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Convective drying of atmospheric pressure cold plasma pretreatment saffron stigmas: kinetic modeling

Seyed Abolfazl Tabibian[a], Mohsen Labbafi[a,*], Gholamreza Askari[b,*], Hamidreza Ghomi[c], Alireza Rezaeinezhad[c]

[a] *Department of Food Science and Technology, Faculty of Agricultural Engineering and Technology, University College of Agriculture and Natural Resources, University of Tehran, Karaj, Iran*
[b] *Transfer Phenomena Laboratory, Department of Food Science and Engineering, College of Agriculture and Natural Resources, University of Tehran, Karaj, Iran*
[c] *Laser and Plasma Research Institute, Shahid Beheshti University, Tehran, Iran*

ABSTRACT

In this study, the drying kinetics of saffron stigmas pretreated by atmospheric pressure cold plasma pretreatment (15, 30, 45 and 60 s) followed by hot air drying (60°C and 1.5 m/s) were modeled using 10 conventional mathematical thin layer models. The use of cold plasma pretreatment reduced drying time and enhanced effective moisture diffusivity (D_{eff}). The most accurate models describing behavior of drying process of stigmas were Two-term, Midilli and Kucuk and Wang–Singh models. These models were determined based on the higher value of coefficient of determination (R^2) and the lower values of root mean square error (RMSE), chi-square (χ^2) and sum of square errors (SSE). In addition, the Newton model did not in accordance with experimental data. The value of D_{eff} of pre-treated saffron stigmas were in the range of 8.7013×10^{-9} and 9.1139×10^{-9} m^2/s depending on the time of pretreatment. The use of cold plasma pretreatment reduced the surface resistance to moisture transfer, improved the diffusion coefficient, and subsequently, reduced the drying time.

Keywords: Saffron stigma; Cold plasma; Drying; Diffusion coefficient; Mathematical modeling

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1. Introduction

Saffron is a dried product of red stigma of *Crocus Sativus Linnaeus* that is cultivated in various countries such as Iran, Spain, Greece, Turkey, and Morocco (Shahi et al., 2016). Iran is considered as the largest producer with more than 90% of world production and also major exporter in the world (Melnyk et al., 2010). Saffron is widely used in the food industry as colorant and flavoring agent and also is applied as anti-cancer and anti-depressant in traditional medicine. Moreover, saffron has miscellaneous application in industries such as cosmetics and textiles (Rodriguez-Ruiz et al., 2016). In order to preserve saffron for longer period, fresh saffron stigma should be dried. Several studies have shown that drying process of saffron stigma is one of the key stages of saffron post-harvesting and processing. Different methods have been proposed for the drying process of the saffron stigmas including shade or sun drying (traditional methods), hot-air drying, freeze drying, microwave drying, and infrared drying (Yao et al., 2019).

Drying, is a complex phenomenon in which mass and heat transfer occur simultaneously. It has a significant impact on the quality and value of the final product (Aghaei et al., 2019). Hot air drying (HAD) is one of the most conventional methods for drying of agricultural products. The main drawbacks of this approach are the low energy efficiency and the long drying time (Szadzińska et al., 2016). In recent years, some physical (thermal and non-thermal) and chemical (aqueous and gaseous) pretreatments have been developed to increase the quality and decrease the time of drying (Deng et al., 2019). Consequently, the high demand for high-quality product has led to the combination of drying methods with non-thermal energy sources.

Cold plasma is one of the emerging technologies that is widely used in several industrial and scientific fields including food

*Corresponding authors.
E-mail address: mlabbafi@ut.ac.ir (M. Labbafi);
iraskari@ut.ac.ir (Gh. Askari).
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industry, medicine, chemistry, and biology (Pankaj & Keener, 2017). The term "plasma" refers to a gas, e.g., argon, oxygen, nitrogen, and helium, or a mixture of gases that is partially or wholly ionized and consists of photons, ions, and free radicals, as well as atoms in basic or excited states (Hou et al., 2019). According to the relative energy levels of electrons and heavy species, plasma is classified into two categories; a) thermal plasma, and b) non-thermal plasma (Ekezie et al., 2017). Non-thermal plasma is conventionally made using strong electric field at ambient temperature and atmospheric pressure (Zhang et al., 2019). The gliding arc discharge (GAD) system has been considered as a straightforward and low-cost method for producing both non-thermal and thermal plasma. In this system, air flows through two metal electrodes, which are connected to a high voltage source. The potential difference between two electrodes results in electric discharge and the air stream causes the flame formation of the plasma (Kong et al., 2018). Cold atmospheric plasma is considered as a novel pretreatment before drying of agricultural products. It has shown that GAD pretreatment increase the drying rate and reduce the drying time (Zhang et al., 2019; Tabibian et al., 2020).

Development of mathematical models for drying processes is of paramount importance in drying technology, particularly in industrial processes. The main objective of the mathematical modeling is to define an accurate drying method to predict the best-operating conditions for obtaining target product (Mazandarani et al., 2017). Numerous studies have been conducted with the focus on the mathematical modeling and kinetics of the drying process of different agricultural products using different pretreatments such as citric acid and blanching, alkaline ethyl oleate solution, ultrasound, and osmotic dehydration.

Most of these studies have focused on the application of cold plasma in the food industry for the purpose of microbial decontamination and considering its effects on the quality and chemical composition and physical properties of foods (Bourke et al., 2018). Nevertheless, no investigation is conducted to the modeling of the drying process of atmospheric cold plasma pretreatment. Hence, the purpose of this study is to examine the mathematical models of thin-layer drying to describe the effect of different cold atmospheric plasma pretreatment times on drying kinetics and define optimum models to describe the drying process.

2. Material and Methods

2.1. Raw material

Saffron flowers were harvested in November 2018 from the Ahmadabad Mostofi, Tehran, Iran. Stigmas were hand separated; they were maintained in cold conditions before conducting pretreatment and main drying processes. The moisture content of fresh stigmas was 79.35 ± 0.70% on a wet basis.

2.2. Cold plasma pretreatment and hot-air drying

The cold plasma pretreatment was conducted using GAD plasma machine (Plasmatech-15B, Kavosh Yaran Fan Pooya, Iran). Briefly, 2 g of the fresh stigma with approximately 0.3 mm thickness were exposed directly to plasma flame (With a length of 11cm and width a of 4 cm) for exposure time of 0, 15, 30, 45 and 60s (Fig. 1). The power supply, electrical potential and frequency were 1 kW, 8 kV and 50 Hz, respectively. During the pretreatment process, the weight of the stigma decreased due to surface moisture evaporation. Therefore, the moisture content of all pretreated stigmas were adjusted to the same level (43.37%) using HAD at 60°C and flow velocity of 1.5 m/s before the main drying process. HAD is done using laboratory scale hot-air dryer (SAT-1373, Iran) with the air temperature of 60°C and air velocity of 1.5 m/s. In order to investigate the kinetics of drying, samples were weighted every 4 minutes using a digital scale. The drying process has been continued until the moisture content of the samples reduced to 12% (wb). In order to increase the accuracy of the results, experiments were performed in triplicate.

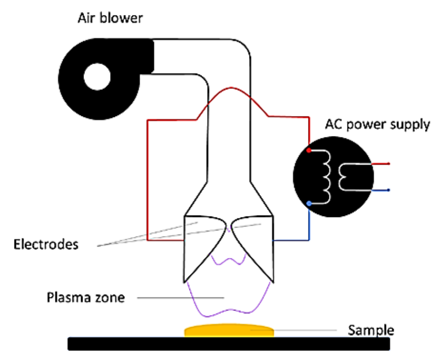

Fig. 1. Schematic diagram of gliding arc discharge setup.

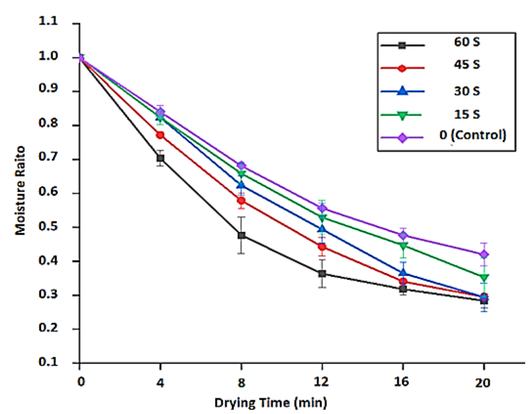

Fig. 2. Variations of moisture ratio with drying time at different pretreatment times for saffron stigmas.

2.3. Mathematical modeling of drying curves

The moisture ratio (MR) of thin layer of saffron stigmas was measured using Eq. (1). MR represents the moisture content of the sample at each time of drying relative to the initial moisture.

$$M_R = \frac{M_t - M_e}{M_0 - M_e} \quad (1)$$

where M_R is the moisture ratio, M_t is the moisture at any time (kg water/kg dry matter), and M_e and M_0 are the equilibrium moisture of samples (kg water/kg dry matter) and the primary moisture of samples (Kg water/kg dry matter), respectively. For the long drying time, M_e would be neglected comparison with M_0 (Izli et al., 2018). Therefore, the M_R equation can be expressed as Eq. (2):

$$M_R = \frac{M_t}{M_0} \quad (2)$$

The mathematical models that have been utilized in this study are listed in Table 1. To determine the coefficients of descriptive models (k, k_1, a, etc.), the experimental data of saffron drying kinetics were estimated using a curve fitting toolbox in the MATLAB software. In order to determine the most accurate model describing saffron stigma drying kinetics, the coefficient of determination (R^2), root mean square error (RMSE), error sum of squares (SSE), and the chi-square (χ^2) were calculated. The most accurate model was chosen among the selected models based on the lowest values of RMSE, SSE, and χ^2, and the highest value of R^2 which were calculated by Eq. (3), Eq. (4), Eq. (5) and Eq. (6), respectively.

$$R^2 = \frac{\sum_{i=1}^{N}(MR_i - MR_{pre,i})\ \sum_{i=1}^{N}(MR_i - MR_{exp,i})}{\sqrt{[\sum_{i=1}^{N}(MR_i - MR_{pre,i})^2][\sum_{i=1}^{N}(MR_i - MR_{exp,i})^2]}} \quad (3)$$

$$RMSE = [\frac{1}{N}\sum_{i=1}^{N}(MR_{pre,i} - MR_{exp,i})]^{\frac{1}{2}} \quad (4)$$

$$SSE = \sum_{i=1}^{N}(MR_{pre,i} - MR_{exp,i})^2 \quad (5)$$

$$\chi^2 = \frac{\sum_{i=1}^{N}(MR_{exp,i} - MR_{pre,i})^2}{N - z} \quad (6)$$

where MR_{exp} is the observed moisture ratio of saffron stigma for N^{th} measurement, MR_{pre} is the predicted moisture ratio of the model, N is the number of observations (Data), Z is the number of coefficients used in each model, and i is the number of terms (Kumar et al., 2016; Keneni et al., 2019).

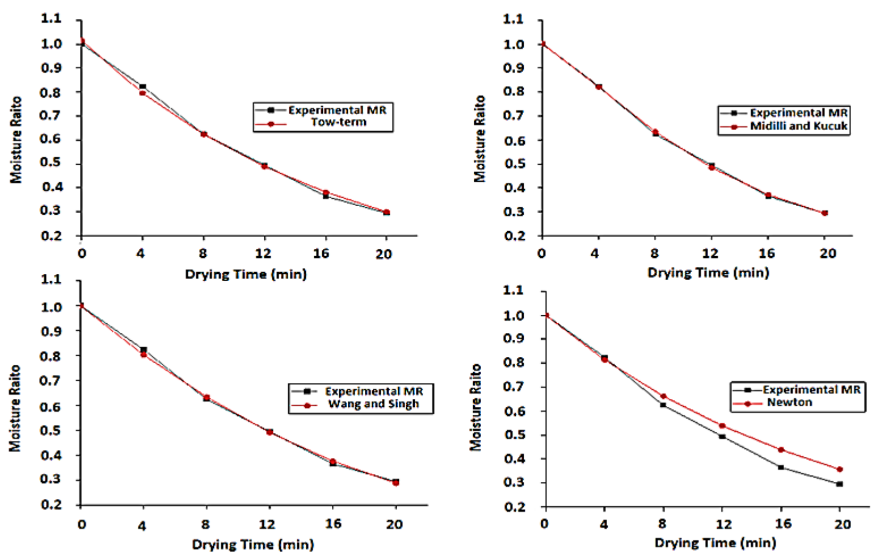

Fig. 3. Comparison of experimental data curves of drying of saffron stigmas in 30 s pretreatment with models predicted result.

Table 1. Common mathematical thin layer models for drying behavior.

No.	Name of Model Mathematical	Model equation	References
1	Newton	MR= exp (−kt)	Alara et al. (2017)
2	Page	MR=exp(−kt^n)	Alara et al. (2017)
3	Henderson and Pabis	MR=a exp(−kt)	Akpinar and Bicer (2008)
4	Logaritmic	MR=a exp(−kt) + b	Akpinar and Bicer (2008)
5	Two-term	MR=a exp(−kt) + a_1 exp(-k_1t)	Mohapatra and Rao (2005)
6	Two Term Exponential	MR=a exp(−kt) + (1−a) exp(-kat)	Younis et al. (2018)
7	Midilli and Kucuk	MR=a exp(−kt^n) + bt	Sobukola et al. (2007)
8	Diffision approximation	MR=a exp(−kt) + (1−a) exp(-kbt)	Blanco-Cano et al. (2016)
9	Verma et al.	MR=a exp(−kt) + (1−a) exp(-gt)	Younis et al. (2018)
10	Wang and Singh	MR = 1 + at + bt^2	Sobukola et al. (2007)

Note: MR is the moisture ratio, t is a time (min) and a, b, g, k and n of the model constant coefficients.

Table 2. Comparison of the effect cold plasma pretreatment on average MC and drying time.

pretreatment (s)	MC after cold plasma pretreatment (% wb)	Time to reach MC% of 43.37 (min)	Total drying time (min)
0	79.35 ± 0.70 [a]	16 ± 1.00[a]	37 ± 1.73[a]
15	66.51 ± 1.95 [b]	11 ± 1.00[b]	31 ± 1.00[b]
30	58.71 ± 2.74 [c]	6 ± 1.00[c]	26 ± 1.00[c]
45	49.29 ± 0.55 [d]	2.33 ± 0.60[d]	22.33 ± 0.57[d]
60	43.37 ± 0.82 [e]	-	20 ± 0.00[e]

Note: All data are expressed as mean ± standard deviations. Different superscripts in each column indicate a significant difference between the data ($p < 0.05$).

Table 3. Model constants and statistical analysis results for different pretreatment times.

Variables	Models	Model parameters							Statistical parameters			
		k	k_1	n	a	a_1	b	g	R^2	SSE	RMSE	χ^2
Control	Newton	0.04599							0.9959	0.001031	0.014360	0.000206
15		0.05153							0.9990	0.000293	0.007658	0.000058
30		0.05153							0.9950	0.001864	0.019310	0.002554
45		0.06578							0.9971	0.001067	0.014610	0.000213
60		0.07814							0.9705	0.011430	0.047810	0.002285
Control	Page	0.05132		0.9577					0.9965	0.000882	0.014860	0.000220
15		0.04952		1.0150					0.9991	0.000272	0.008248	0.000067
30		0.04423		1.1180					0.9986	0.000521	0.011420	0.000130
45		0.07290		0.9591					0.9976	0.000879	0.014830	0.000219
60		0.13800		0.7674					0.9897	0.003980	0.031540	0.000995
Control	Henderson and Pabis	0.04582			0.9980				0.9959	0.001025	0.016010	0.000256
15		0.05186			1.0040				0.9991	0.000271	0.008234	0.000090
30		0.06131			1.0180				0.9962	0.001401	0.018710	0.000358
45		0.06551			0.9970				0.9971	0.001055	0.016240	0.000263
60		0.07510			0.9703				0.9733	0.010330	0.050820	0.002582
Control	Logaritmic	0.06078			0.8457		0.16170		0.9978	0.000554	0.013590	0.000184
15		0.05185			1.0040		-0.00010		0.9991	0.000271	0.009508	0.000092
30		0.04866			1.1670		-0.15680		0.9976	0.000898	0.017310	0.000305
45		0.07880			0.9104		0.09540		0.9986	0.000534	0.013350	0.000178
60		0.13400			0.7851		0.22190		0.9972	0.001100	0.019150	0.000366
Control	Two-term	-0.27460	0.04876		0.0001	1.00700			0.9989	0.000272	0.011660	0.000136
15		0.04525	0.07591		0.7469	0.25840			0.9990	0.000296	0.012180	0.000152
30		0.06120	0.06033		0.8785	0.13770			0.9962	0.001407	0.026520	0.000718
45		-0.42450	0.06817		0.0000	1.00400			0.9997	0.000105	0.007267	0.000052
60		0.10180	-0.08407		0.9741	0.02996			0.9985	0.000581	0.017060	0.000290
Control	Two Term Exponential	0.07963			0.3876				0.9972	0.000697	0.013200	0.000174
15		0.05164			0.9531				0.9990	0.000293	0.008562	0.000073
30		0.05972			1.0080				0.9950	0.001864	0.021580	0.000465
45		0.10010			0.4562				0.9981	0.000682	0.013120	0.000172
60		0.24840			0.2345				0.9890	0.004258	0.032630	0.001061
Control	Midilli and Kucuk	0.04286		1.1910	1.0000		0.01014		0.9999	0.000016	0.002850	0.000008
15		0.04829		1.0650	1.0020		0.00244		0.9992	0.000224	0.010600	0.000114
30		0.04070		1.2160	1.0020		0.00409		0.9992	0.000280	0.011850	0.000142
45		0.05938		1.1460	0.9997		0.00668		0.9998	0.000071	0.005981	0.000035
60		0.08660		1.1240	0.0010		0.01071		0.9993	0.000265	0.011510	0.000132
Control	Diffision approximation	0.04656					0.90580		0.9959	0.001028	0.018510	0.000342
15		0.04777					0.70140		0.9990	0.000287	0.009792	0.000095
30		0.05027					0.52110		0.9964	0.001346	0.021180	0.000448
45		0.07400					0.41090		0.9979	0.000785	0.016180	0.000261
60		0.10040					-0.87790		0.9985	0.000599	0.014130	0.000199
Control	Verma et al.	0.04770			0.9999			-0.32470	0.9987	0.000313	0.010220	0.000546
15		0.05656			0.7997			0.03443	0.9989	0.000315	0.010250	0.000104
30		0.05957			1.0070			0.03961	0.9950	0.001858	0.024880	0.000618
45		0.06722			0.8427			0.05856	0.9971	0.001056	0.018760	0.000351
60		0.10040			0.9730			-0.08813	0.9985	0.000599	0.014130	0.000199
Control	Wang and Singh				-0.0468		0.00088		0.9989	0.000282	0.008399	0.000705
15					-0.0484		0.00081		0.9990	0.000296	0.008609	0.000073
30					-0.0525		0.00084		0.9981	0.000708	0.013310	0.000176
45					-0.0635		0.00142		0.9999	0.000474	0.003445	0.000011
60					-0.0814		0.00232		0.9962	0.001453	0.019060	0.000363

2.4. Determination of moisture diffusion coefficient

Diffusion is an important feature in the drying modeling of agricultural food products. This parameter depends on the temperature as well as moisture content of drying materials (Mirzaee et al., 2010). The diffusion equation of Fick's second law (Eq. (7)) implies the diffusion of mass (moisture) during falling rate period of drying for agricultural and food products. For applying the Fick's second law, it is assumed that the food product is one-dimensional, initially distribution of moisture in food is uniform, the main mechanism of mass transfer is diffusion, and external resistance is negligible. Calculating the effective moisture diffusion coefficient (EMDC) using the Fick's second law is a useful tool to describe the drying process and possible mechanisms for the transfer of moisture into food.

$$\frac{\partial X}{\partial t} = D\,\frac{\partial^2 X}{\partial Z} \tag{7}$$

where *X* is the amount of moisture (kg water / kg dry matter), *D* (m^2/s) is the effective moisture diffusivity, t (s) is the time of drying, and *Z* (m) is the distance for moisture penetration of fresh stigma. The above equation is mainly used for different geometrics including slab, cylinder and sphere and boundaries as well as initial conditions. The saffron stigmas were considered as an unlimited cylinder. Regarding to the geometry and boundary conditions the following equation (Eq. (8)) was used to determine the MR as a function of drying time (Dak & Pareek, 2014):

$$M_R = \frac{M_t - M_e}{M_0 - M_e} = \frac{4}{a^2}\sum_{n=1}^{\infty}\frac{1}{\beta_n^2}\exp(-D_{eff}\beta_n^2 t) \tag{8}$$

where M_R is the moisture ratio (dimensionless), D_{eff} is the effective moisture diffusivity (m^2/s), β and n is Bessel function roots of the first type and zero degree, a is the cylinder radius (m) and t is the drying time (s).

The EMDC coefficient is determined by plotting the natural logarithm of experimental moisture ratio against drying time applying following formula (Touil et al., 2014):

$$\ln(M_R) = \ln\left(\frac{4}{{\beta_n}^2}\right) - \frac{\beta_n^2}{a^2}D_{eff} \tag{9}$$

2.5. Statistical analysis

Statistical analysis of data was performed using SPSS software (IBM statistical analysis Version, 21). All results were expressed as mean ± standard deviations and to compare the means, Duncan's test was applied at a significant level of $p < 0.05$. MATLAB 17 software was used for mathematical modeling.

3. Results and Discussion

3.1. Influence of cold plasma pretreatment on drying kinetics

Saffron samples were dried to 12% moisture content (wb). Pretreatment by GAD decreased the moisture content of the all samples ($p < 0.05$), and it is intensified at longer pretreatment times (Table 2). This is due to the increase in surface temperature of stigmas and evaporation of surface moisture, in this case, the surface temperature difference between of the control sample and pretreated sample for 60s was 26ºC. The drying curves of the saffron stigma subjected different pretreatment time are shown in Fig. 2. As demonstrated in this figure, the increase of the pretreatment time led to a reduction in the drying time of saffron stigmas. Plasma active species ($OH^\bullet$, $NO^\bullet$, N^{2+}, O^*, etc.) may cause the stress, surface deformation and perforation of the surface (etching). It leads to reduce barriers against diffusion phenomena, and increase moisture transfer (Tabibian et al., 2020). On the other hand, creation of pores and cracks on the outer layer of the stigma may control the case hardening phenomena; which decreased resistance against water diffusion. Therefore, effective moisture diffusivity increased in subsequent falling rate drying compared with untreated samples. Enhancement of EMDC and diffusion rate of water molecules during drying led to improvement of quality characteristics of dried product (Shishir et al., 2019).

3.2. Kinetic modeling and selection of the best model

Nonlinear regression analysis was performed based on ten different drying models for GAD pretreated saffron. The values of the constants and statistical parameters of each model were obtained through fitting the experimental data to the models (Table 3). The best fitted models were selected based on the highest value of R^2 and lowest value of SSE, χ^2, and RMSE. The results indicated that the Two-term, Midilli and Kucuk and Wang–Singh models were more accurate among others for predicting the thin layer-drying process of saffron. According to the results, the Newton model was less consistent with experimental data. The moisture ratio curves of saffron stigma as a function of drying time for experimental data versus the data derived from Two-term, Midilli and Kucuk and Wang–Singh models were shown in Fig. 3. Experimental results were obtained from samples subjected to 30s GAD pretreatment and dried at temperature of 60ºC and hot-air velocity of 1.5 m/s. As it can be observed, the curves derived from Two-term, Midilli and Kucuk and Wang–Singh models are more consistent with obtained data, than that from Newton's model. Fig. 4 indicated the differences between the moisture ratios obtained from the experimental data against the moisture ratios predicted by Two-term, Midilli and Kucuk and Wang–Singh models.

Akhondi et al. (2011) have investigated the effect of six temperatures for drying thin layer of saffron in infrared dryer considering four models including Lewis, Handerson and Pabis, Page, and Midilli and Kucuk in which, Midilli and Kucuk model had higher accuracy. Mortezapour et al. (2014) used 11 different thin-layer models to describe the drying behavior of saffron stigma in heat pump assisted hybrid photovoltaic-thermal solar dryer. Their results showed that the two-term model was the best model to describe the drying characteristics.

3.3. Effective moisture diffusivity

In order to determine the EMDC, the natural logarithm of MR versus time was taken into account. EMDC values were calculated according to Fick's second law by using Eq. (8).

The values of EMDC and coefficient of determination as a function of pretreatment time are shown in Table 4. The results indicated that the highest value of EMDC, i.e., 9.1139×10^{-9} m^2/s, was achieved at 60s of pretreatment time, and the lowest value was contributed to the control sample, which was 8.7013×10^{-9} m^2/s. The cold plasma pretreatment affected the mass transfer (increased the effective moisture diffusivity) during the drying process and its effect is more evident at higher pretreatment times especially due to the reaction of active species of plasma with surface of saffron stigmas. There is no study done on the effect of plasma pretreatment on the effective moisture diffusivity. However, the effect of temperature and air velocity in hot-air drying is studied widely. In addition, the EMDC estimated in their study was in the range of $1.5658 \times 10^{-6} – 9.5024 \times 10^{-6}$ m^2/s.

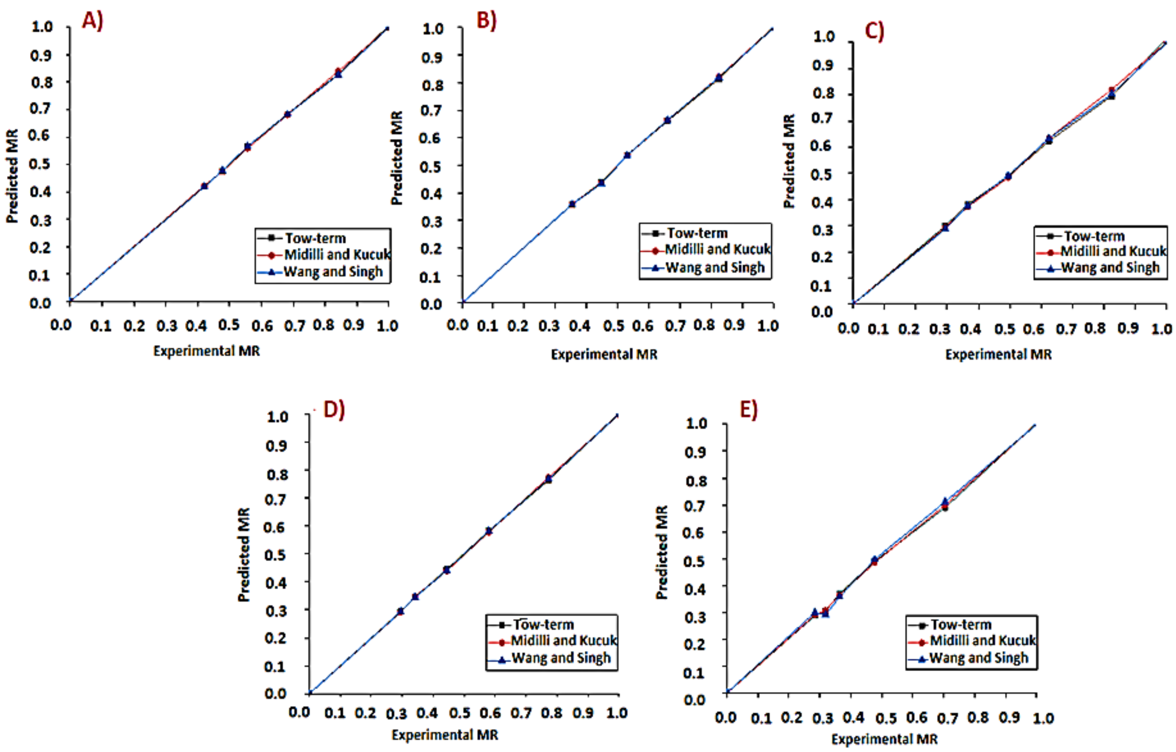

Fig. 4. Comparison between the actual (experimental) moisture ratio and the moisture ratio predicted by the three models Two-term, Midilli and Kucuk and Wang–Singh respectively for pretreatment (a) control, b) 15, c) 30, d) 45 and, e) 60 s.

Table 4. Effective moisture diffusion coefficient from Fick's second law at different pretreatment times of atmospheric cold plasma process.

Pretreatment conditions (s)	Effective diffusion coefficient (m^2/s)	Coefficient of determination (R^2)
0 (Control)	8.7013×10^{-9}	0.9934
15	8.8566×10^{-9}	0.9987
30	9.0905×10^{-9}	0.9972
45	9.0948×10^{-9}	0.9924
60	9.1139×10^{-9}	0.9490

4. Conclusion

The results revealed that the increase in the pretreatment time led to an increase in effective moisture diffusion, enchantment of drying rate, and the decrease in drying time. Irradiation, especially in a long period, may increase the number of pores on the cell wall and cause cracks on the surface of stigmas. This phenomenon led to improve in the rate of outflowing water from the crop. The statistical analysis was performed on ten chosen models and the results indicated that the Two-term, Midilli and Kucuk and Wang–

Singh models were the most accurate models that fitted well with the obtained data.

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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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