

DRYING OF POT MARIGOLD WHOLE FLOWERS AND PETALS UNDER CONTROLLED DRYING AIR TEMPERATURE AND RELATIVE HUMIDITY.

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ABSTRACT

A study was carried out to test and evaluate the drying behavior of pot marigold whole flowers and petals using a laboratory scale dryer with controlled air temperature and relative humidity.

The studied parameters included four different levels of drying air temperature (55, 60, 65 and 70°C) and three levels of air relative humidity (40, 50 and 60%). All the experimental runs were conducted at constant air velocity of (0.6 m/sec). The drying behavior of pot marigold whole flowers and petals during the drying process were simulated using three different thin layer drying models (Lewis's 1921, Henderson and Pabis's 1961 and Page's 1949 model). Final quality of the dried pot marigold whole flowers and petals was also determined. The results show that, drying rate of pot marigold whole flowers and petals increased with the increase of drying air temperature while, it was decreased with the increase of relative humidity. All studied models could describe the drying behavior of both whole flowers and petals satisfactorily. However, Page's model considered the most proper for describing the drying data in terms of higher values of (R^2) and lower values of (χ^2), (MBE), (RMSE) and (SE). In general, the drying air temperature of 70°C and relative humidity of 40% achieved the best quality in terms of total carotenoids. However, drying of pot marigold petals showed higher content of total carotenoids and shorter drying time.

INTRODUCTION

The production of medicinal and aromatic plants considered as a good source of natural income as potential exportable crops, among these plants pot marigold (*Calendula officinalis* L.). Pot marigold is an annual or biennial plant, with yellow or orange flowerheads (Paradić et al., 2013; Erhatic et al., 2014).

The main components on pot marigold flowers and petals are total carotenoids which are antioxidants, and the source of the yellow-orange coloration. The petals contain total carotenoids of 7.71% (mg/100 g dry weight) (Zaman, 2003; Bako *et al.*, 2002).

Pot marigold is grown as medicinal and aromatic plant used in food, cosmetic and pharmaceutical industry. Its dried petals are used to heal wounds and its yellow or orange color (carotenoids) is used as a coloring element in cosmetics and foods (Marisol *et al.*, 2003). Due to the great importance of this seasonal plant and in order to preserve, make it available to consumers during the whole year and to avoid quality losses, it should be exposed to specific technological treatments, such as drying (Park *et al.*, 2002).

Drying is the most common and fundamental method for preservation of medicinal and aromatic plants because it allows for quick conservation of the medicinal qualities of the plant material in an uncomplicated manner. It is a preparation process, carried out to meet the needs of the pharmaceutical industry, which does not have the suitable conditions to use fresh plants on the scale required by industry (Lorenzi and Matos, 2002).

To analyze the drying behavior of pot marigold whole flowers and petals, it is quintessential to study the drying kinetics of the plant. Thin layer drying models have found to be the widest application in crop drying because of their simplicity in use. It can also correlate the changes in moisture content of the material at any given point of time with the drying parameters (Midilli *et al.*, 2002; Togrul and Pehlivan, 2002).

The present study aims to provide a rational basis for the artificial drying of pot marigold flowers and petals, in which forced heated air under controlled temperature and relative humidity was used to remove the excess moisture content. The final qualities of the dried pot marigold whole flowers and petals were also determined.

MATERIALS AND METHODS

Freshly harvested pot marigold whole flowers were collected by hands from the experimental station of Mansoura University. The flowers of pot marigold were separated from stems and petals were also separated from receptacles to be used for the experimental work. The initial moisture content of the freshly harvested pot marigold ranged between 83.5 and 87.3 % wb for the whole flowers and between 84 and 89.5 % wb for the petals.

To achieve the objective of the present study, a controlled drying air temperature and relative humidity laboratory scale dryer developed and installed at the Agricultural Engineering Department, Faculty of Agric. Mansoura University was used. The dryer could generate any desired condition of the drying air temperature, relative humidity and velocity.

The main components of the dryer included 1.3 kW centrifugal blower with straight impeller, humidity control system in which water was spread and circulated through a humidification tower in order to provide and maintain the drying air at the desired dew point temperature by means of a thermostat with an accuracy of $\pm 0.1^\circ\text{C}$. The air temperature was controlled using air heating unit with a temperature controller for precise adjustment of the drying air temperature. The samples were accommodated in drying chamber consisted of galvanized steel cylinder (27 cm diameter and 70 cm long) and a drying tray placed inside the cylinder as shown in Fig. (1).

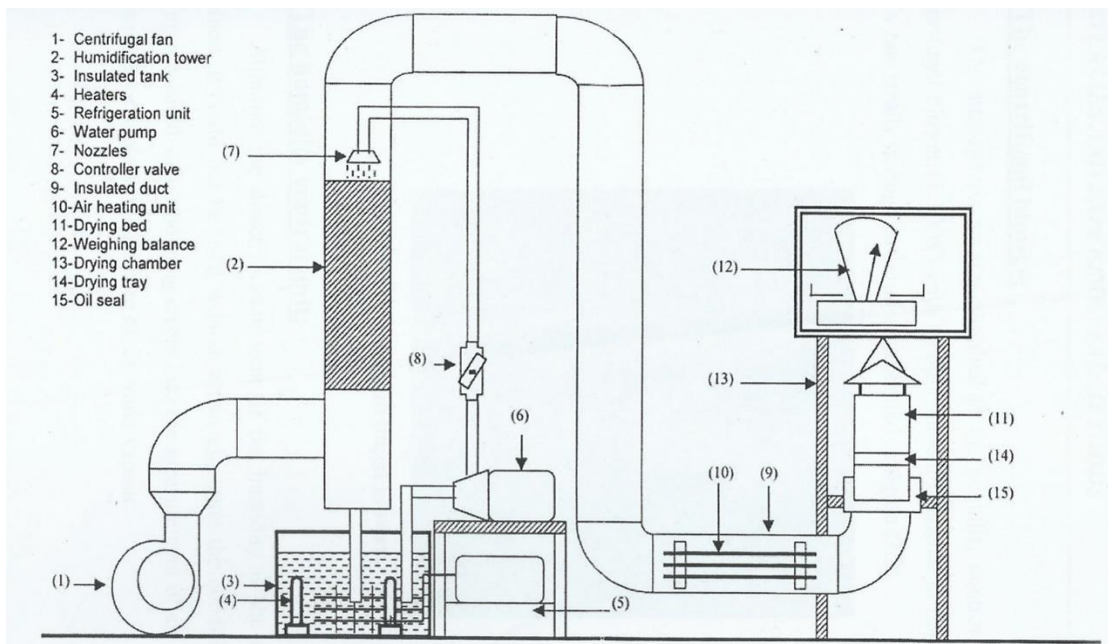


Fig. (1): Diagrammatic section of the laboratory dryer.

Experimental Measurements and Measuring Equipment.

1-Air temperature and relative humidity:

A temperature and relative humidity meter model (Trotec -2000S) connected to an Iron-Constantine thermocouple type (T) was used to measure both parameters.

2-Air velocity:

A TRI-SENSE temperature/ humidity/ air velocity meter (model Trotec 2000S) was used for measuring air velocity over the samples surface with an accuracy of 0.01 m/s.

3-Mass measurement:

The mass of samples was recorded using a digital balance with accuracy of 0.01g.

4-Moisture content of pot marigold whole flowers and petals:

Initial and final moisture contents of pot marigold whole flowers and petals were determined using a German electric oven 1.2 kW (BINDER) at temperature of 103°C for 24 hours as described by the method of AOAC (1990).

Experimental procedure:

For the pot marigold flowers drying tests, stems were cut to about 0.5 cm from the receptacles. However, for the petals drying tests, the petals were separated manually from the receptacles under careful observation. Prior to each experimental run, air temperature, relative humidity and velocity had been stabilized, the pot marigold samples were uniformly spread in thin layers of 20 g for the whole flowers and 10 g for the petals in the drying trays. At the same time three sub samples each of 5 g were taken from both fresh whole flowers and petals and kept in an aluminum tin to determine the initial moisture content, the weight changes of the samples were recorded every 5 minutes during the first two hours, every 10 minutes during the second two hours and every 20 minutes up to the end of

each drying run, or in other words until the moisture content of pot marigold whole flowers and petals had approached the equilibrium condition with the drying air. At the end of each drying run the final weight of whole flowers and petals were assessed the final moisture content was determined as explained before. In order to minimize the experimental errors of each run, it was replicated three times, and the average was considered.

Simulation of the Drying Data:

The obtained data of the laboratory experiments were employed to examine the applicability of the three studied thin layer drying models on describing and simulating the drying behaviour of both pot marigold whole flowers and petals.

The examined drying models could be presented as follows:

Lewis's model:

$$MR = \frac{M - M_f}{M_o - M_f} = \exp(-k_L t) \dots \dots \dots (1)$$

Where:

- MR: Moisture ratio, dimensionless.
- M: Instantaneous moisture content during the drying process, % (d.b).
- M_o: Initial moisture content of pot marigold whole flowers or petals samples, % (d.b).
- M_f: Final moisture content of pot marigold whole flowers or petals samples, % (d.b).
- k_L: Drying constant, min⁻¹.
- t: Drying time, min.

The values of the drying constants (k'_L) and (k''_L) of Lewis's model could be obtained from the relationship between Ln (MR) of the tested samples versus the drying times (t) as follows:

Ln MR = - k'_L t (for pot marigold whole flowers)
 Ln MR = - k''_L t (for pot marigold Petals)

The drying constants (k'_L) and (k''_L) were represented by the curves slopes of the equations.

Henderson and Pabis's model:

$$MR = A_H \cdot \exp(-k_H t) \dots \dots \dots (2)$$

Where:

k_H : Drying constant, min^{-1} .
 A_H : Drying constant, dimensionless.

The drying constants of Henderson and Pabis's equation could be obtained from the relationship between $\text{Ln}(MR)$ versus the drying time (t) as follows:

$$\text{Ln } MR = \text{Ln } A'_H - k'_H t \text{ (for pot marigold whole flowers)}$$

$$\text{Ln } MR = \text{Ln } A''_H - k''_H t \text{ (for pot marigold Petals)}$$

The drying constants (k'_H) and (k''_H) were represented by the curves slopes. While, the constants (A'_H) and (A''_H) represented by the intercepts.

Page's model:

$$MR = \exp(-k_p t^u) \dots \dots \dots (3)$$

Where:

k_p : Drying constants, min^{-1} .
 u : Drying constants, dimensionless.

The drying constants of Page's model were determined after plotting the values of $\text{Ln}(-\text{Ln}(MR))$ versus the drying time ($\text{Ln}(t)$) as follows:

$$(\text{Ln}(-\text{Ln}(MR))) = \text{Ln}(k'_p) + u' \text{Ln}(t) \text{ (for pot marigold whole flowers)}$$

$$(\text{Ln}(-\text{Ln}(MR))) = \text{Ln}(k''_p) + u'' \text{Ln}(t) \text{ (for pot marigold Petals)}$$

The drying constants (k'_p) and (k''_p) represented by curves slopes. While, the constants (u') and (u'') represented by the intercepts.

Statistical analysis:

Regression analyses were proceeded by using the Statistical routine. Correlation coefficient (r) was one of the primary criterions for selecting the most appropriate equation to define the thin layer drying curves of the dried samples. In addition to (r), the various statistical parameters such as; reduce chi-square (χ^2), mean bias error (MBE) and root mean square error (RMSE) were used to determine the quality of the fit. The best fit was decided for the highest value of (R^2) and minimum value of (χ^2), (MBE) and (RMSE) as stated by Togrul and Pehlivan (2002); Demir *et al.* (2004); Erenturk *et al.* (2004) and Goyal *et al.* (2007).

The following mathematical relationships were utilized to calculate the mentioned statistical parameters:

$$\chi^2 = \frac{\sum_{i=1}^N (MR_{obs,i} - MR_{calc,i})^2}{N - n} \dots \dots \dots (4)$$

$$MBE = \frac{1}{N} \sum_{i=1}^N (MR_{calc,i} - MR_{obs,i}) \dots \dots \dots (5)$$

$$RMSE = \left[\frac{1}{N} \sum_{i=1}^N (MR_{calc,i} - MR_{obs,i})^2 \right]^{1/2} \dots \dots \dots (6)$$

Where:

$MR_{obs,i}$: observed moisture ratio.
 $MR_{calc,i}$: calculated moisture ratio.
 N : number of observations.
 n : number of constants.(Pangavhane *et al.*, 1999).

Quality evaluation of the dried pot marigold whole flowers and petals:

The total carotenoids (mg/g) were determined according to the method of Mackinny (1941).

RESULTS AND DISCUSSION

Moisture content of pot marigold whole flowers and petals:

The changes in moisture content of both whole flowers and petals as related to drying time are illustrated in Fig. (2). It clarified that, both drying air temperature and relative humidity had a great effect on the drying behavior of pot marigold whole flowers and petals. As the drying air temperature increases and the relative humidity decreases the drying rate of both whole flowers and petals increased.

Drying analysis of pot marigold whole flowers and petals using Lewis's model:

Fig. (3) illustrates the method of determining the drying constants of Lewis's model (k'_L) for whole flowers and (k''_L) for petals, respectively. While, Table (1) presented the obtained data of constants (k'_L) and (k''_L) at different levels of drying air temperature and relative humidity for both whole flowers and petals, respectively.

As shown in Table (1), both drying constants (k'_L) and (k''_L) increased with the increase of drying air temperature, while they were decreased with the increase of drying air relative humidity.

A multiple regression analysis were proceeded to relate the drying air temperature (T_a) and relative humidity (RH) with both drying constants (k'_L) and (k''_L). The nature of dependence could be expressed by the following equations:

$$k'_L = 0.001013 T_a - 0.000125 RH - 0.03842 \dots \dots \dots (7)$$

(S.E. = 0.00059 $R^2 = 0.99226$ $r = 0.99612$)

$$k''_L = 0.00176 T_a - 0.00028 RH - 0.07242 \dots \dots \dots (8)$$

(S.E. = 0.00257 $R^2 = 0.95352$ $r = 0.97648$)

Drying analysis of thin layer drying of pot marigold whole flowers and petals using Henderson and Pabis's model:

Fig. (4) illustrates the method of determining Henderson and Pabis's drying constants. While, Table (2) presents the obtained data under different levels of drying air temperature and relative humidity for both pot marigold whole flowers and petals.

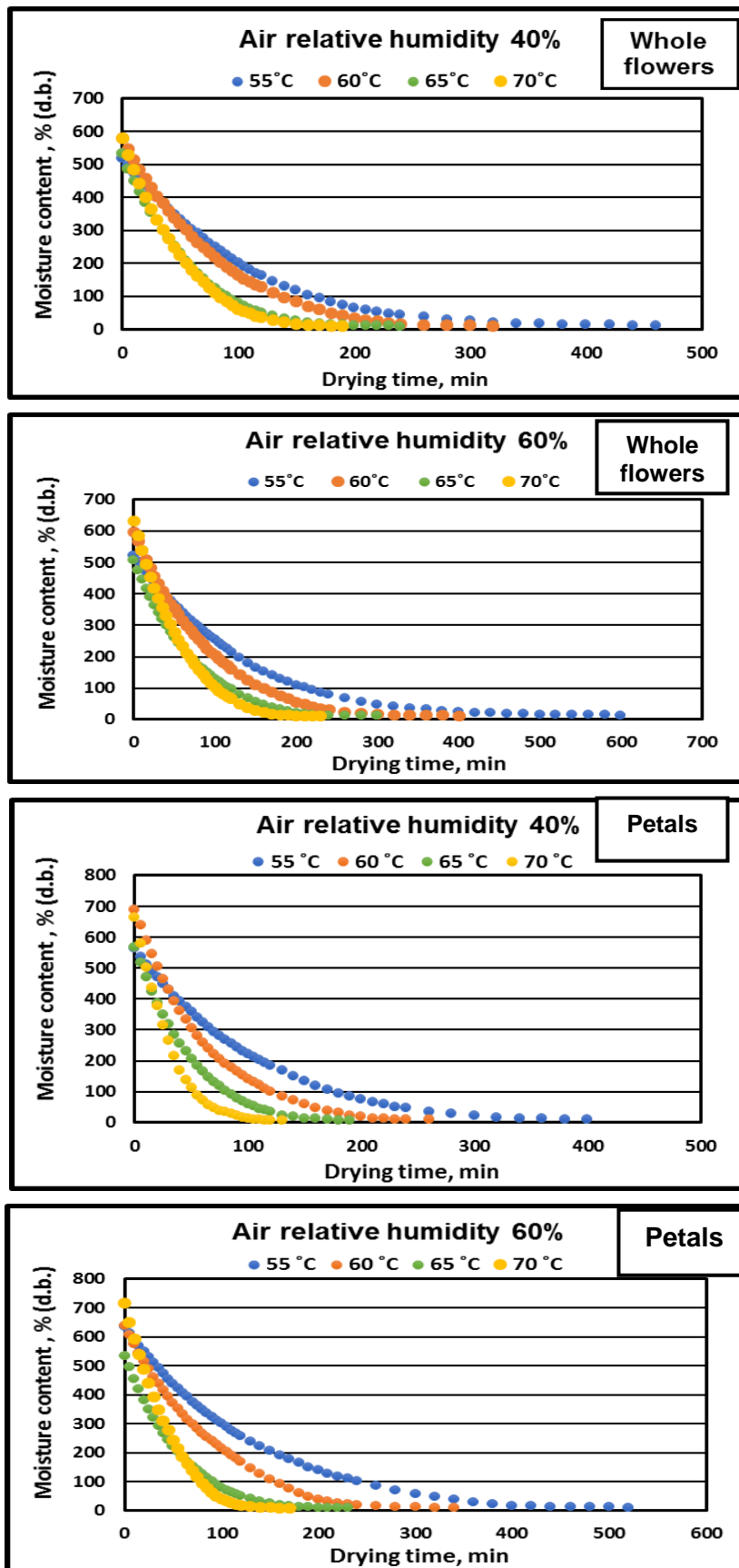


Fig. (2): Changes in moisture content of pot marigold whole flowers and petals as related to drying time at different levels of drying air temperature and relative humidity of 40 and 60%.

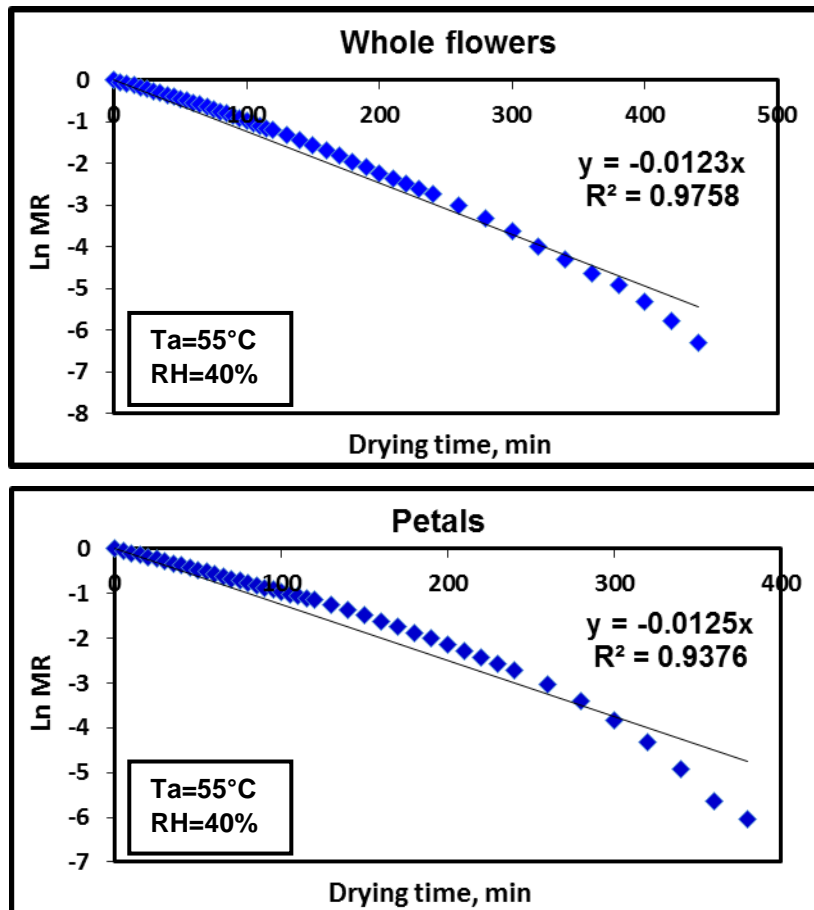


Fig. (3): Determination of Lewis's model drying constants (k'_L) and (k''_L) for pot marigold whole flowers and petals.

Table (1): Values of Lewis's model drying constants (k'_L) and (k''_L) for pot marigold whole flowers and petals.

Air temp., °C	Air relative humidity, %					
	40		50		60	
	k'_L	k''_L	k'_L	k''_L	k'_L	k''_L
55	0.012	0.013	0.011	0.012	0.010	0.011
60	0.017	0.021	0.016	0.018	0.015	0.016
65	0.024	0.028	0.021	0.026	0.020	0.025
70	0.027	0.045	0.026	0.038	0.025	0.033

As shown in Table (2), both drying constants (k'_H) and (k''_H) increased with the increase of drying air temperature, while they were decreased with the increase of drying air relative humidity. However, the drying constants (A'_H) and (A''_H) increased with the increase of both drying air temperature and relative humidity.

A multiple regression analysis was proceeded to relate the drying air temperature (T_a) and relative humidity (RH) with drying constants (k'_H), (k''_H), (A'_H) and (A''_H). The nature of dependence could be expressed by the following equations:

$$k'_H = 0.00117 T_a - 0.000125 RH - 0.04492 \dots\dots (9)$$

(S.E. = 0.00061 $R^2 = 0.99367$ $r = 0.99683$)

$$k''_H = 0.00211 T_a - 0.0003 RH - 0.08917 \dots\dots (10)$$

(S.E. = 0.00272 $R^2 = 0.96299$ $r = 0.98132$)

$$A'_H = 0.02163 T_a + 0.007585 RH - 0.12223 \dots (11)$$

(S.E. = 0.05469 $R^2 = 0.89164$ $r = 0.94427$)

$$A''_H = 0.01927 T_a + 0.00649 RH - 0.13333 \dots\dots (12)$$

(S.E. = 0.02552 $R^2 = 0.96723$ $r = 0.98348$)

Drying analysis of thin layer drying of pot marigold whole flowers and petals using Page's model:

Fig. (5) illustrates the method of determining Page's model drying constants. While, Table (3) presents the obtained data at different levels of air temperature and relative humidity for both pot marigold whole flowers and petals.

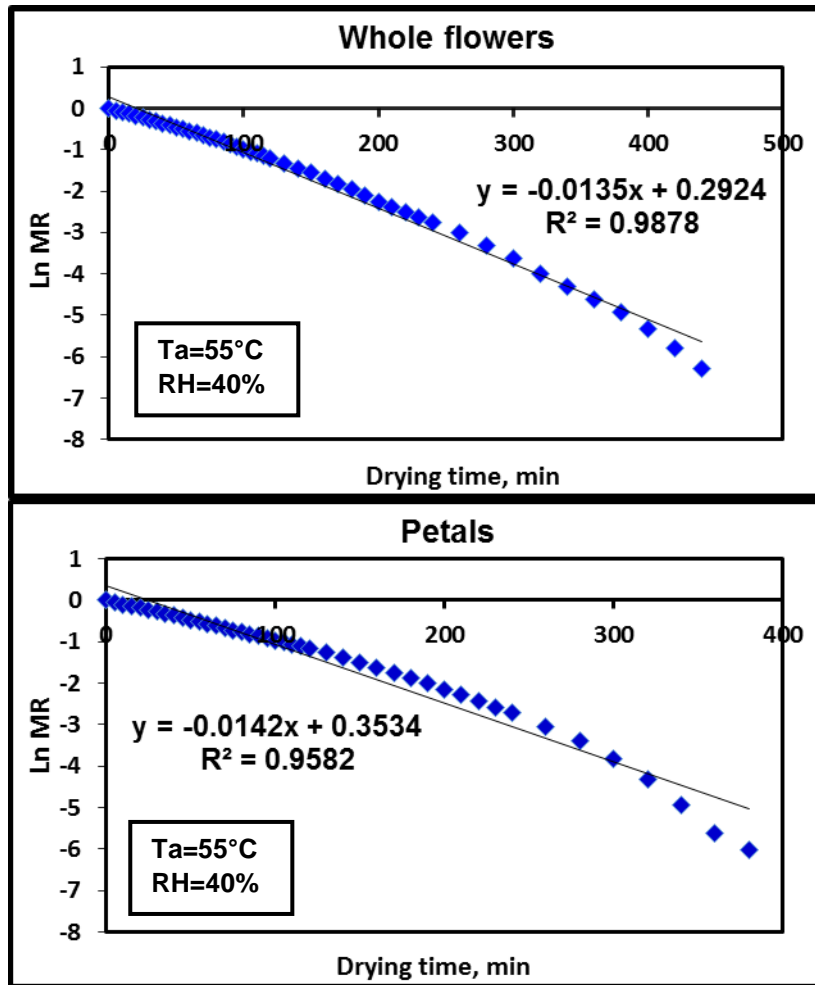


Fig. (4): Determination of Henderson and Pabis's model drying constants (k'_H), (A'_H), (k''_H) and (A''_H) for pot marigold Whole flowers and petals.

Table (2): Values of Henderson and Pabis's model drying constants (k'_H), (A'_H), (k''_H) and (A''_H) for pot marigold whole flowers and petals.

Air temp., °C	Air relative humidity, %											
	40				50				60			
	k'_H	A'_H	k''_H	A''_H	k'_H	A'_H	k''_H	A''_H	k'_H	A'_H	k''_H	A''_H
55	0.014	1.3397	0.014	1.4239	0.013	1.4261	0.013	1.5354	0.011	1.4535	0.012	1.5569
60	0.020	1.5461	0.024	1.5700	0.019	1.6090	0.021	1.6218	0.018	1.6329	0.019	1.6846
65	0.027	1.5920	0.033	1.6706	0.025	1.7228	0.030	1.7435	0.024	1.7926	0.029	1.7651
70	0.031	1.6182	0.052	1.7047	0.030	1.7525	0.044	1.7923	0.029	1.8238	0.039	1.8820

As shown in Table (3), both drying constants (k'_p) and (k''_p) increased with the increase of drying air temperature, while they were decreased with the increase of air relative humidity. However, the drying constants (u') and (u'') increased with the increase of both drying air temperature and relative humidity.

A multiple regression analysis was proceeded to relate the drying air temperature (T_a) and relative humidity (RH) with both drying constants (k'_p), (k''_p), (u') and (u''). The nature of dependence could be expressed by the following equations:

$$k'_p = 0.00038 (T_a) - 0.00011 (RH) + 0.00991 \dots (13)$$

(S.E. = 0.00049 $R^2 = 0.96647$ $r = 0.98309$)

$$k''_p = 0.00044 (T_a) - 0.00018 (RH) + 0.00907 \dots (14)$$

$$u' = 0.003299 T_a + 0.000355 RH + 0.91588 \dots (15)$$

(S.E. = 0.00572 $R^2 = 0.93416$ $r = 0.96652$)

$$u'' = 0.00862 T_a + 0.00105 RH + 0.56714 \dots (16)$$

(S.E. = 0.013098 $R^2 = 0.94901$ $r = 0.97417$)

The applicability of the studied models in simulating the laboratory drying data:

Figs. (6) and (7) illustrate the observed and calculated values of moisture content of pot marigold whole flowers and petals at 55°C drying air temperature and 40% air relative humidity. The results show that, all studied models described the drying behavior of both whole flowers and petals satisfactorily as indicated by the high values of (R^2) and low values of (SE).

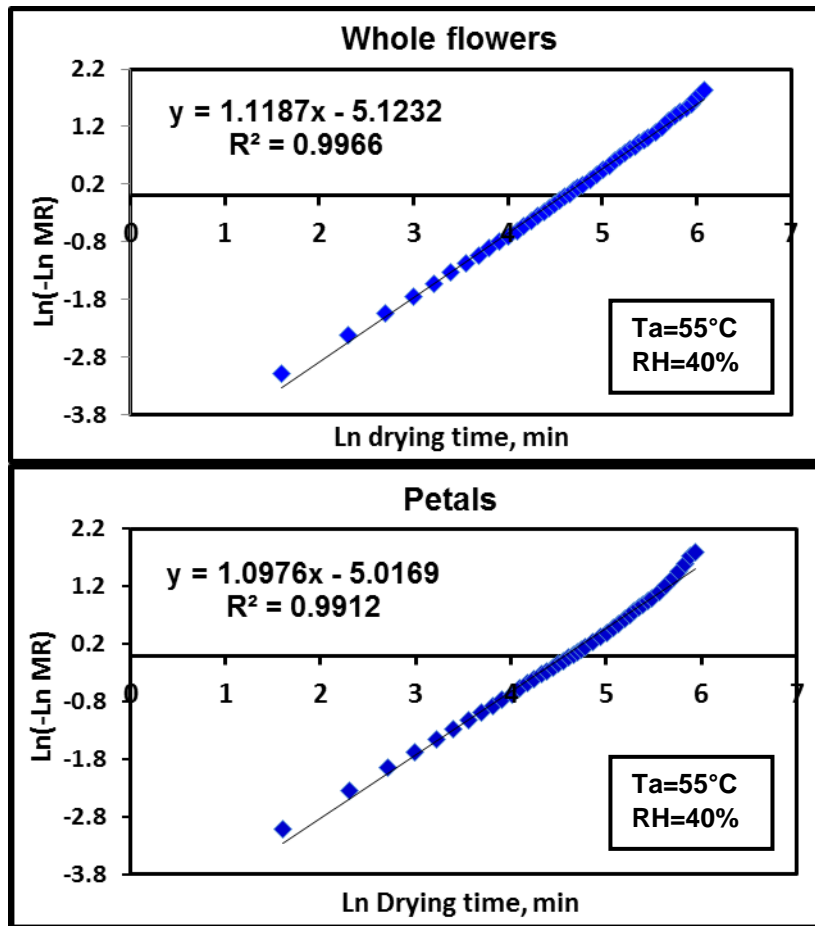


Fig. (5): Determination of the drying constants (k'_p), (u'), (k''_p) and (u'') of Page's model for pot marigold whole flowers and petals.

Table (3): Values of Page's model drying constants (k'_p), (u'), (k''_p) and (u'') for pot marigold whole flowers and petals.

Air temp., °C	Air relative humidity, %											
	40				50				60			
	k'_p	u'	k''_p	u''	k'_p	u'	k''_p	u''	k'_p	u'	k''_p	u''
55	0.0060	1.1187	0.0066	1.0976	0.0052	1.1203	0.0058	1.1003	0.0046	1.1211	0.0049	1.1119
60	0.0081	1.1216	0.0111	1.1055	0.0072	1.1269	0.0080	1.1307	0.0068	1.1301	0.0066	1.1475
65	0.0111	1.1401	0.0116	1.1661	0.0090	1.1426	0.0102	1.1684	0.0082	1.1488	0.0098	1.1729
70	0.0125	1.1646	0.0160	1.2218	0.0105	1.1694	0.0116	1.2349	0.0095	1.1734	0.0097	1.2428

Comparative evaluation of the studied drying models:

In general, all the studied models could describe the drying behavior of both pot marigold whole flowers and petals as indicated from the high values of (R^2). However, to assess the most proper model for describing the drying behavior of pot marigold whole flowers and petals and in addition to the high values of (R^2), various statistical parameters such as (r), (χ^2), (MBE) and (RMSE) were calculated. The results show that, Page's model recorded the highest value of (r) and the lowest values (χ^2), (MBE), (RMSE). This means that, Page's model is the most proper model for describing the drying behavior of both pot marigold whole flowers and petals under the studied ranges of drying air temperature and relative humidity.

Quality of pot marigold whole flowers and petals:

Total Carotenoids content:

Table (4) illustrates the changes in total carotenoids content of both pot marigold flowers and petals at different levels of drying air temperature and relative humidity. The results show that the total carotenoids content declined with the increase of drying time. As shown in Table (4), pot marigold dried petals recorded higher content of total carotenoids in comparison with the whole dried flowers due to shorter drying time.

In general, the quality analysis for both whole flowers and petals showed that the optimum conditions for keeping the highest level of total carotenoids content (12.50 mg/100g) in the whole flowers and (12.65 mg/100g) in the petals, are 70°C air temperature and 40% (RH).

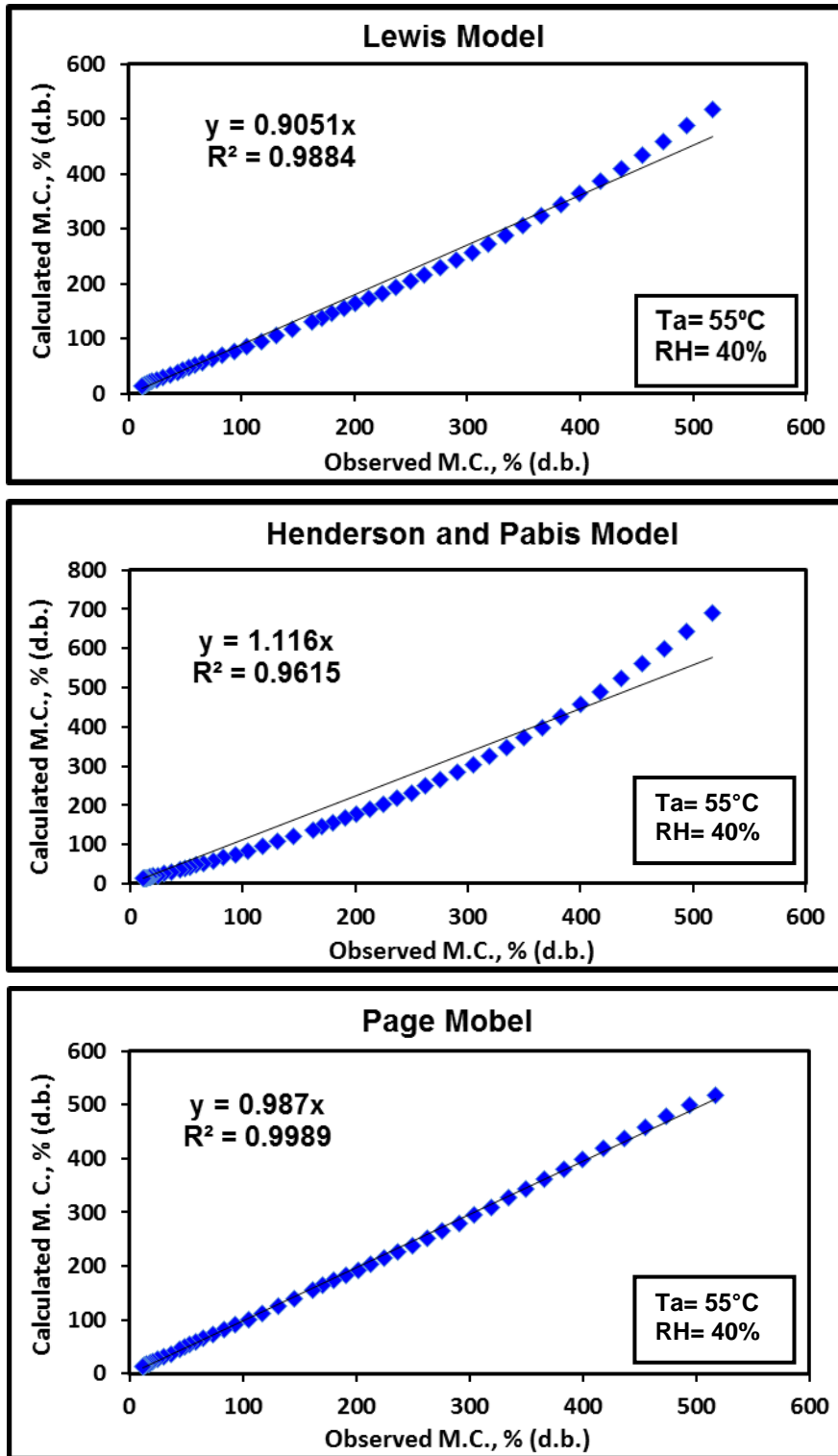


Fig. (6): The observed and calculated moisture content values of pot marigold whole flowers using all studied models.

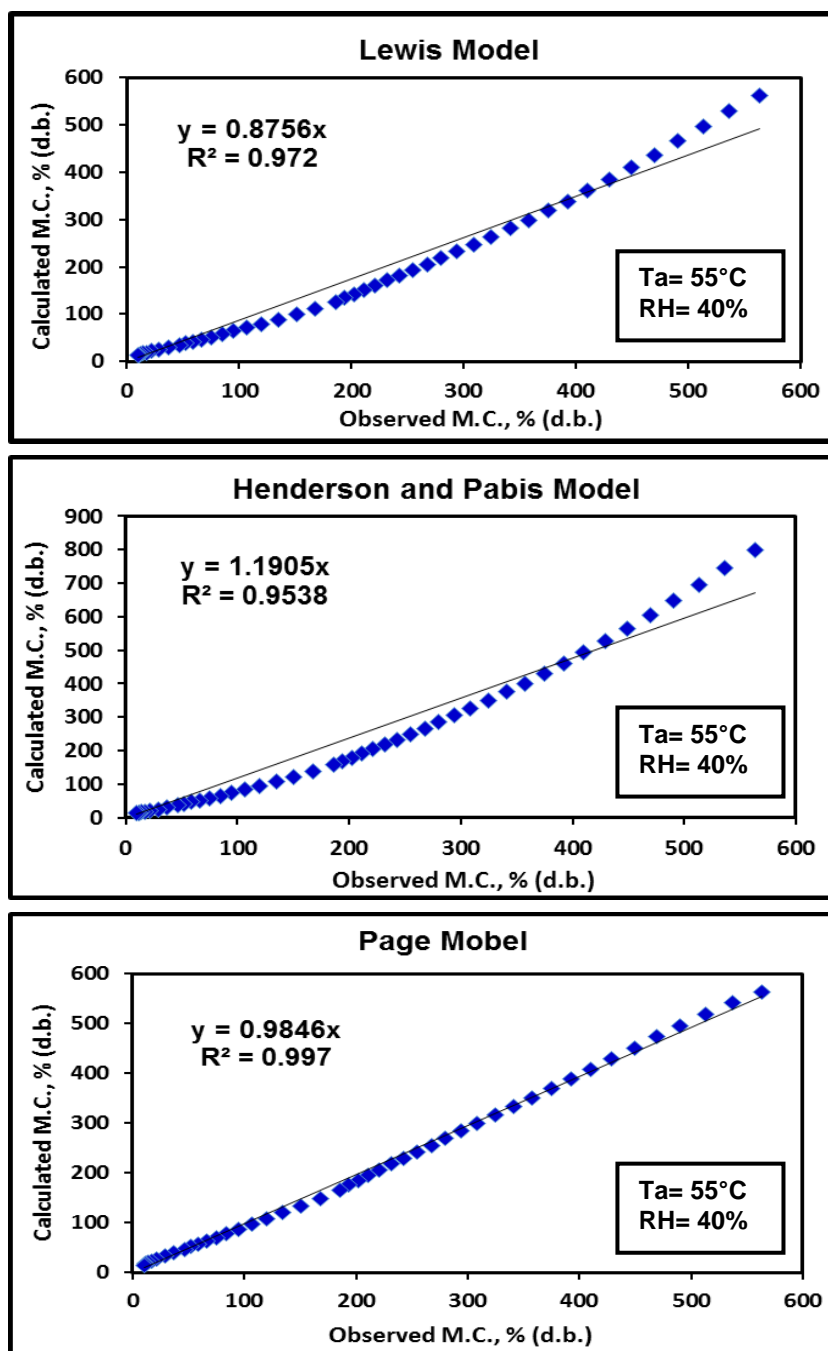


Fig. (7): The observed and calculated moisture content values of pot marigold petals using all studied models.

Table (4): Total carotenoids content of pot marigold whole flowers and petals as influenced by the drying parameters

Treatments	Fresh Samples	Dried flowers	Dried petals
	total carotenoids (mg/100 g)	total carotenoids (mg/100 g)	Total carotenoids (mg/100 g)
55°C / 40%	4.84	12.15	12.2
55°C / 50%	4.92	12.2	12.25
55°C / 60%	4.82	12	12.2
60°C / 40%	4.84	12.2	12.3
60°C / 50%	4.78	12.05	12.1
60°C / 60%	4.9	12.15	12.2
65°C / 40%	4.78	11.95	12.1
65°C / 50%	4.84	12.25	12.3
65°C / 60%	4.86	12.30	12.35
70°C / 40%	4.88	12.50	12.65
70°C / 50%	4.86	12.40	12.50
70°C / 60%	4.82	12.35	12.40

CONCLUSIONS

- 1- The moisture ratio of both pot marigold whole flowers and petals increased with the increase of drying air temperature. While, it was decreased with the increase of drying air relative humidity.
- 2- All studied models (Lewis's, Henderson and Pabis's and Page's model) could describe the drying behavior of pot marigold whole flowers and petals satisfactory however Page's model considered the most precise model for describing the drying behavior of pot marigold whole flowers and petals.
- 3- The optimum conditions keeping the final quality for both whole flowers and petals are 70°C air temperature and 40% (RH).

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تجفيف أزهار الأقحوان الكاملة والبتلات تحت ظروف التحكم في درجة الحرارة والرطوبة النسبية لهواء التجفيف.

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يهدف البحث إلى دراسة تأثير كل من درجة حرارة هواء التجفيف ورطوبة النسبية على خصائص تجفيف أزهار الأقحوان الكاملة والبتلات واختبار ومقارنة ثلاث نماذج رياضية مختلفة لوصف عملية التجفيف في طبقات رقيقة وتحديد النموذج الأمثل لوصف منحنيات التجفيف واختبار إمكانية تطبيقه للتنبؤ بالتغير في المحتوى الرطوبي لأزهار الأقحوان الكاملة والبتلات أثناء عملية التجفيف ثم إجراء اختبارات الجودة لأزهار الأقحوان الكاملة والبتلات المجففة لتحديد التغيرات التي طرأت عليها أثناء عملية التجفيف.

ولتحقيق أهداف الدراسة تم استخدام مجفف معمل يمكنه التحكم في درجة حرارة هواء التجفيف ورطوبة النسبية وتم إجراء التجارب المعملية لأزهار الأقحوان الكاملة والبتلات عند أربعة مستويات من درجة حرارة هواء التجفيف (50، 60، 70، 80) وثلاثة مستويات مختلفة لرطوبة الهواء النسبية (40، 50، 60) % كما تم إجراء جميع التجارب المعملية عند سرعة هواء ثابتة (0.6 م/ث).

وكانت أهم النتائج المتحصل عليها كالآتي:

1. زاد معدل التناقص في المحتوى الرطوبي لأزهار الأقحوان الكاملة والبتلات أثناء عملية التجفيف بزيادة درجة الحرارة بينما إنخفض بإخفاض الرطوبة النسبية للهواء.
2. تمكنت جميع النماذج الرياضية المختبرة من وصف سلوك التجفيف لكل من أزهار الأقحوان الكاملة والبتلات بصورة مرضية. ووجد أن معادلة Page هي النموذج الأمثل للتنبؤ بسلوك التجفيف مقارنة بالنماذج الأخرى حيث تمكنت من وصف منحنيات التجفيف والتنبؤ بالمحتوى الرطوبي بصورة أكثر.
3. المعاملات المثلى التي حافظت على أعلى محتوى من صبغة الكاروتين لكلاً من أزهار الأقحوان الكاملة والبتلات هي درجة حرارة هواء التجفيف 70°م ورطوبة نسبية 40%، ومع ذلك، يفضل تجفيف البتلات المنفصلة حيث أنها سجلت أعلى محتوى من صبغة الكاروتين مع زمن أقل لعملية التجفيف.