

Cold Plasma: A Novel Pretreatment Method for the Drying of Canola Seeds: Kinetics Study and Superposition Modeling

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Abstract

Accurate investigation of kinetics and development of high-precision drying models will help better studying the drying process by identifying effective parameters. Present study investigates the application of cold plasma (CP), as a pretreatment process, in air drying of canola seeds. This may bring about some complication into the drying kinetics investigation. Canola seeds with an initial moisture content of $27.5 \pm 1\%$ (dry basis) were exposed to CP for 0, 15, 30, and 60 s prior to fluidization by air at temperatures of 40, 50 and 60 °C in a pilot scale fluidized bed heated by a solar panel. The results showed a decreasing trend in drying time from 40 to 60 °C. The shortest drying time corresponds to samples dried at 60 °C with no CP pretreatment. The longest period however occurred for samples dried at 40 °C with 60 s of CP pretreatment. It was also observed the greatest effect of CP on reducing the drying time at temperatures of 40 and 50 °C was at the CP exposure time of 15 and 60 s, respectively. A reasonably accurate study of drying kinetics was accomplished using the superposition method. Accordingly, using experimental data, curves correspond to different drying conditions were plotted and in two steps these were shifted to a reference curve to acquire a final drying curve. The curve then was fitted to a second-order equation, and was validated using the experimental data. The correlation coefficients, mean square error and mean absolute error were 0.99, 0.03, and 0.023, respectively.

Keywords

Canola seeds, Superposition, Shifted factor, Mean comparison

1- Introduction

Drying is a process by which the water in food is reduced by evaporation or sublimation using a heat source with precise and controlled conditions. Drying, while reducing the

risk of mold, decreases the final weight of the product and makes the material easier to transport. Canola (*Brassica napus*) is a plant of the nightshade family, annual, allogamy, and one of the most important crops whose seeds are used to produce oil. (McVetty and Duncan, 2016). The initial moisture content of canola seeds at the harvesting time is about 25%, which must be dried to prevent spoilage of wet seeds. Today, the use of hybrid methods helps speeding up the drying process. Cold Plasma (CP) is a non-thermal technology that includes gases containing reactive electrons, ions, and neutral species which shown to possess antimicrobial characteristics, change molecular structure, causes physical and chemical changes at the polymer surface, inactivate enzymes, reduce or eliminate toxins (Amin and Ghoran nevis, 2016; Pankaj et al., 2015; Pankaj & Keener, 2018). Food types, shapes, their ingredients and even their moisture content have a significant effect on the efficacy of CP (Li et al., 2019). CP is a promising new technology which has been widely used in food and agriculture and may have the potential to enhance the drying process, in turn improves the nutritional value of the products. There are small number of studies which investigated the influence of pretreatment by CP on the drying kinetics (Zhang et al., 2019).

Modeling the drying process not only ensure achieving an optimum design but also save time and money when scaling up the process. There are theoretical, semi-empirical, and empirical methods in mathematical modeling of the drying process (Ghasemi et al., 2021; Benseddik et al., 2018). Theoretical models are based on the physical principles of drying such as heat, mass, and momentum transfers. These methods are less accurate and in the majority of cases require fast and expensive computing capabilities due to a large computational processing. Semi-empirical modeling on the other hand may be one of the best methods to describe the drying process. In the semi-empirical technique, we use recognized equations developed by other researchers and for other products. In comparison, the semi-empirical models are more accurate than theoretical ones. However, there are some drawbacks too, as lack of standard models and the use of a specific template has practically made the application of the semi-empirical more difficult for some products. It is well known that highest accuracy might be obtained when applying empirical models (Simha et al., 2016; Benseddik et al., 2018). Among the empirical models, the application of the superposition method, in addition to identifying the direct effect of traits, can increase the accuracy of the final model. It was

used to explain the drying kinetics of thyme leaves at temperatures of 30, 40, and 50 °C, and three air velocities of 0.5, 0.8, and 1.2 m/s. The final curve, obtained using this method was an exponential function of two parameters (temperature and air velocity), with a determination coefficient of 0.996 (Khazaei et al., 2008). In another study, the method of superposition for modeling the drying kinetics of Aloe vera gel slices under different temperatures and air velocities was investigated. The findings showed that the developed model possess acceptable accuracy (Moradi et al., 2019).

To the best of our knowledge, no article has been published to incorporate the influence of cold-plasma pretreatment in the modelling of drying process. In order to investigate the effect of CP pretreatment on the drying kinetics of canola seeds, a fluidized bed dryer operating at 3 inlet air temperatures was set up. The seeds were dried with and without CP pretreatment. A model based on the superposition was developed to simulate the drying kinetics of canola seeds.

2- Materials and Methods

2-1- Sample preparation

Fresh canola seeds were harvested from a farm located in the School of Agriculture of Shiraz University. The initial moisture content of seeds was determined to be $27.5 \pm 1\%$ (dry basis). The drying in a fluidized bed was carried out using 250 g of fresh seeds. The drying was continued until the average moisture content was around 8% (dry basis).

2-2- Dryer

A solar assisted fluidized bed dryer was deployed to investigate the drying kinetics of canola seed (Figure 1). An electric blower (1) (3 phases with a maximum power of 3 kW) was used to blow ambient air on a black galvanized flat plate solar collector (2) with an area of 4 m². The air then enters an auxiliary heating channel (4) which includes an electrical heater (2 kW) after leaving the collector. By adopting a control system, the electrical heater is turned on if the air outlet temperature from the collector is less than the desired drying temperature. The air velocity (6) was adjusted to ensure that the grains are in vibrating motion within the chamber (5) throughout the drying process. Based on the preliminary experiments, the air velocity of 3 m/s was determined to be suitable. This velocity was used throughout this experimental study. The drying chamber was cylindrical in shape made of transparent Plexiglas having a diameter and

height of 190 and 400 mm, respectively. To adjust the air temperature to the desired value, a thermocouple (type K with an accuracy of 1 °C) was installed just before the drying chamber. Fresh canola seeds with an initial moisture content of about 27.5 ± 1 % (dry basis) were poured in the drying chamber and was dried to moisture content of 8% (dry basis).

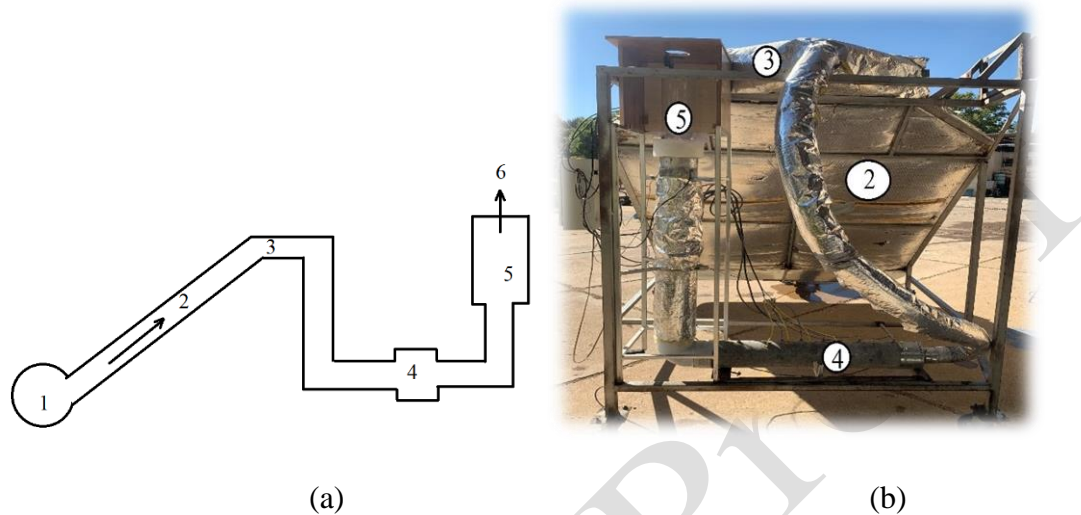


Fig.1. a) Schematic and b) back view of the solar dryer deployed in the present study

- 1- Electric blower, 2- Solar collector, 3- Collector outlet, 4- Heating channel, 5- Drying chamber, 6- Air outlet

2-3-Cold plasma

The cold plasma device (Nik Plasma Tech Co., Tehran, Iran) was applied for the pretreatment process. The equipment consisted of a generator and a reactor. The reactor consisted of two electrodes and two dielectric plates, an inlet for the flow of selected gas. In the process, after adjusting the generator to desired set up values, fresh canola seeds were spread on a plate between two electrodes in a thin layer manner. It was then exposed to CP for 15, 30 or 60 s, depending on the experiment predetermined times. The unit was operated at a voltage of 11 kV, a frequency of 14 kHz with N₂ gas at pressure of about 1 atm, as the CP gas. The whole process was operated at the atmospheric pressure.

2-4-Experimental design

In order to investigate the effect of different operating conditions on the drying time, three levels of air temperature (40, 50 and 60 °C) at an air velocity of 3 m/s were selected. Prior to the drying, the seeds were exposed to 0, 15, 30 or 60 s of nitrogen CP. The procedure was carried out in a completely randomized factorial design. All runs

were executed in triplicates and analysis of variance and mean comparison were performed on SPSS 16 software based on the Duncan's test.

2-5-Modeling

The superposition (SP) is an empirical method which has relatively high accuracy while being quite simple to be used in a simulation (Cheung et al., 2019). In simulating the drying process by SP, different curves should be shifted to a reference one. To accomplish this, firstly, the moisture ratio during drying time for different drying conditions was obtained using the experimental data. Then, the six standard models in the literature were fitted to the experimental moisture ratio and, the most appropriate one was selected to describe the moisture ratio of canola seeds at one-minute intervals. Thereafter, by selecting a temperature as the reference (in the present work, 50 °C), the drying curves for other temperatures (40 or 60 °C) were shifted to the reference curve by shift factors. The moisture ratio versus the logarithmic times for each run (with and without CP exposure) for 3 temperatures were plotted (Figs. 6 to 9), and the points with the same values of moisture ratio in the reference curve and the other curve, were identified and shifted by a reduced time factor (D), obtained from equation (1), while shift factor (a) is calculated using equation (2):

$$D = \text{Log}(t_r) - \text{Log}(t_i) \quad \text{Eq. (1)}$$

$$a = 10^D \quad \text{Eq.(2)}$$

Where the same value of moisture ratio was observed in time of " t_r " on the reference curve (50 °C) and in the time of " t_i " on the other curves (40 or 60 °C).

Thus, a shifted drying curve was achieved for each CP exposure time. In the next step, as in the previous step, by selecting a CP exposure time (15 s) as the reference, and by shift the other drying curves to it, a single drying curve was obtained. Therefore, by two stages of superposition, a final curve was obtained. Multiplying the shift factors, by the drying time at each step was performed to achieve the final reduced time, and using regression analysis, the equation of the final curve was attained as a function of the reduced time. To validate the model, the produced moisture ratios by the superposition model were compared to the experimental moisture ratio.

3- Results and Discussions

3-1- Drying kinetics

Moist canola seeds were harvested with an initial moisture content of 27 ± 1 % (dry basis) from the field. The seeds in a thin layer manner exposed to CP for 0 (no CP exposure), 15, 30 and 60 s prior to drying at 40, 50 and 60 °C. The drying air velocity was kept constant at 3 m/s throughout the experiments. Using analysis of variance, the effect of independent parameters on the drying time was investigated (Table 1).

Table 1- Analysis of variance The effect of CP pretreatment and air temperature on drying time

Variables	DF	Sum squares	Mean squares	F
T	2	5390.92	2695.46	2343.89**
P	3	9.56	3.20	2.80*
T×P	6	117.95	19.66	17.11**
Error	24	24	1.15	
Total	35	5542.43		

* and **: significant at a level of 5 % and 1 %, respectively.

Accordingly, two factors of temperature ($P < 0.01$) and CP exposure time ($P < 0.05$) and their interaction ($P < 0.01$) have a significant effect on the drying time. Therefore, any alteration in the said mentioned parameters results in change in the drying time. In similar studies, the highly significant effect of the drying temperature on the drying time was reported (Moradi et al., 2020; Yousefi et al., 2013). Other researchers' data indicates the significant effect of CP on the drying rate when drying wolfberry and corn kernels (Zhou et al., 2020; Li et al., 2019).

3-2- Means Comparison

The mean values of the drying time for different drying modes are given in Fig. 2. Accordingly, with increasing the drying air temperature, the drying time was decreased. When drying at 40 °C, CP pretreatment (15 s) was applied, the drying time was reduced by 3% in comparison with runs with no CP pretreatment. On the contrary, at longer CP exposure (30 and 60 s), to our surprise the drying time increased by 0.5 and 4.6%, respectively, in comparison to samples which did not experienced CP. Thus, at lower temperature range, the influence of longer duration of CP is negative in term of drying time; i.e. the optimum CP exposure time is 15 s. In another study, the effect of CP in four exposer times of 15, 30, 45, and 60 s on the drying time of red pepper at a constant temperature of 70 °C and air velocity of 6 m s^{-1} , was investigated. The optimum exposure time of 30s was reported to give rise to the lowest drying time (Zhang et al., 2019). At air temperature of 50 °C, increasing CP exposure duration from 15 to 60 s,

lead to decrease in the drying time from about 3% to over 12%, compared to no CP exposure. Here, we observed the positive trends in effect of CP pretreatment on drying time. Drying at 60 °C; we observe a negative effect of exposure to CP. The drying time went up between 10-16% as the seeds were pretreated between 15 and 60 s by CP. Hence, when drying at 60 °C, we would be better off not to exposed the canola seeds prior to the air drying. CP applied on the surface of the drying material, creates small cracks in the cell wall. This is good for water release from the seeds; but increasing the exposure duration may lead to cell wall destruction and loss of porosity which in turn reduces the water vapor evaporation from the seed's surface (Zhang et al., 2019). Similar adverse effect may be the reason for this discrepancy in the trend of drying time while drying canola seeds.

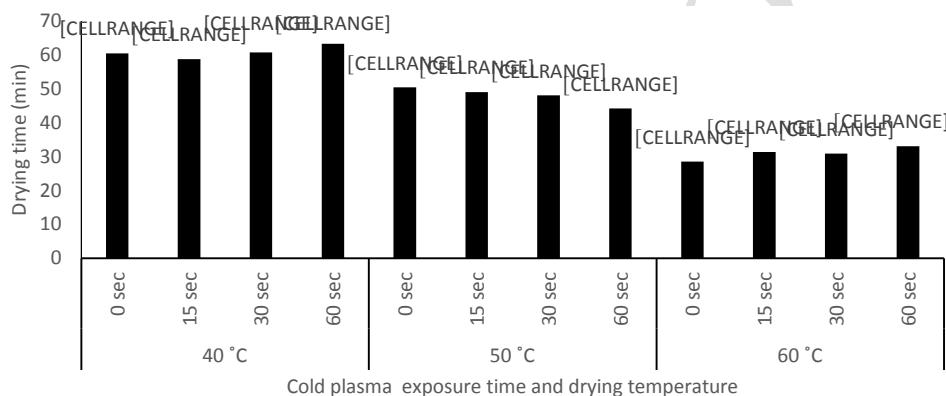


Fig. 2. Mean drying time for different drying conditions with or without pretreatment by CP

Figures 3, 4, and 5 show the changes in the moisture ratio versus the drying time for temperatures of 40, 50, and 60 °C, respectively. Accordingly, the best pretreatment of CP in the temperatures of 40, 50, and 60 °C, were 15, 60, and 0 s, respectively. The reason for the change in CP behavior with the change in drying air temperature may be related to the effect of temperature on the seed shell microstructure under the influence of CP pretreatment. Probably, CP produces some sort of case hardening on the surface of the seeds hence moisture cannot easily exit the seeds surface. This may be attributed to the nature of CP being an amalgam of various particles. Another reason could be the drying effect of CP gas on seeds as the exposure duration is increased.

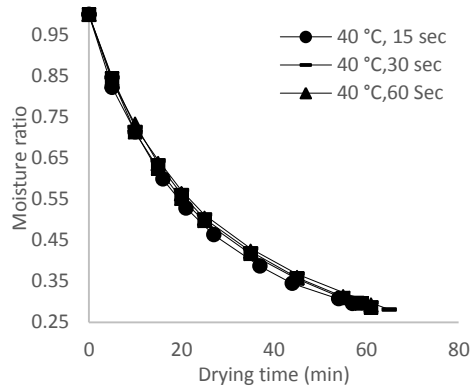


Fig.3. Variations of moisture ratio versus drying time for drying temperature of 40 °C

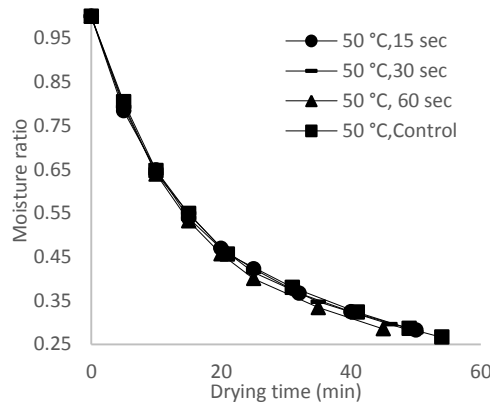


Fig.4. Variations of moisture ratio versus drying time for drying temperature of 50 °C

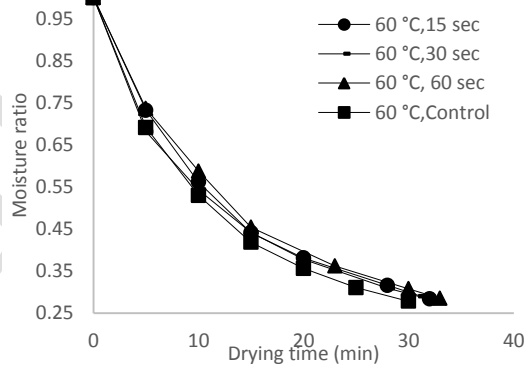


Fig.5. Variations of moisture ratio versus drying time for drying temperature of 60 °C

In another study, a CP air with ambient pressure and flow of 3 L/min and frequency of 20 kHz and power consumption of 750 W was used as a pretreatment of drying of red pepper. Red pepper samples were exposed to CP in four different time treatments (15, 30, 45, and 60 s). In all-time conditions, drying time was significantly reduced compared to the control treatment that did not use CP pretreatment. However, the

greatest reduction in drying time was associated with a CP time of 30 s (Zhang et al., 2019). In another study, the cooking time in half of rice was reduced after the application of CP. In that research, after CP pretreatment, cracks and depressions were observed on the surface of rice grain (Sarangapani et al., 2015). Increasing the exposure time to the CP can potentially lead to the formation of larger diameter cavities and thus reduce the drying time (Nishime et al., 2017). However, the drying time cannot be less than a certain amount, and with increasing CP time, cell wall disruption may occur; resulting in a reduced porosity and increased resistance to moisture movement within the material (Saengrayap et al., 2015). Jujube slices were dried at three temperatures of 50, 60, and 70 °C with (15,30 and 60s) or without CP pretreatment. The results showed that the interaction between the plasma pretreatment time and the drying temperature was significant so that at the temperature of 50 °C the usage of CP pretreatments showed less effect on the drying time than the control treatment (no CP pretreatment). While the use of drying temperatures of 60, and 70 °C with a cold plasma pretreatment of 15 s showed the greatest effect in reducing the drying time. However, with increasing the CP pretreatment time to 30 and 60 s, it was observed that the drying time was increased (Bao et al., 2021).

3-4-Developing a model based on superposition

In order to obtain the superposition model, at first, the moisture ratio of canola seeds should be determined during drying time. Therefore, standard semi-empirical models developed by the previous researchers, were used and the most appropriate model was selected based on the correlation between the seed moisture ratio predicted by the models and those obtained from the experimental results (Table 2).

Table 2- Coefficients of standard models and their correlation with experimental results

Model name	Model Coefficients	R ²	RMSE	X ²	MAE
Page	K=0.0464, n=0.8490	0.90	0.0864	0.0075	0.0628
Newton	K=0.0302	0.88	0.0966	0.00949	0.0759
Modified page	K=0.0269, n=0.849	0.90	0.0863	0.00758	0.0628
Henderson and Pabis	K=0.0291, a=0.9834	0.87	0.0944	0.0090	0.0749
Logarithmic	K=0.0449, a=0.7534	0.86	0.0999	0.0101	0.0655
Two term	a=0.5686, k ₀ =0.0115, b=0.4333, k ₁ =0.0726	0.92	0.086	0.0075	0.0595

K, n, a, k₀, and k₁ are model coefficients. Since Two term model, has a higher correlation coefficient and a lower error coefficient, it was selected as the most suitable

model to predict the instantaneous moisture ratio of the canola seeds. In another study, the drying of rice paddy grains in a fluidized bed dryer was simulated using the above standard models. The results indicate that Midilli model was best fitted to the experimental data (Khanali et al., 2012). Therefore, in the present study, to determine the sum of drying effects, the moisture ratio of canola seeds at one-minute intervals was predicted using Two term model. The variations of moisture ratio against the logarithmic time were obtained and plotted for different experiments (Figures 6 to 9).

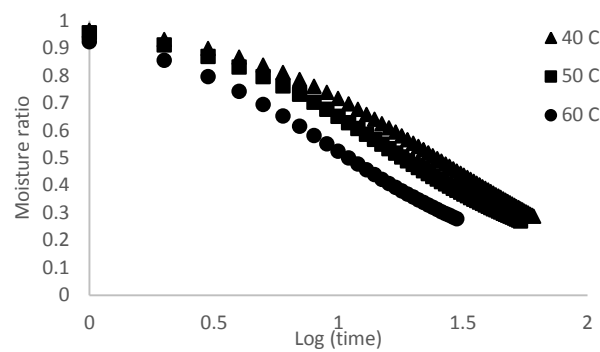


Fig. 6. Moisture ratios versus Log (time) for control experiments (no CP treatment) at different temperatures

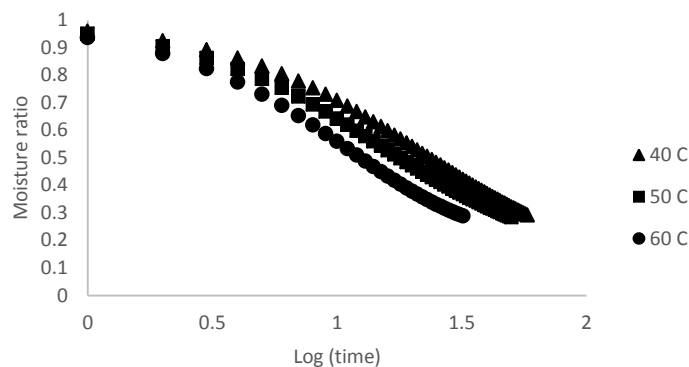


Fig. 7. Moisture ratios versus Log (time) for experiments (15 s CP treatment) at different temperatures

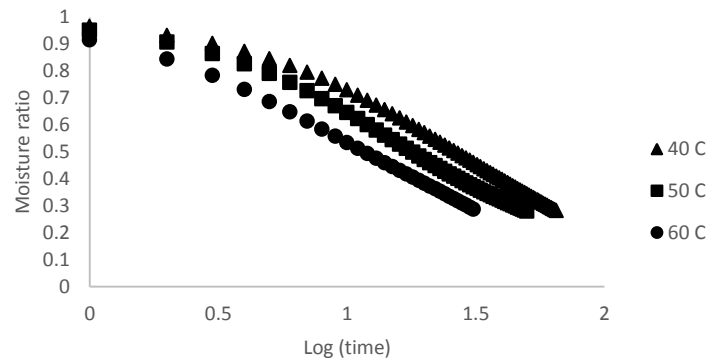


Fig. 8. Moisture ratios versus Log (time) for experiments (30 s CP treatment) at different temperatures

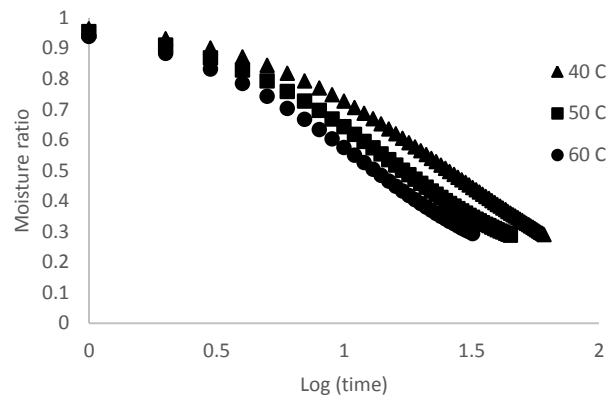


Fig. 9. Moisture ratios versus Log (time) for experiments (60 s CP treatment) at different temperatures

As there are two independent parameters, namely temperature and CP duration, the transfer process ought to be materialized in two stages in order to med to generate a final drying curve. Hence, in the first stage, in figures 6-9, points with the same value of moisture ratio were identified, and by application of equations (1) and (2), temperature shift factors (aT) were calculated (Table 3). Afterwards, in each CP duration, by shifting the curves with temperatures 40 and 60 °C to the reference curve (50 °C), four shifted curves were obtained. For the second transfer stage, in those curves, points with the same moisture ratio values were identified, and CP time shift factors (aP) were calculated using Equations (1) and (2) (Table 3). Here, 15s CP exposure time was designated as the reference curve and the other three curves (0, 30, and 60 s CP treatment) were shifted to this curve using the aP and the final drying curve was obtained (Fig. 10). " aT " for the experiments at 40 °C is smaller and at 60 °C is greater

than one. This is an indication that drying time for 40 °C is longer and 60 °C is shorter compared to 50 °C. For 40 °C, the greatest "aT" value of 0.78 is observed when the CP exposure time was 15 s. This indicates a shorter drying time in comparison with other plasma modes is at 40 °C. When setting the air temperature to 50 °C, at longer exposure time of seeds by CP, the aP is increased to the highest value of 1.031 at the CP exposure time of 60 s. Bearing in mind that the highest value of "aT" at 60 °C corresponds to 0 exposure duration (The control runs). Therefore, it is shown that the shortest drying time at 60 °C is when the seeds are not exposed to the cold plasma prior to hot air drying process. These interesting findings reveals that the results obtained in the superposition model are in good agreement with those acquired in the experimental works which presented and discussed in the previous section.

Table 3- Shift factors at different plasma times and temperatures

T (°C)	P (sec)	aT	aP
40	0	0.753	0.931
50	0	1.000	0.931
60	0	1.659	0.931
40	15	0.780	1.000
50	15	1.000	1.000
60	15	1.480	1.000
40	30	0.698	0.991
50	30	1.000	0.991
60	30	1.548	0.991
40	60	0.693	1.031
50	60	1.000	1.031
60	60	1.330	1.031

Based on Figure 10, the final curve obtained from the shifting of 12 different curves, can accurately predict the moisture ratio with a quadratic equation (Equation 3).

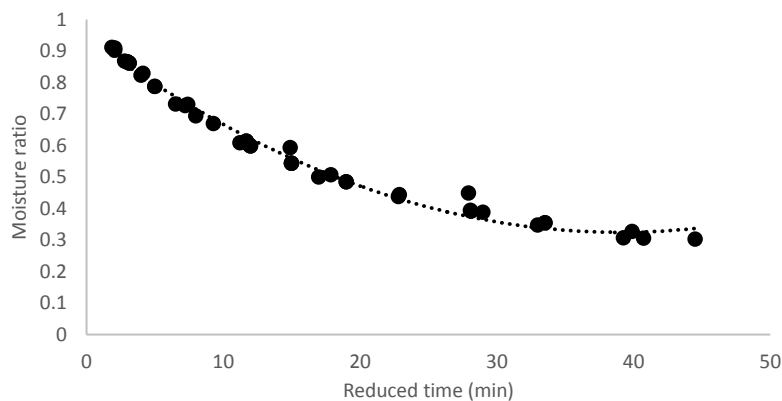


Fig.10. Final shifted curve with the polynomial regression equation

$$MR = 0.0004 t'^2 - 0.0318 t' + 0.9441 \quad R^2 = 0.988 \quad \text{Eq. (3)}$$

Where t' is the reduced time, obtained from Equation (4):

$$t' = t \times aT \times aP \quad \text{Eq. (4)}$$

That " t " is the actual drying time. Thus, in order to simulate the moisture ratio of the drying product, at first, the reduced time should be calculated using Equation (4) and the amounts of " aT " and " aP " given in the Table (3). Then, moisture ratio can be obtained using Equation (3).

3-5- Evaluation of model

Using Equation (3), all the moisture ratios of different experiments were obtained and the results were plotted against the actual moisture ratios obtained by the experiment (Figure 11).

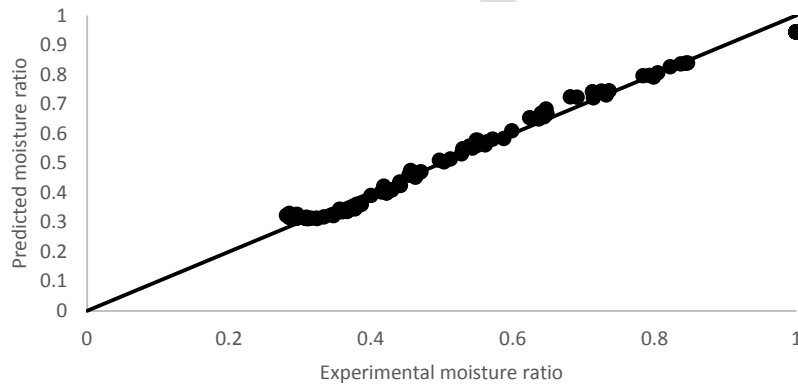


Fig. 11. Comparison between experimental and predicted moisture ratio

Accordingly, good agreement between predicted and measured moisture ratio was observed. The mean absolute error (MAE), k^2 , and R^2 were 0.022, 0.0009, and 0.98, respectively. These are indications that the developed superposition model accurately predicts the drying kinetics of canola seed pretreated with CP. Simulation of drying of Aloe vera gel slices in a cabinet dryer with two drying modes of with and without osmotic pretreatment was performed using the superposition method. The results showed that the final model has a good accuracy compared to the other theoretical and semi-empirical methods. (Moradi et al., 2019). In another study, modeling of drying

thyme leaves in a cabinet dryer was performed using the superposition technique, that the results of which revealed the final model has a good correlation with the experimental results (Khazaei et al., 2008). In another study, the Guava drying process was simulated using the Midilli-Kucuk model and the superposition model. The results showed that the second-order equation of superposition can predict simulation with great accuracy (Kek et al., 2014). Therefore, the method of superposition, which is generally used to simulate processes in engineering, in comparison with the other modeling methods, in addition to very high accuracy, has a very little modeling time and cost (Wang and Sharma, 2018). Such models have a very high power and validity in predicting existing processes in engineering (Wang et al., 2018). The superposition method can help the final design of the device by separating the effect of independent parameters on the dependent one. As can be concluded in the present study, the effect of CP and temperature on the drying kinetics can be seen in full detail.

4- Conclusion

A fluidized bed dryer was employed to investigate the effect of cold plasma pretreatment on the drying kinetics of canola seeds. In the experiment, canola seeds were dried at 40, 50 and 60 °C following exposure to 0, 15, 30 and 60 s of CP. The shortest drying time of less than 30 min was accomplished when drying seeds at 60 °C without exposure to CP.

Also, the shortest drying time for the temperature of 40 and 50 °C was at the CP exposure time of 15 and 60 s, respectively. However, further investigation is required to examine the effect of CP on other properties of dried canola seeds. The superposition technique used to simulate the drying process offered the simplicity as well as reasonably good accuracy. It exhibits the influence of each independent variable on the drying time. The developed model accurately predicted the time in seeds drying with different CP exposure time. Thus using an empirical model, we can investigate the effect of independent parameters of temperature and CP exposure time in full detail.

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پلاسمای سرد: یک روش پیش تیمار جدید برای خشک کردن دانه های کلزا: مطالعه سینتیک و مدل سازی جمع آثار

چکیده

بررسی دقیق سینتیک و توسعه مدل های خشک کردن با دقت بالا با شناسایی پارامترهای موثر به مطالعه بهتر فرآیند خشک کردن کمک می کند. تحقیق حاضر به بررسی کاربرد پلاسمای سرد (CP) به عنوان یک فرآیند پیش تیمار، برای خشک کردن بذر کلزا با هوای گرم شده می پردازد. این پدیده ممکن است باعث ایجاد پیچیدگی هایی در بررسی سینتیک خشک کردن شود. دانه های کلزا با رطوبت اولیه 27.5 ± 1 درصد (بر اساس خشک) ابتدا تحت زمان های صفر، ۱۵، ۳۰ و ۶۰ ثانیه در معرض CP قرار گرفته و سپس در خشک کن بستر سیال که گرمای آن توسط یک جمع کننده خورشیدی تامین می شود، در دماهای ۴۰، ۵۰ و ۶۰ درجه سلسیوس خشک شد. نتایج بدست آمده حاکی از روند کاهشی زمان خشک شدن از ۴۰ تا ۶۰ درجه سلسیوس بود. کوتاه ترین زمان خشک شدن مربوط به نمونه هایی است که در دمای ۶۰ درجه سلسیوس بدون پیش تیمار CP خشک شده اند. با این حال، طولانی ترین دوره برای نمونه های خشک شده در دمای ۴۰ درجه سلسیوس با پیش تیمار CP ۶۰ ثانیه رخ داد. همچنین بیشترین تاثیر پلاسمای سرد بر کاهش زمان خشک کردن در دماهای ۴۰ و ۵۰ درجه سلسیوس به ترتیب با پیش تیمار CP ۱۵ و ۶۰ ثانیه مشاهده شد. مطالعه دقیق سینتیک خشک کردن با استفاده از روش جمع آثار انجام گرفت. بر این اساس، با استفاده از داده های تجربی، منحنی های مربوط به شرایط مختلف خشک کردن رسم شده و در دو مرحله به منحنی مرجع انتقال داده شدند تا منحنی خشک کردن نهایی به دست آید. سپس منحنی به یک معادله مرتبه دوم برازش داده شده و با استفاده از داده های تجربی اعتبار سنجی انجام گرفت. ضرایب همبستگی، میانگین مربعات خطا و میانگین خطای مطلق به ترتیب ۰/۹۹، ۰/۰۳ و ۰/۰۲۳ بودند.

واژه های کلیدی

دانه کلزا، جمع آثار، ضریب انتقال، مقایسه میانگین