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Determination of moisture desorption isotherm curves of *Bidens bipinnata* L. leaves and stems

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ABSTRACT

Determining the product's moisture desorption isotherm curves is essential in order to estimate the drying time, the moisture reduction rate, and consequently estimate the dryer capacity and save time and energy consumption. Therefore, in the present study, the moisture desorption isotherm curves of *Bidens bipinnata* L. were determined at 30, 40, 50, and 60 °C and water activity ranging from 11-85 by using the gravimetric static method, and the Chung-Pfost, Halsey, Henderson, Oswin, and GAB mathematical models were used to evaluate the experimental data. According to the results, at a given water activity, the equilibrium moisture content decreased with increasing temperature, but at each temperature, the equilibrium moisture content of the samples increased with increasing water activity. Also, the Halsey model had a better fit than other models due to the high value of R^2 and the lower values of MRD, RSS, and SEE, and it can be used to estimate the equilibrium moisture content of samples at different temperatures and relative humidities.

1-Introduction

Bidens bipinnata L. belongs to the family Asteraceae and the genus Bidens. It is an annual, herbaceous, glabrous plant with green stems and typically tubular yellow flowers. The plant has a taproot system, and its flowering period occurs during July and August. This species has become naturalized and widely distributed in northern regions of Iran and the areas surrounding Tehran, where it predominantly grows as a weed in moist habitats. The plant has medicinal, culinary (as a seasoning), and forage applications. Its antidiabetic, anti-gastric ulcer, hepatoprotective, and cytotoxic effects in laboratory rats have been demonstrated. According to previous investigations, no research has yet been conducted on the drying of this plant, despite its high medicinal and export value [1]. In the analysis of biochemical, physical, and microbiological processes in food materials, knowledge of moisture adsorption and desorption isotherm data plays a crucial role. These isotherms provide valuable information regarding water interactions with food matrices. Notably, they are widely applied in the design and optimization of processes such as drying, storage, and packaging. According to previous studies, adsorption and desorption isotherms are generally used in storage processes, whereas desorption isotherms are primarily employed in drying operations [2]. Drying is recognized as one of the most economical methods for preserving food and agricultural products. In addition to extending shelf life, it inhibits enzymatic activity and microbial growth. Drying conditions, including applied temperature and processing time, significantly affect the quality of the final product. Therefore, knowledge of moisture isotherms—particularly desorption isotherms—is essential for determining drying time, moisture reduction rate, dryer capacity estimation, and optimizing time and energy consumption during drying. Equilibrium moisture content is commonly determined using either static or dynamic methods. The static method offers higher accuracy, whereas the dynamic method provides faster results. Reliable outcomes in these methods depend on maintaining constant temperature and relative humidity of the surrounding air. To achieve constant relative humidity, saturated salt solutions are used, as they generate specific and stable relative humidity values at given temperatures [3]. Moisture adsorption and desorption isotherms typically exhibit a sigmoidal shape. However, their exact form depends on the physical structure, chemical composition, and water-binding characteristics of the food material. Therefore, these isotherms must be determined individually for each product. Numerous studies have been conducted to determine adsorption and desorption isotherms for various food and agricultural products due to their importance in many processing operations [4, 5, 6 and 7]. In this context, one study determined the moisture adsorption and desorption isotherms of *Hypericum perforatum* flowers at four temperatures (40, 50, 60, and 70 °C) within a water activity range of 0.11 to 0.84 using the static gravimetric method. According to the results, the Chung–Pfost and Halsey models provided the best fit for adsorption and desorption isotherms, respectively [3]. In another study, adsorption and desorption isotherms of *Citrus reticulata* leaves were determined at 30, 40, and 50 °C within a water activity range of 0.05 to 0.90 using the static gravimetric method. The best fit for adsorption and desorption isotherms was obtained using the GAB and BET models, respectively [8]. Arabhosseini et al. (2005) determined the adsorption and desorption isotherms of stems and leaves of French and Russian varieties of tarragon at three temperatures (25, 50, and 75 °C) and relative humidity levels ranging from 0.05 to 0.90 using the static gravimetric method. Nonlinear regression analysis indicated that the Halsey and GAB models provided the best fit for adsorption and desorption isotherms [9]. In another study, adsorption and desorption isotherms of *Maytenus ilicifolia* L. leaves were determined at 30, 40, and 50 °C within a water activity range of 0.11 to 0.85 using the static gravimetric method. The Chung–Pfost and Henderson models were identified as the most suitable models for describing adsorption and desorption behavior, respectively [10]. Additionally, moisture adsorption and desorption isotherms of two medicinal plants, *Carthamus tinctorius* L. and *Artemisia dracunculus* L., were determined at 15, 25, and 35 °C within a water activity range of 8 % to 95 % using the static gravimetric method. The BET model was introduced as the most appropriate model for describing sorption behavior in these plants [11]. In this regard, the moisture desorption isotherms of the medicinal plant lavender (*Lavandula officinalis* L.) were determined at three temperatures (30, 40, and 50 °C) within a water activity range of 11 % to 85 % using the static gravimetric method in accordance with the COST90 protocol. Based on nonlinear regression analysis, the Halsey model was identified as the most appropriate model for describing the moisture desorption isotherm of this plant [12]. Gholami (2007) determined the desorption isotherms of white seedless raisins at 30, 40, 50, 60, 70, and 80 °C using the static gravimetric method following the COST90 guideline. The GAB, Smith, Oswin, Henderson, and RC-Watt models were employed to evaluate equilibrium moisture behavior. Among the tested models, the Halsey model at 30 °C and the RC-Watt model at 40–80 °C were proposed as the most suitable models for describing moisture desorption behavior [13]. In another study, the effects of seed type (hybrids 647 and 704), temperature (six levels ranging from 5 to 55 °C), and relative humidity (10–90 %) on moisture adsorption and desorption isotherms were investigated. The results indicated that seed type, temperature, and relative humidity had significant effects on equilibrium moisture content for both adsorption and desorption ($\alpha = 1\%$). Under identical environmental conditions, hybrid 704 exhibited higher equilibrium moisture content than hybrid 647, indicating greater storage stability and lower water activity in hybrid 704. Furthermore, the Oswin model provided the best fit for adsorption and desorption isotherms of hybrid 704 and for adsorption of hybrid 647, while the Chung–Pfost model showed the best fit for desorption isotherms of hybrid 647 [14]. Rahnama et al. (2011) determined the equilibrium moisture content and isosteric heat of adsorption and desorption of Estamaran date at four temperatures (40, 50, 60, and 70 °C) within a water activity range of 0.05 to 0.85. The results showed that at 40 and 50 °C, increasing temperature resulted in a decrease in equilibrium moisture content at water activities below 0.3, whereas at water activities above 0.3, equilibrium moisture content increased. However, at 60 and 70 °C, this behavior was observed at a water activity of 0.05. The GAB model was identified as the best-fitting model. Notably, the isosteric heat decreased with increasing water activity, and its value was higher for adsorption than for desorption [15]. As previously stated, determining equilibrium moisture content at different temperatures and relative humidity levels enables the construction of adsorption and desorption isotherms. Determination of moisture desorption isotherms is an essential step for defining the final moisture content during the drying process. Based on these data, dryer capacity can be accurately calculated, drying time and energy consumption can be optimized, and overall product quality can be preserved. Since the content of active compounds and the visual quality (commercial value) of medicinal plants are highly affected by drying conditions, determining appropriate drying temperature and duration is of particular importance. Therefore, in the present study, the moisture desorption isotherms of the medicinal plant *Bidens bipinnata* L. were determined at four temperatures (30, 40, 50, and 60 °C) within a water activity range of 11 % to 85 % using the static gravimetric method, and an appropriate mathematical model was proposed to describe this phenomenon.

2-Materials and Methods

Materials

The plant *Bidens bipinnata* L. used in this study was collected in July 2025 from farms and orchards surrounding Shahriar County. The salts used in this research for the preparation of saturated solutions and the establishment of the required relative humidity levels included lithium chloride (LiCl), potassium acetate (CH_3COOK), magnesium chloride ($MgCl_2$), potassium carbonate (K_2CO_3), magnesium nitrate ($Mg(NO_3)_2$), sodium chloride (NaCl), and potassium chloride (KCl). All chemicals were purchased from Merck (Germany) with a purity of 98 %.

Preparation of Experimental Samples

The static gravimetric method was employed to determine equilibrium moisture content due to its simplicity and high accuracy, in accordance with the COST 90 protocol [9]. Plant samples were harvested before the flowering stage. After transfer to the laboratory, the samples were thoroughly washed, and the leaves and stems were cut into approximately 2-mm pieces. Subsequently, approximately 200 g of plant material was weighed using a digital balance (Sartorius, model PT210, Germany) with an accuracy of ±0.001g and distributed into five previously sterilized Petri dishes. The

Petri dishes were then placed inside plastic containers previously filled with distilled water (providing an environment with more than 95 % relative humidity) and kept at room temperature (25 °C). To prevent fungal growth and mold formation, toluene and capillary tubes were used. Capillary tubes containing toluene were attached to the inner wall of the plastic containers. After five days, the samples were weighed at one-day intervals. This procedure continued until the difference between two consecutive weightings reached 0.001 g. The samples reached constant weight after 12 days and were then ready to be transferred to containers containing different saturated salt solutions.

Preparation of Saturated Salt Solutions

Seven saturated salt solutions were used, according to Table 1, to establish constant relative humidity levels. To ensure that the salt solutions remained in a saturated state, they were prepared at 70°C (10 °C higher than the maximum experimental temperature). The presence of salt crystals accumulated at the bottom of the container indicated that the solutions were saturated. Subsequently, the samples were transferred to an oven set at 60 °C, which was the highest experimental temperature. After six hours, if salt crystals were still present at the bottom of the container, the solution was considered saturated; otherwise, additional salt was added, and the above procedure was repeated. Since the experiments were conducted in duplicate, 200 mL of each saturated salt solution was poured into two glass containers and transferred to an incubator adjusted to the desired experimental temperature.

Table 1 Water activity of saturated salts solutions at different temperatures

Salt	Water activity (a_w)				
	30 °C	**40 °C**	**50 °C**	**60 °C**	**Reference**
LiCl	0.1128	0.1121	0.1110	0.1095	[16]
CH3COOK	0.2256	0.2060	0.1892	0.1747	[17]
Mg Cl2	0.3244	0.3160	0.3045	0.2926	[16]
K2CO3	0.4396	0.4330	0.4268	0.4211	[17]
Mg (NO3)2	0.5253	0.5059	0.4884	0.4725	[17]
NaCl	0.7524	0.7506	0.7473	0.7457	[17]
KCl	0.8362	0.8232	0.8120	0.8025	[16]

Determination of Equilibrium Moisture Content

To determine the equilibrium moisture content (EMC), the incubator was first adjusted to each experimental temperature, and the containers holding the saturated salt solutions were then placed inside it. Once the temperature of the solutions reached the incubator temperature, 2 g of plant sample was placed in a small aluminum dish, and the samples were suspended inside each container containing the saturated salt solutions. After five days, the first weighing was performed, and subsequent weightings were carried out at three-day intervals. When the difference between two consecutive weightings reached 0.001 g, the samples were considered to have reached moisture equilibrium. On average, this process took approximately 3 to 4 weeks. After reaching equilibrium, the samples were placed in a vacuum oven at 70 °C and 150 mbar for 8 hours to determine their final moisture content. The samples were then weighed, and the equilibrium moisture content was calculated using Equation (1) [18].

$$X_e = \frac{M_w - M_d}{M_d} \times 100$$

(1)

Where: X_e – equilibrium moisture content (%, dry basis), M_w – weight of wet sample (g), M_d – weight of dry sample (g).

Data Fitting

Since the most commonly used models for determining moisture sorption isotherms of agricultural products are the Chung–Pfost, GAB, Halsey, Henderson, and Oswin models, these models were also applied in the present study. After calculating the equilibrium moisture content at each relative humidity level, data fitting was performed using these models through nonlinear regression analysis with MATLAB software (Table 2).

Table 2 Mathematical models applied to desorption isotherms data of *Bidens bipinnata* L.

Row	Model	Model name	Reference
1	$EMC = A + Bln(-ln(ERH))$	Chung-Pfost	[9]
2	$EMC = \frac{ABC(ERH)}{(1 - C(ERH))[1 - C(ERH) + BC(ERH)]}$	Gugenheim-Anderson-de Boer (GAB)	[9]
3	$EMC = (\frac{-A}{ln(ERH)})^{\frac{1}{B}}$	Halsey	[9]
4	$EMC = (\frac{-ln(1 - ERH)}{A})^{\frac{1}{B}}$	Henderson	[9]
5	$EMC = A(\frac{ERH}{1 - ERH})^{B}$	Oswin	[9]

Where: EMC – equilibrium moisture content (dry basis); ERH – equilibrium relative humidity; A, B, and C – model constants. Furthermore, to determine the best-fitting model, four statistical criteria were employed: coefficient of determination (R^2), mean relative deviation (MRD), residual sum of squares (RSS), and standard error of estimate (SEE). The model exhibiting the highest R^2 value and the lowest values of the other statistical parameters was selected and introduced as the best model [19].

Table 3 Statistical indicators used to evaluate mathematical models

Row	Model name	Model	Reference
1	Standard Error Estimation (SEE)	$SEE = \sqrt{\frac{\sum_{i=1}^{m}(EMC - \overline{EMC})^2}{d_f}}$	[9]
2	Mean Relative Deviation (MRD)	$MRD = \frac{1}{m}\sum_{i=1}^{m}\frac{\lvert EMC - \overline{EMC}\rvert}{EMC}$	[9]
3	Residual Sum of Square (RSS)	$RSS = \sum_{i=1}^{m}(EMC - \overline{EMC})^2$	[9]
4	Degree of freedom	$d_f = m - n$	[9]

Where: EMC – experimentally determined equilibrium moisture content (dry basis); ($\overline{EMC}$)– model-predicted equilibrium moisture content (dry basis); m – number of observations for each model; n – number of model constants.

3-Results and Discussion

In general, at a constant temperature, the moisture content of a food material changes until it reaches equilibrium with the relative humidity of the surrounding air. Under these conditions, the weight of the material remains constant. The moisture content at this state is referred to as the equilibrium moisture content, and the relative humidity of the surrounding air is called the equilibrium relative humidity. The results related to the mean equilibrium moisture content of the samples and the water activity of the saturated salt solutions at each experimental temperature are presented in Table 4.

Table 4 Equilibrium moisture content of treatments and water activity of saturated salt solutions at different temperatures

Temperature (°C)	ERH	EMC	Temperature (°C)	ERH	EMC
	0.1128	5.31		0.1110	3.21
	0.2256	6.31		0.1892	5.21
	0.3244	10.73		0.3045	7.51
30	0.4396	15.52	50	0.4268	8.01
	0.5253	18.41		0.4884	10.25
	0.7524	20.52		0.7473	15.11
	0.8362	21.15		0.8120	17.60
	0.1121	4.11		0.1095	2.11
	0.2060	6.10		0.1747	4.11
	0.3160	9.54		0.2926	7.20

40	0.4330	10.30	**60**	0.4211	7.35
	0.5059	15.41		0.4725	7.51
	0.7506	18.11		0.7457	11.50
	0.8232	19.95		0.8025	12.21

According to the results, at a specific water activity corresponding to a given salt, increasing temperature led to a decrease in equilibrium moisture content. Furthermore, at each temperature, increasing water activity increased the equilibrium moisture content of the samples. This behavior is attributed to the vapor pressure difference between the material and the surrounding environment. In other words, as the relative humidity of the environment increases, the partial pressure of water vapor approaches the saturated vapor pressure, thereby reducing the vapor pressure gradient between the material and the environment [3].

Table 5 Estimated values and statistical parameters for Chung-Pfost, GAB, Halsey, Henderson and Oswin equations fitted to desorption isotherm data of *Bidens bipinnata* L. stem and leaves at four temperatures (30, 40, 50 and 60 °C)

Model	T (°C)	A	B	C	R2	MRD	RSS	SEE
Chung-Pfost	**30**	12.32	20.32	-	0.98	0.152	0.0410	0.026
	40	10.56	15.41	-	0.98	0.148	0.0252	0.051
	50	13.45	12.56	-	0.95	0.214	0.0353	0.031
	60	14.12	25.41	-	0.95	0.125	0.0215	0.015
GAB	**30**	0.48	0.54	1.42	0.96	0.257	0.0542	0.084
	40	0.56	0.68	1.52	0.98	0.314	0.0643	0.064
	50	1.52	0.45	1.76	0.95	0.361	0.0421	0.066
	60	1.89	1.23	0.98	0.95	0.289	0.0256	0.057
Halsey	**30**	-3.21	1.28	-	0.98	0.087	0.0012	0.015
	40	-3.14	1.33	-	0.98	0.069	0.0052	0.022
	50	-2.89	1.54	-	0.98	0.088	0.0041	0.016
	60	-3.43	1.30	-	0.98	0.074	0.0024	0.031
Henderson	**30**	0.047	1.41	-	0.95	0.289	0.0510	0.041
	40	0.076	1.23	-	0.95	0.356	0.0446	0.042
	50	0.054	1.52	-	0.98	0.247	0.0356	0.089
	60	0.065	1.32	-	0.98	0.189	0.0632	0.063
Oswin	**30**	0.211	1.65	-	0.98	0.099	0.0112	0.015
	40	0.125	1.84	-	0.98	0.178	0.0451	0.064
	50	0.203	1.12	-	0.95	0.164	0.0361	0.043
	60	0.124	1.87	-	0.95	0.201	0.0412	0.039

Table 5 presents the fitting results of the tested models at 30, 40, 50, and 60 °C. The Halsey model showed the best performance due to its higher R^2 value and lower MRD, RSS, and SEE values compared to the other models. Therefore, it can be used to estimate the equilibrium moisture content of the samples at different temperatures and relative humidity levels. Other researchers have also reported similar findings in their studies [3, 4, 7, 8, 9 and 12].

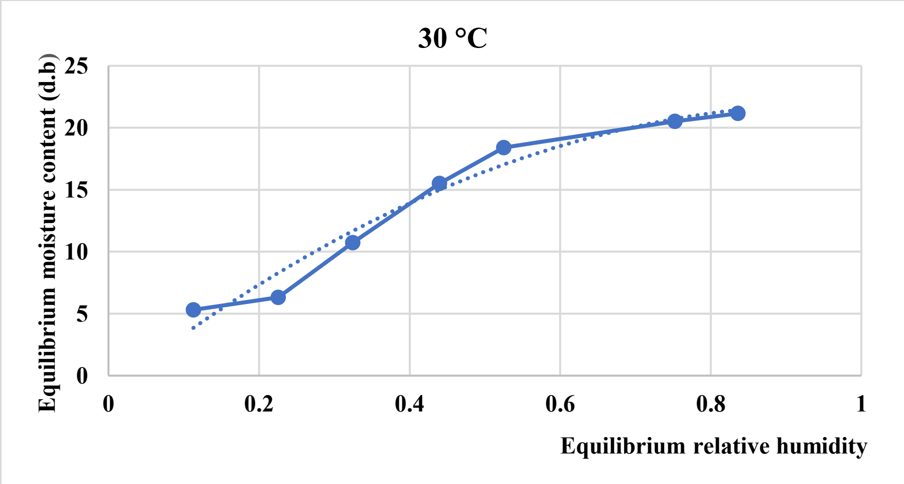

Fig 1. Desorption isotherm data of *Bidens bipinnata* L. stem and leaves at 30 °C and fitted curves of the Halsey equation

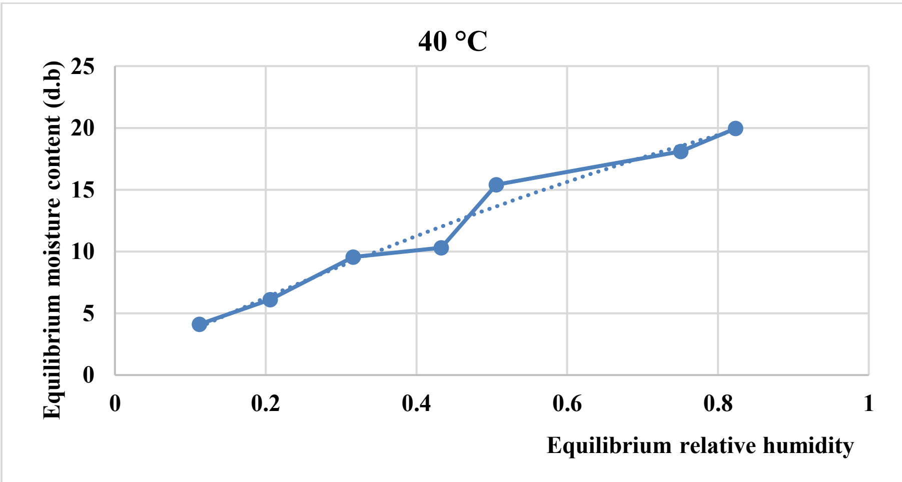

Fig 2. Desorption isotherm data of *Bidens bipinnata* L. stem and leaves at 40 °C and fitted curves of the Halsey equation

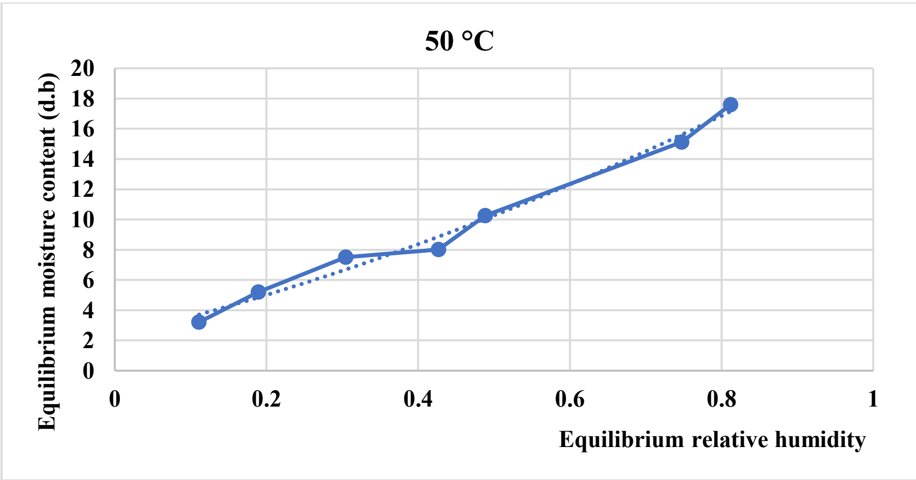

Fig 3. Desorption isotherm data of *Bidens bipinnata* L. stem and leaves at 50 °C and fitted curves of the Halsey equation

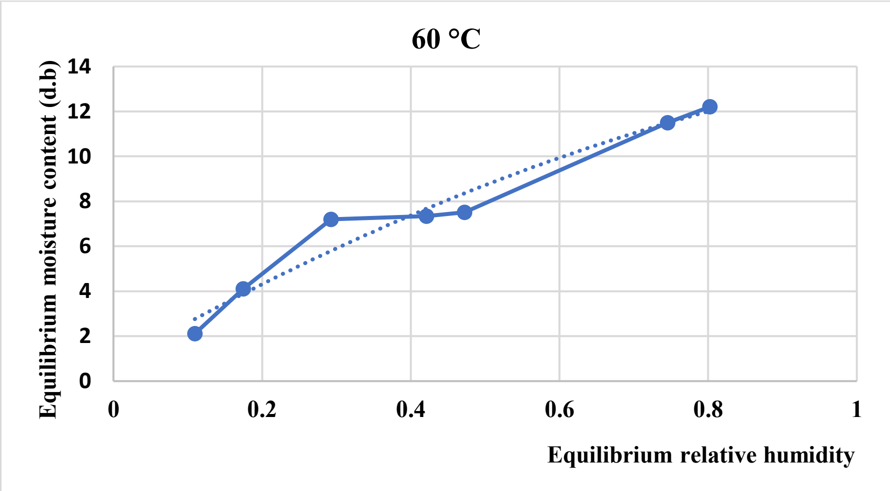

Fig 4. Desorption isotherm data of *Bidens bipinnata* L. stem and leaves at 60 °C and fitted curves of the Halsey equation

4-Conclusion

Considering the permissible moisture content for storage of dried plant materials to be within the range of 4–8 % (dry basis), the results of this study indicate that: at 30 and 40 °C, if the relative humidity exceeds 31 %, at 50 and 60 °C, if the relative humidity exceeds 42 % and 47 %, respectively, proper drying does not occur, and there is a risk of mold growth during storage. Moreover, among the evaluated models, the Halsey model provided the best fit for describing the moisture desorption isotherms and can be effectively used to estimate equilibrium moisture content at different drying temperatures.

Data Availability

All data relevant to the study are included in the article.

Conflict of 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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Authors Contributions

Leila Yousefi: Conceptualization, Investigation, Writing –original draft, Writing – review & editing.

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تعیین منحنی‌های هم دمای دفع رطوبت برگ و ساقه گیاه *Bidens bipinnata* L.

لیلا یوسفی

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چکیده

تعیین منحنی‌های هم‌دمای دفع رطوبت محصول، به منظور مشخص کردن مدت زمان خشک‌کردن، آهنگ کاهش رطوبت و در نتیجه برآورد ظرفیت خشک‌کن و صرفه‌جویی در زمان و مصرف انرژی، ضروری به نظر می‌رسد. لذا در پژوهش حاضر، منحنی‌های هم دمای دفع رطوبت *Bidens bipinnata* L. در دماهای ۳۰، ۴۰، ۵۰ و ۶۰ درجه سلسیوس و در محدوده‌ی رطوبت نسبی ۱۱ تا ۸۵ درصد به روش وزن سنجی ایستا تعیین گردید و از مدل‌های ریاضی چانگ-فاست، هالسی، هندرسون، اسوین و GAB برای برآزش داده‌های حاصل از آزمایش استفاده شد. طبق نتایج، در یک فعالیت آبی معین، با افزایش دما، مقدار رطوبت تعادلی کاهش اما در هریک از دماها، با افزایش فعالیت آبی، مقدار رطوبت تعادلی نمونه‌ها افزایش یافت. همچنین مدل هالسی به دلیل بالا بودن مقدار R^2 و کمتر بودن مقادیر *MRD*، *RSS* و *SEE* نسبت به سایر مدل‌ها از برازش مطلوب‌تری برخوردار بود و می‌توان از آن در برآورد رطوبت تعادلی نمونه‌ها در دماها و رطوبت‌های نسبی مختلف استفاده کرد.