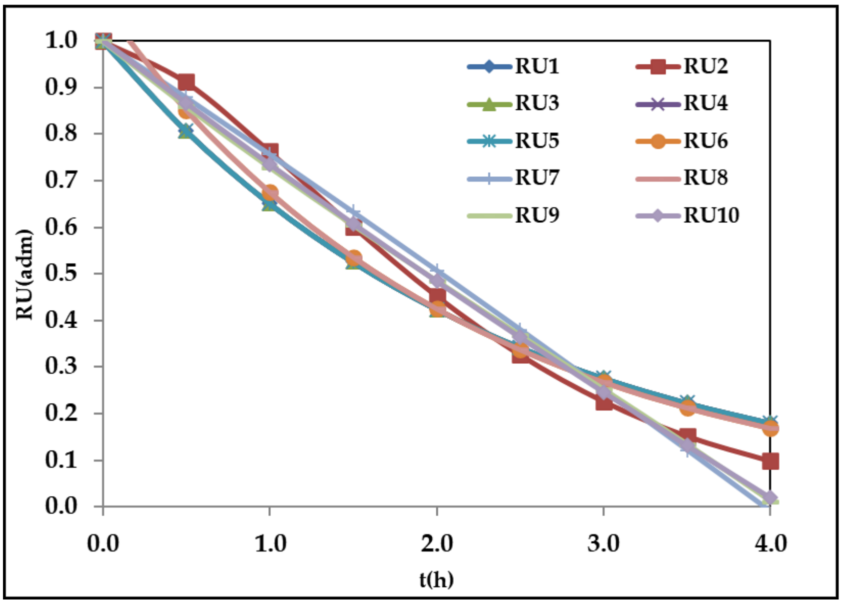
The total flavonoid content was estimated spectrophotometrically by reaction with $AlCl_{3}$, using quercetin as standard (Dowd, 1959; Woiskyesalatino, 1998; Frederice *et al.*, 2010). The aqueous extracts were diluted in methanol to obtain solutions with a concentration of 10 mg mL^{-1} . To a 0.2 mL aliquot of this solution was added 4.4 mL of EtOH and 0.4 mL of 2% $AlCl_3$ aqueous solution. After 30 minutes, the absorbances of the samples were measured at 425 nm. As a reference, an analytical curve was obtained with quercetin, which provided the straight line equation for converting the measured absorbance into equivalent milligrams of quercetin per gram of extract (mg EQ g^{-1}).

3 RESULTS AND DISCUSSION

3.1 Drying

Figure 2 shows the curves representing the drying of scaly leaf bulb *Allium cepa* L bark under conditions of constant temperature.

Figure 2 – Representation of the behavior of mathematical models



where ; RU1-Newton; RU2- Page; RU3- Modified Page; RU4- Herderson & Pabis ; RU5- Logarithmic; RU6- Two Terms ; RU7- Two terms Exponential; RU8- Herderson & Pabis Modified; RU9- Worm ; RU10 -Midilli-Kucuk .

Source: Authors(2022)

It was observed through Figure 2, that when starting the drying process of the scaly leaf bulb of *Allium cepa* L, there is consistency in relation to its drying rate until dehydration has taken place for a period of 2 hours, however after this time there is a reversal in the behavior of the graph showing itself as a descending curve.

This behavior attributed by the curves is justified by the fact that the surface of the material has become absent of saturation with water and its respective drying rate is evaluated through the diffusion of moisture inside the solid to the surface, from this context there is linearization of the curves. Thus, the rate of water reduction in the face of the mathematical models tested did not perceive abrupt changes in relation to its peaks, thus determining the efficiency of the experiment carried out (Silva *et al.*, 2018).

In relation to this process, several parameters concentrated in different mathematical models were made possible to attest to the drying kinetics, these evaluations were properly guided and predicted by the Statistica software 10.0.

The values of the variables (a, b, n, k, c, k_1, g, h, k_0) are presented in Table 3.

Table 3 – Variables of drying prediction models for *A. cepa*

Model	k	The	B	n	ç	k_1	g	H	k_0
1	0.4285	-	-	-	-	-	-	-	-
2	0.2708	-	-	1.5493	-	-	-	-	-
3	0.6546	-	-	0.6546	-	-	-	-	-
4	0.4633	1.0739	-	-	-	-	-	-	-
5	0.6546	-	-	0.6546	-	-	-	-	-
6	-	0.9587	0.1152	-	-	0.4633	-	-	0.4633
7	-0.0019	12.1943	-	-	-	-	-	-	-
8	0.4633	0.3558	0.4886	-	0.2295	-	0.4634	0.4630	-
9	-0.3401	-0.1121	-	-	-	-	0.2265	-	-
10	0.3599	1.0012	-0.1441	0.3599	-	-	-	-	-

where ; 1- Newton **;** two- Page **;** 3- Page Modified **;** 4- Herderson & Pabis ; 5- Logarithmic ; 6- Two Terms; 7- Two exponential terms;8- Modified Herderson & Pabis ; 9 -Worm ; 10 -Midilli-Kucuk .

Source: Guterres (2022)

Statistical parameters for mathematical models with correlation coefficients, squared deviation (DQM) and mean relative error (P) are shown in Table 4.

Table 4 – Statistical parameters for drying *A. cepa*

Model	DQM	P	R^2
1	0.0799	0.0324	0.9676
2	0.0398	-0.0919	0.9921
3	0.0799	0.0324	0.9676
4	0.0799	0.0324	0.9727
5	0.0799	0.0324	0.9991
6	0.0734	-0.1113	0.9727
7	0.0225	0.0804	0.9975
8	0.0734	0.1114	0.9727
9	0.0121	0.0108	0.999
10	0.0128	0.0001	0.9992

where ; 1-Newton; 2- Page; 3- Modified Page; 4- Herderson & Pabis ; 5- Logarithmic ; 6- Two Terms; 7- Two exponential terms;8- Modified Herderson & Pabis ; 9 -Worm ; 10 -Midilli-Kucuk .

Source: Guterres (2022)

As shown in Table 4, the mathematical models of drying kinetics used in the present study demonstrated adequate adjustment ($R^2 > 0.90$).

The models that stood out were by Verma and Midilli-Kucuk, this statement is based on the parameters established in Table 4, since they are structured in the highest correlation coefficient of (≥ 0.98) and lowest mean square deviations, as well through the observed parameters, the highlighted models are more suitable for analyzing the prediction of the drying kinetics of the scaly leaf bulb of *A. cepa* L according to the conditions that were exposed.

3.2 phytochemical prospecting

Table 5 presents the secondary metabolites identified for the aqueous extract of *A. cepa* L .

Table 5 – Phytochemical profile of the aqueous extract of *A. cepa*

Classes	**1**	**two**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
Allium cepa L	-	+	+	-	-	+	+	-	-

where ; 1-Alkaloids;2:Steroids;3:Phenols;4:Phenols;5:Flavonoids;6:Glycosides;7:Cardiac Glycosides; 8:Saponins;9:Tanins;(+) presence;(-) absence.

Source : Guterres (2022)

According to Table 5, the presence of steroids can be attested in the evaluated extracts, this metabolite confers functions and properties of regulation and metabolism of neuromuscular tissues, analgesic, anti-inflammatory, antimicrobial, in addition to having vitamin D in its composition as described by the essays of (Romam Junior *et al*., 2015; Santana *et al*., 2017).

According to Keyghobadi *et al*. (2021) through phytochemical prospecting, it was observed by the aqueous extract of the *A. cepa bulb*, some classes of secondary metabolites with pharmacological principles, among them are steroids in addition to phenolics, flavonoids, saponins, polyphenols and quercetin. Thus, the described organic classes have properties to suppress lipid oxidation, thus they can be useful and applicable for the functional food industry.

Cardiac glycosides are conceptualized as compounds that contain a steroid nucleus, taking into account that this nucleus has two hydroxyls. This class of secondary metabolite has the ability to enhance the strength of the heart and its frequency of contractions, and is employed in the cellular sodium-potassium ATPase pump. In this context, cardiac glycosides are applied in drugs for heart disease and anticancer activities (Botelho *et al*., 2019; Reddy *et al.*, 2020).

In this conjecture, another class that was identified is phenolic, which is conceptualized for its diversity in terms of structures, and thus encompassing a significant variety with around 10,000 compounds already identified, being considered abundant, its characterization and identification becomes effective. It should be noted that their presence is associated with the ability to neutralize oxidative species, that is, they behave as antioxidant agents, classifying them in such a way as to inhibit oxidative chain reactions in our body. In this scenario, it can be said that such attributions prevent neurological and cardiovascular diseases, diabetes and cancer (Oliveira *et al.*, 2022). In this way, compounds rich in phenolics attenuate the action of pro-oxidants, as they are substances responsible for triggering oxidative processes (Oliveira *et al.*, 2022).

3.3 Quantification of total phenolic and flavonoid content

It is evident that through Table 6, the quantitative assessment of total phenolics and flavonoids in the aqueous extract of *A. cepa can be carried out*.

Table 6 – Quantification of total phenolics and flavonoids in *A. cepa extract*

aqueous extract *the strain*	**Total Content**	**R^2**
Total Phenolics - TP	106.54 mg EAT g^{-1}	0.9994
Total flavonoids - TF	26.36 mg EQ^{-1}	0.9845

where ; TF- total phenolics; TP- total flavonoids.

Source : Authorship (2022)

Regarding the data established in Table 6, it can be observed that for the species *A. cepa* its quantitative for total phenolics and flavonoids were significant, this argument becomes relevant when comparing the results obtained by the Masood work *et al* . (2023, p. 6), which consists of the characterization of bioactive compounds by solvent extraction (distilled water) of *A. cepa* L, in which the following values for TP and TF of 89.67 mg EAT g^{-1} were determined and 40.33 mg EAT g^{-1} respectively.

Another work that supports the present study was described by Kumar *et al*. (2022), in which the extract was used by maceration, using the solvent at higher temperatures for the species *A. cepa*, in which it was cultivated and extracted in Muan in South Korea, obtaining the following values for TP of 10.6-120.60 mg EAT g^{-1} and 30.5-415.3 mg EQ g^{-1}, respectively. In view of the indicated data, the results obtained are within the confidence interval mentioned in the present work, characterizing it with excellent levels of phenolic compounds and flavonoids.

The observed differences are justifiable when listing factors such as water availability, exposure to radiation, genotypic and phenotypic factors, however both results prove that the species *A. cepa* is also a viable alternative for antioxidant activity, since the presence of phenolics and flavonoids is associated with this property.

4 Conclusions

When predicting the drying kinetics of scaly leaf bulb of *Allium cepa* L, it was confirmed that the Verma and Midilli-Kucuk models were adequate to predict the process. Through the present study, it is possible to evaluate the presence of secondary metabolites in the aqueous extract of *A. cepa* L. The identified metabolites promote action for biologically active activities, encouraging possible applications in this context, mainly for antioxidant activity. This statement is corroborated by the presence of total phenolic and flavonoid classes, qualifying it as a promising product with an antioxidant component.

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How to quote this article

Guterres, C. V. F.; Almeida, R. de A. de.; Sousa, B. A.; Chagas, B. J. R. das.; Matos, M. A. A.; Lima, T. P. de.; Pereira, A. P. M.; Mouchrek Filho, V. E. M.; Everton, G. O. Drying study and phytochemical profile of the aqueous extract of the scaly leaf bulb of Allium cepa L (var. roxa) .*Ciência e Natura*, Santa Maria, 46, e73679. DOI 10.5902/2179460X73679. Available from: https://doi.org/10.5902/2179460X73679. Accessed in: day month abbr. year.