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ORIGINAL ARTICLE

Drying Kinetics, Colour Characterization, Water Activity and Alkaloid Content of *Tinospora cordifolia* Drying

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ABSTRACT
Dried Tinospora cordifolia (Giloy) powder has an important role in Ayurvedic medicine formulations because of its immense potential to improve the immune system of the human. Both leaves and stems of Tinospora cordifolia were dried using conventional hot air drying at three temperatures (40°C, 50°C and 60°C) in a lab scale hot air dryer. Drying kinetics, effective moisture diffusivity and activation energy were determined for all the drying conditions under consideration. Color characteristics, water activity and alkaloid content of the dried products were also evaluated. A shorter drying time was observed for the samples (stems) dried at 60°C followed by 50°C and 40°C respectively. Activation energy based on Arrhenius equation was found to be ~33 kJ/mol for both leaves and stems with effective diffusivity in the range of $5.06\times10^{-9} - 11.1\times10^{-9}$ m^2/s and $8.1\times10^{-9} - 17.5\times10^{-9}$ m^2/s respectively. Five different semi-empirical models were fitted to the drying data and the most efficient model was chosen based on the statistical performance parameters (R^2 and RMSE). The Tinospora cordifolia found to follow the drying behaviour in alignment with the "Henderson and Pabis model" for stems and for leaves "Midilli and others model" showed the highest goodness of fit. The color retention was best at 40°C for stems and at 60°C for leaves. The retention of alkaloid content was best at 40°C for stems and leaves. The maximum water activities (a_w) reduction was 0.369 and 0.328 at 60°C in stem and leaves respectively followed by at 40°C (0.451 and 0.548 respectively).
Keywords: *Tinospora cordifolia (Giloy), hot air drying, mathematical modeling, color, water activity, alkaloid content*

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INTRODUCTION

Tinospora cordifolia (TC), plant of significant medicinal importance, is found in tropical Indian subcontinent and China up to an altitude of 300 m. It is known as *Rasayana* in Sanskrit language which is termed as the science of expanding lifespan [1]. TC has potential to be employed as an adjuvant therapy in chronic disease management characterized by hypertriglyceridemia, hyperinsulinemia, insulin resistance and aggravated antioxidant status [2]. TC is one of the most adaptable rejuvenating herbs due to its property of curing multiple diseases in the traditional system of medicine [3]. It is widely used in many Ayurvedic medicine formulations because of its potential to improve the resistance of the body and immune system against infections [4]. The starch obtained from the stems of TC is highly nutritive and digestive [5]. It has potential applications both in the food systems as an antioxidant and in the biological systems as a nutraceutical [6].

Generally, the freshly harvested TC is voluminous and creates trouble during handling and storage. For handling and better storage purposes, reducing the water content of freshly harvested TC is imperative. By reducing the water content, the materials becomes easier to handle and lesser prone to degradation. Due to lack of knowledge on postharvest handling/drying system (identified as key issues which need to be addressed) are major factors contributing to wastage, poor quality of TC and consequently fetching of lower income by the farmers.

Traditionally, TC is air dried in open condition under direct sunlight. It may lead to deterioration of end product by contamination and reduction in overall quality and acceptability. Inadequate work has been done on the area of postharvest practices applications, especially on dehydration techniques and their effect on the quality of TC. Under these circumstances, extensive study is required to standardize the drying practices for maintaining the overall quality of TC.

The current research study about *Tinospora cordifolia*, *for the first time*, reports the modeling approach for TC drying and thin-layer drying equations are employed for this purpose. The aim of the study was to see the impact of different hot air drying (HAD) temperatures on drying curves, water activity (a_w) and quality parameters (*color and alkaloid content*) of stems and leaves of TC .

MATERIAL AND METHODS

Sample Preparation

For the study purpose, raw materials were procured from the herbal garden and verified by the botanist for its purity. The TC stems (TCS) were cut into slab of size 1×1×0.5 cm with help of secateurs/ knife and stainless steel measuring scale. TC leaves (TCL) samples were prepared with the help of a cutter appropriate enough to cut circular pieces of leaves of 7.5 cm diameter.

Drying Experiment

The drying of TC stem (TCS)/ TC leaves (TCL) samples was conducted in convective hot air dryer. Hot air drying (HAD) of the TCS/ TCL samples was executed at three temperatures (40, 50 and 60°C). The experiments for the drying of stems and leaves of TC were conducted separately and the experiment was replicated three times for each temperature. During drying, weight of the TCS/ TCL samples was monitored and recorded in regular time interval. The drying experiment was continued until constant weight was observed. Based on scientific literature survey, it was found that information about M_e of hot air dried stems and leaves of TC is very limited at 40 to 60°C temperature. The M_e values, for the temperature range (40 to 60°C), were calculated by drying the TCS/ TCL samples at each temperature for 24 hours in hot air dryer [7].

2.3 Mathematical modeling of the drying process

The moisture ratio (MR) of the TCS/TCL samples at each M.C. was calculated by Eq. (1)

$$MR = \frac{Mt}{Mi} \qquad (1)$$

Eq. (1) can be further simplified in Eq. (2) as given below:

$$MR = \frac{M_t - M_e}{M_i - M_e} \qquad (2)$$

Where,

MR: Moisture ratio, which is dimension less; M_t: M.C. at a given time (t); M_i: Initial M.C.; M_e: Equilibrium M. C.

Five thin-layer drying models viz. Page Model, Henderson and Pabis Model, Midilli and Others Model, Logarithmic Model, and Peleg Model were employed to explicate the drying behaviour and identification of appropriate drying model for TCS and TCL. The MATLAB (version 12), based on Trust Region algorithm, was applied for non-linear regression analysis to find out the drying rate constant (k) and coefficients (a, b, c, and n)

For knowing the parameters of employed models, the goodness of fit was determined using statistical parameters such as root mean square error (RMSE) and adjusted R^2.

2.4 Determination of effective moisture diffusivity of TCS

TCS samples were prepared of size 1×1×0.5 cm and dried in the form of infinite slabs in thin layer drying. Fick's second law of diffusion was used to determine the D_{eff} (m^2/s). For infinite slab, an Eq.(3) was proposed by the Crank to determine the D_{eff} . Crank Eq.(3) is based on the statement that the present moisture in entire sample is uniformly distributed, mass transfer is unidirectional and moisture content on the surface of the sample achieves equilibrium instantly with the surrounding environment.

$$MR = \frac{8}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{(2n-1)^2} \exp\left(-\frac{(2n-1)^2 \pi^2 D_{eff} t}{4L^2}\right) \qquad (3)$$

Where, thickness of slab before drying is L (m), the number of integers in the series is (n). For slab, the Eq. (3) can be further simplified in Eq. (4) as recommended [8, 9, 10]) by taking the first term under consideration for longer diffusion time.

$$\ln(MR) = ln\left(\frac{8}{\pi^2}\right) - \left(\frac{\pi^2 D_{eff} t}{4L^2}\right) \qquad (4)$$

Determination of effective moisture diffusivity of TCL
For calculation of effective diffusivity values of TC-leaves (TCL), cylindrical geometry was considered as the sample retains this shape, in better way in comparison to samples of leaves which were cut in slabs, during the drying process. TCL samples were prepared with the help of a cutter appropriate enough to cut circular pieces of leaves of 7.5 cm diameter. For the samples having cylindrical geometry, Fick's second law is given by Guiné et al.[11] in Eq.5:

$$MR = \frac{(M-M_e)}{(M_0-M_e)} = \sum_{n=1}^{\infty} \frac{4}{b_n^2} \exp\left(\frac{-D\, b_n^2 t}{r^2}\right) \quad (5)$$

Where, b_n are the roots of Bessel function and t is the time (s). For longer drying times, the first term of the series in above equation is considered only; thus the solution of the Fick's equation becomes as shown in Eq. (6):

$$MR = \frac{4}{b_1^2} \exp\left[-D\, t \left(\frac{b_1^2}{r^2}\right)\right] \quad (6)$$

Where r is radius of the cylinder and $b_1 = 2.4048$
Effective diffusivity (D_{eff}) was determined with slope of the line plotted between ln(MR) against the time. Reliance of moisture diffusivity on the temperature of drying medium, during drying process, was determined using Arrhenius relation as given in Eq.7:

$$D_{eff} = D_o exp\left(\frac{E_a}{RT}\right) \quad (7)$$

Where, D_o: pre-exponential factor of Arrhenius equation (also known as diffusivity constant, m^2/s); T : Temperature (°K); R: Universal gas constant; E_a : Activation energy (kJ/mol). E_a can be calculated from the slope of Arrhenius plot of $ln(D_{eff})$ versus 1/T.

2.6 Color, Water Activity and Alkaloid Content Determination
The average color value and water activity of the fresh as well as dried samples of stems and leaves of TC were estimated using the Chroma meter (Model :CR-400, Konica Minolta) and Water activity meter (Meter Group Inc, AqauLab pre water activity meter, Pullman, WA).
The color changes were estimated using the Eq. (8):

$$\Delta L^* = \frac{L^* - L_i^*}{L_i^*} \quad \Delta a^* = \frac{a^* - a_i^*}{a_i^*} \quad \Delta b^* = \frac{b^* - b_i^*}{b_i^*} \quad (8)$$

Where,
a^* denotes the redness, b^* denotes the yellowness and L^* denotes the lightness of the dried samples respectively whereas a_i^* represents redness, b_i^* represents yellowness and L_i^* represents the lightness of the fresh TC samples respectively. The total color change (ΔE) was determined with the Eq. (9):

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (9)$$

Water activity (a_w) shows that how strongly water is chemically or structurally bound within the substance. It is measured by the Eq. (10):

$$a_w = \frac{p}{p_o} \quad (10)$$

Where, p = vapor pressure of water in a given sample; p_o= vapor pressure of pure water at the same temperature.
The *alkaloid content* was determined by UV-Spectrophotmeter method as suggested by Ajanal et al. [12]. The samples were reacted by bromocresol green (BCG) and formed yellow colored complex which is extracted easily with chloroform. Extracted solutions were taken absorbance for alkaloid determination. In 2 N HCl, a part of plant extract was disbursed and thereafter it is filtered. 1 ml of this solution was taken in separatory funnel and it is washed with 10 ml chloroform. The pH of this solution with the help of 0.1 N NaOH was regulated to neutral. Then, 5 ml of each (BCG and phosphate buffer) solution were put to 1 ml of this solution. The mixture was stirred and the complex extracted was fractioned with 1, 2, 3 and 4 ml chloroform by brisk shaking. The extract was taken in volumetric flask of 10 ml and diluted with chloroform. The absorbance of the complex in chloroform was measured in UV-Spectrophotometer (SHIMADZU Model-1800) at 470 nm.

RESULTS AND DISCUSSION

Drying Kinetics

TCS and TCL were dried from initial moisture content 367.29 percent and 455.5 percent on dry basis (db) to the final moisture content of 23.36, 11.78, 9.32 and 9.95, 13.68, 8.34 percent at the temperature of 40, 50, and 60°C respectively.

Drying curves of TCS and TCL are shown in fig. 1 and fig.2 respectively. It is observed from the fig.1 and fig.2 that HAD of TCS and TCL follow the typical drying trends which is very closed to falling rate drying and no evidence of constant rate drying was observed for the selected HAD temperature of 40, 50, and 60°C. The drying curves approached to steeper slopes as the temperature of drying is increased. It is an indication that rate of drying increases with increase in drying air temperature which is a well-known fact during the drying of biological materials. It is in concurrence with the past work by the researchers [13, 14, 15]. It is also evident from the fig. 1 and 2 that total drying time diminishes notably as the temperature of drying air increases which may be due to increase in vapour pressure gradient in the TCS and TCL at elevated temperature. It may persuade to higher heat transfer rates [16].

TCS and TCL dried at different hot air drying temperature were fitted to five thin layer drying models (Page model, Henderson and Pabis Model, Midilli and Others Model, Logarithmic Model, Peleg Model). The acquired constant values of the fitted models and their corresponding statistical values, adj.R^2 (coefficient of determination) and RMSE (root mean square error), were calculated and summarized in table 1 for selecting the best model and finding the goodness of fit. For best fit, the value of R^2 should be higher and closer to one; however, the value of RMSE should be lower approaching to zero [17, 18, 19, 20].

For all the drying temperatures and fitted models in TCS drying, the values of RMSE and adj.R^2 fluctuated from 0.02408 to 0.06808 and 0.9577 to 0.9961 respectively. Henderson and Pabis Model showed the highest R^2 and lowest RMSE values for TCS. For TCL drying, the values of RMSE and adj.R^2 fluctuated from 0.03143 to 0.09498 and 0.9483 to 0.9932 respectively for all the selected drying temperatures during the study. Midilli and Others Model was observed to have the lowest RMSE and highest adj.R^2 values for TCL drying at chosen temperatures for the study.

3.2 Activation Energy and Effective Moisture Diffusivity

The calculated values of E_a and D_{eff} are presented in table 2. According to Arrhenius plot as shown in fig. 3A, D_o and E_a were calculated and have the values of 2.47×10^{-3} m^2/s and 33.06 KJ/mol respectively for TCS; whereas the TCL has the calculated values of D_o and Ea, as shown is fig.3B, are 1.88×10^{-3} m^2/s and 33.75 KJ/mol respectively. It is reported that the boost in drying temperature from 40 to 60°C results in a considerable increase in D_{eff} value from 8.10×10^{-9} to 17.5×10^{-9} m^2/s and 5.06×10^{-9} to 11.1×10^{-9} m^2/s for TCS and TCL respectively. Similar results for carrots and kale have been reported by other researchers [21, 22].

Ironically the D_{eff} value for the TCL, at the drying temperature 50°C, is reported 4.81×10^{-9} m^2/s which might be due to temperature fluctuations which was observed during the experiment. The fluctuations in set drying temperature might have resulted in collapsing of the product structure thus reducing the porosity and diffusion. Similar results were reported on potato cubes drying [23].

3.3 Color, Water Activity and Alkaloid Content

Color and a_w of fresh and dried samples of TCS and TCL were calculated and presented in table 3. It is always advisable to have lower Δ E as the elevated value of Δ E is an indication of loss of color attributes of the product.

Lowest change in L^*, a^*, b^* values of TCS samples were found at 40°C HAD. The recorded L^*values of fresh as well as TCS samples dried at 40°C were 43.7 and 41.7 respectively, which is an indication of slight increase in whiteness of TCS sample. The negative a^*values of TCS samples were not taken into consideration as the effect of recorded negative a^*values in color attributes is not significant. The smallest Change in b^* values were also reported in TCS samples dried at 40°C.

The lowest change in L^*, a^* b^* values of TCL samples were found at HAD temperature of 60°C as presented in table 3. Most decisive and enviable color attribute in leaves are negative a^* values. The highest negative a^* values were reported in TCL samples dried at 60°C. The lowest ΔE was found at 40°C for TCS and at 60°C for TCL, which is a sign of better colour retention. Green color (chlorophyll) retention in leafy green vegetables was found better at faster drying conditions [24]. Similar results are also reported while drying [25].

Table 1 Values of mathematical models (constants and statistical parameters) applied in Hot air drying of *Tinospora cordifolia*-stem and leaves.

Model	Constants/ Statistical Parameters	Hot air drying temperature for Stem			Hot air drying temperature for Leaves		
		40°C	50°C	60°C	40°C	50°C	60°C
Page Model	Equation $MR = \exp(-kt^n)$						
	n	1.732	1.35	1.282	1.707	2.672	1.956
	k	3.523×10^{-5}	0.002808	0.001453	2.425×10^{-5}	8.653×10^{-8}	2.515×10^{-5}
	RMSE	0.02477	0.06808	0.03152	0.03923	0.04269	0.04208
	adj. R^2	0.9956	0.9577	0.9932	0.9888	0.9883	0.9899
Henderson and Pabis Model	Equation $MR = a\exp(-kt^n)$						
	n	1.665	1.52	1.312	1.909	2.73	1.885
	k	5.389×10^{-5}	9.202×10^{-5}	0.001223	6.419×10^{-6}	5.652×10^{-8}	3.818×10^{-5}
	a	1.025	0.9411	0.984	0.9541	0.9543	1.024
	RMSE	0.02408	0.06813	0.03341	0.03516	0.3919	0.04363
	adj. R^2	0.9961	0.9609	0.9934	0.9915	0.9912	0.9905
Midilli and Others Model	Equation $MR = a\exp(-kt) + bt$						
	a	1.117	0.9723	1.012	1.032	1.097	1.098
	b	-0.0001501	-0.0005158	-0.0001716	-0.0004861	-0.001586	-0.0003914
	k	0.00251	0.001023	0.005659	0.00091	1.195×10^{-5}	0.003657
	RMSE	0.6117	0.04456	0.03526	0.03143	0.0769	0.0904
	adj. R^2	0.975	0.9805	0.9927	0.9932	0.9659	0.9591
Logarithmic Model	Equation $M = a\exp(-kt) + c$						
	a	1.319	2.657	1.122	2.123	-1.638	1.442
	c	-0.2053	-1.687	-0.1122	-1.104	2.691	-0.345
	k	0.002169	5.559×10^{-4}	0.005106	6.802×10^{-4}	7.42×10^{-4}	0.002976
	RMSE	0.05904	0.04485	0.03313	0.0359	0.0695	0.08814
	adj. R^2	0.9767	0.9831	0.9935	0.9912	0.9722	0.9611
Peleg Model	Equation $MR = 1 - t/(a + bt)$						
	a	444.9	596.8	157	789.7	954.5	291.5
	b	0.4695	0.2762	0.6466	0.1377	-0.424	0.3876
	RMSE	0.0743	0.04412	0.0396	0.03335	0.07229	0.09498
	adj. R^2	0.9605	0.9822	0.9892	0.9919	0.9669	0.9483

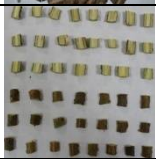
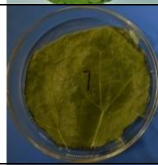
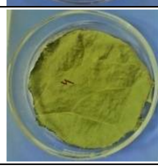
a, b, c, k, n constants; *t* time; *adj.* R^2= coefficient of determination; *RMSE*= root mean square error

Table 2 Effective moisture diffusivity and activation energy values of *Tinospora cordifolia* - Stem and leaves.

Temp. (°C)	D_{eff}, E_a of TCS		D_{eff}, E_a of TCL	
	Effective diffusivity (m^2/s)	Activation energy (KJ/mol)	Effective diffusivity (m^2/s)	Activation energy (KJ/mol)
40	8.10×10^{-9}	33.06	5.06×10^{-9}	33.75
50	9.36×10^{-9}		4.81×10^{-9}	
60	17.5×10^{-9}		11.1×10^{-9}	

D_{eff} = effective moisture diffusivity; E_a =activation energy; TCS= *Tinospora cordifolia-Stem*; TCL= *Tinospora cordifolia-Leaves*

Table 3 Color and water activity value of *Tinospora cordifolia*-stem and leaves.

Temp.		Color and water activity value of *Tinospora cordifolia*-stem						Color and water activity value of *Tinospora cordifolia*-leave				
		L^*	a^*	b^*	ΔE	a_w		L^*	a^*	b^*	ΔE	a_w
		43.71	-2.47	16.07	-	0.973		49.51	-18.31	32.30	-	0.962
40 °C		41.95	-1.66	14.61	6.38	0.451		41.84	-6.03	16.50	21.84	0.548
50 °C		40.13	-1.40	13.17	8.69	0.382		43.64	-9.48	18.80	17.63	0.413
60 °C		38.75	-1.09	11.98	10.77	0.369		44.84	-10.79	19.44	16.01	0.328

Note - Fresh Stem sample size was 1cm×1cm×0.5cm and Leaf Sample was 7.5cm in diameter

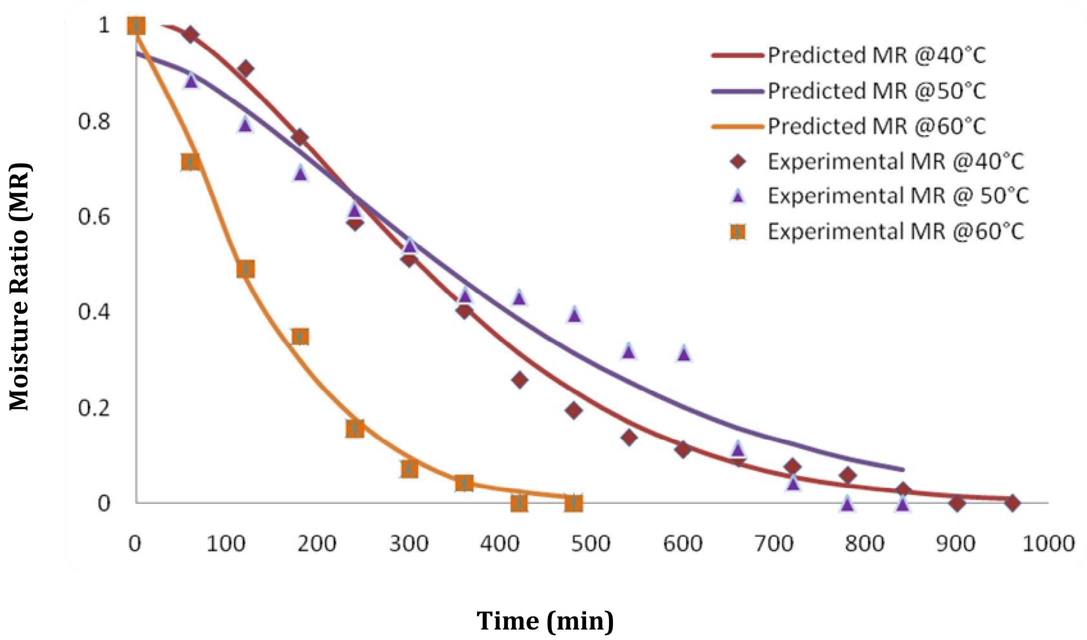


Fig. 1 Moisture ratio curves of experimental and predicted values (Henderson and Pabis Model) of Tinospora cordifolia-stem

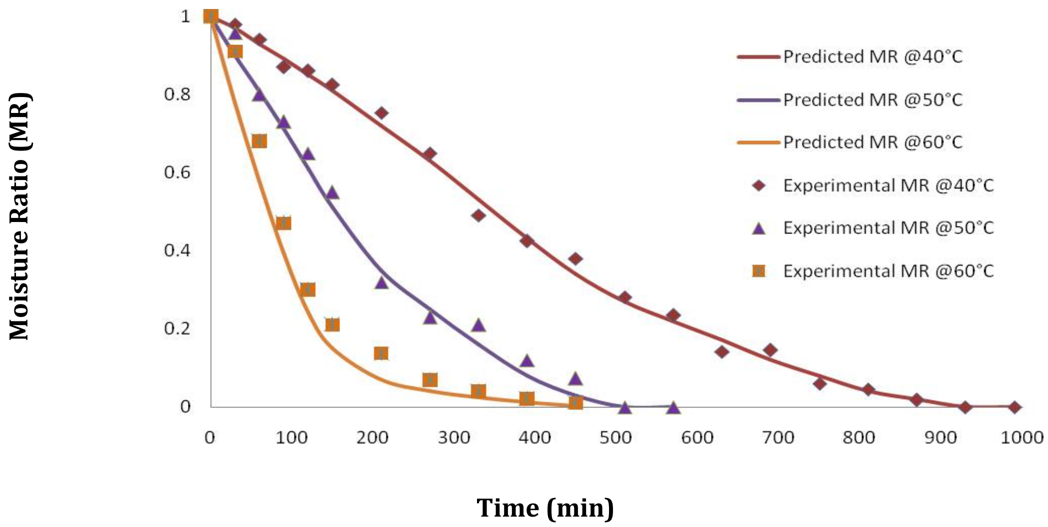


Fig. 2 Moisture ratio curves of experimental and predicted values (Midilli and Others Model) of Tinospora cordifolia-leaves

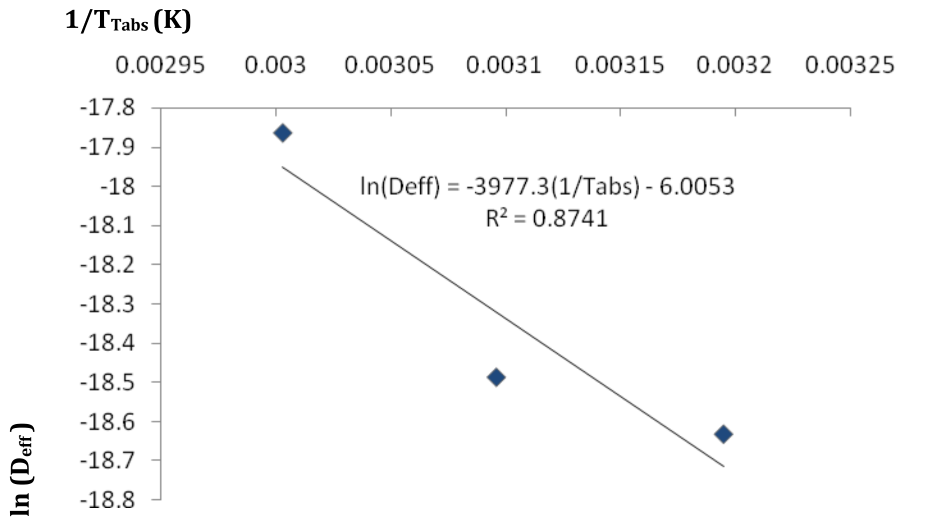


Fig. 3A Tinospora cordifolia-stem

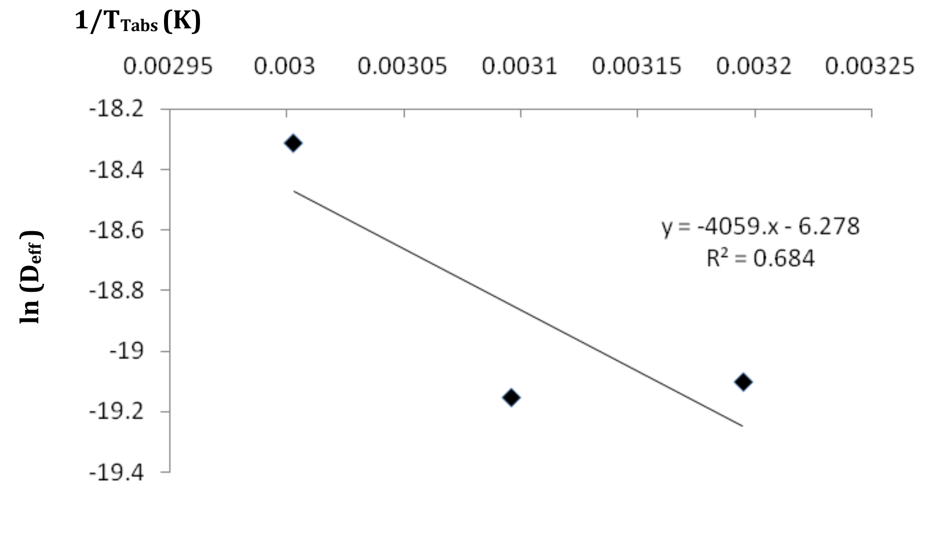


Fig. 3 Arrhenius plot relationship of effective diffusivity and reciprocal of absolute temperature of Tinospora cordifolia

The a_w values of TCS and TCL are presented in table 3. It was found that a_w of fresh samples of TCS and TCL were 0.973 and 0.962 respectively. The lowest a_w of the TCS and TCL samples were 0.369 and 0.328 respectively which were recorded for the samples dried at 60°C. It was observed that a_w of the samples reduced with an increase in temperature. Similar findings are reported while seeing the temperature's effect on a_w of food [26].

The *total alkaloid contents* were estimated by the method illustrated in section 2.6. The results show that alkaloid content was higher at 40ºC temperature in HAD for both stems and leaves of TC. At the HAD (40ºC, 50ºC and 60ºC) temperatures, the values of alkaloid content in TC stems were found to be 0.48 %, 0.44 %, and 0.39 % respectively whereas in leaves of TC, the values of alkaloid content were observed 0.37 %, 0.33 %, 0.29 % respectively at 40ºC, 50ºC and 60ºC HAD temperatures. HAD of TCS and TCL at 60ºC causes in destruction of approx. 17 %, 21 % alkaloid content in comparison to HAD at 40ºC . Higher destruction of alkaloids in leaves may be due to less thickness of leaves and more surface area exposed to hot air. The results of the study show the loss of alkaloid contents with varying temperatures and the similar observations were also recorded in drying of Giloy stems subjected to mechanical drying [27, 28]. It is recommended to employ low hot air temperature for maximum retention of alkaloid contents.

CONCLUSION

Tinospora cordifoia stem and leaves were dried in this study. Based on the drying kinetics, it was observed that as the drying air temperature increased from 40-60°C; drying time diminishes significantly for the leaves and stems of TC. Henderson-Pabis model for TC stems and for leaves Midilli and others model showed the highest goodness of fit. Activation energy was found to be ~33 kJ/mol for both leaves and stems with effective diffusivity in the range of $5.06\times10^{-9} - 11.1\times10^{-9}$ m^2/s and 8.1×10^{-9}-17.5×10^{-9} m^2/s respectively. The color retention was found the best at 40°C for stems and at 60°C for leaves**.** The water activities (a_w) reduction in dried stems and leaves from 0.973 and 0.962 respectively were maximum at higher temperature i.e. 60°C (a_w = 0.369 and 0.328), than that at 40°C (a_w =0.451 and 0.548) respectively. For highest retention of alkaloid contents in stems and leaves of TC, 40°C temperature was reported most appropriate. There are many factors like maturity of the stems and leaves, size, and harvesting period that may affect the drying rate, color, and quality parameters of TC. Based on the investigated kinetic modeling, color, water activity and alkaloid contents data of the dried leaves/ stems of TC; low cost dryers may be designed for drying of TC and other medicinal crops and these dryers may be installed directly at the farm gate to fetch better price and to improve the livelihood of the farmers engaged in cultivation of medicinal crops.

CONFLICT OF INTEREST/ 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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Abbreviations	
a_w	Water activity
E_a	Activation energy, kJ/mol
D_{eff}	Effective moisture diffusivity, m^2/s.
M.C.	Moisture content
M_e	Equilibrium moisture content
MR	Moisture ratio
M_t	M.C. at a given time (t)
M_i	Initial M.C.
D_o	Diffusivity constant, m^2/s
TC	Tinospora cordifolia
TCS	Tinospora cordifolia stems
TCL	Tinospora cordifolia leaves
HAD	Hot air drying

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