Experimental Computational Study on Drying of Black Cumin (*Nigella sativa)* in a Fixed Bed Dryer and in a Thermo-gravimetric Analysis System

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KEYWORDS

Black cumin seeds; drying; thermal analysis; mathematical modeling; kinetic

ABSTRACT

In this study, drying of black cumin seeds (BCs) (*Nigella sativa*) with initial moisture content (MC) of %56.74 (dry basis (d.b)) was investigated in a novel designed fixed bed drying system (isothermal condition) and in a thermo gravimetric analysis system (non-isothermal condition). In isothermal conditions, the drying experiments were carried out at different temperatures (40, 60, and 80 °C) and air velocities (0.25, 0.5 and 1 m/s) and bed heights (14 mm and 28 mm). Several models in literature were selected to fit the experimental data. The fit quality of models was evaluated using the coefficient of determination (R^2), sum square error (SSE) and root mean square error (RMSE). Two term model has a good agreement with the experimental data and gave the best results for BCs. The activation energy was calculated to be as 19.92 kJ/mol and effective diffusivity values were calculated to be between $2.85 x 10^{-10}$ and $6.77 x 10^{-10}$ m^2/s depending on air temperatures. Furthermore, drying and decomposition behaviours of BCs in thermo gravimetric analysis system were studied in nitrogen flow of 0.850 ml/s and of constant heating rate of 5 °C/min towards 250 °C by means of non-isothermal methods.

تجربة دراسة محوسبة على تجفيف الكمون الأسود أو الحبة السوداء في مجفف سرير ثابت ونظام التحليل الحراري *(Nigella Sativa)*

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الكلمات الدالة

بذور الكمون الأسود؛ التجفيف؛ التحليل الحراري؛ النمذجة الرياضية

المُستلخص

استهدفت هذه التجربة دراسة تجفيف حبوب الكمون الأسود *Nigella sativa* في حاوية بنسبة رطوبة 56.74% إلي جانب استقصاء التحليل الحراري ووضع الأنموذج الرياضي **الأنسب** وذلك باستخدام جهاز تجفيف ذات درجة حرارة مئوية متفاوتة مابين (40°C / 80°C /60°C) وسرعة سريان هواء يتراوح ما بين (0. 25m/s، 0.5 m/s ، 1 m/s) يوضع على مستوي ارتفاع يتراوح ما بين (28mm /14mm) . تم إختيار نماذج مختلفة لتثبيت بيانلت التجربة، كما تم تقييم مدى جودة هذه النماذج باستخدام معامل التحديد(R^2) ومجموع مربع الخطأ (SSE) وجذرمتوسط مربع الخطأ (RMSE). إستنتجت التجربة توافق نموذجان من هذه النماذج المستخدمة مع بيانات التجربة بمستوي جيد إذ أعطت أفضل نتائج تجفيف الكمون االأسود *Nigella sativa*. تم حساب نشاط طاقة االتجفيف (19.92 kJ/mol) كما تم حساب قيم فاعلية انتشار طاقة التجفيف والذي يتراوح مابين ($2.85x 10^{-10}$ الي 6.77 $x 10^{-10}$) في درجة حرارة الهواء . بالإضافة الى ذلك، تمت دراسة كيفية التجفيف والتحليل لحبوب الكمون الأسود *Nigella* sativa على نظام التحليل الوزني بانسياب غاز النتروجين بسرعة (0.85 مم/ الثانية) في معدل التسخين الراتب (5°درجات مئوية / الدقيقة) حتى درجة (250° درجة مئوية) بكيفيات غير متساوية الحرارة.

Nomenclature: BC : black cumin/ D_e : effective moisture diffusivity (m^2/s)/ D_0 : constant (-)/ E_a : activation energy (J/mol) /K : slope/ L : half thickness of slab (m)/ M_t : moisture content at time t (g water/g dry matter)/ M_0 : initial moisture content (g water/g dry matter)/ M_e : equilibrium moisture content (g water/g dry matter)/ MR : moisture ratio (dimensionless)/ MR_{ei}: ith experimental moisture ratio (dimensionless)/ MR_{pi} i^{th} : predicted moisture ratio (dimensionless)/ M_t : the MC at time t (g water/g dry matter)/ M_{t+dt} : Moisture content at t+dt (g water/g: dry matter)/ N: number of observations/ R_g : universal gas constant (kJ/ mol K)/ RMSE: root mean square error/ SSE: sum square error/ R^2 : coefficient of determination/ T: drying temperature (°C)/ TG: thermo gravimetric/ T_a: absolute temperature (K)/ β: hating rate.

Introduction

Black cumin seeds (BCs) (*Nigella sativa*) is known as a spice, food preservative and one of the greatest form of healing medicine plants available in the world. They have been used for therapeutic purposes in the "Alternative Medicinal" by many Asian, Middle Eastern and Far Eastern Countries to treat headache, coughs, abdominal pain, diarrhea, asthma, rheumatism and other diseases. The aqueous and oil extracts of the seeds have been shown to possess antioxidant, anti-inflammatory, anticancer, analgesic and antimicrobial activities (Gali-muhtasib *et al*. 2006). In folk medicinal practices, BCs are ingested with food or mixed with honey and are primarily used as lactogoguse, carminitative and antihelmnthic agents. Also the seeds have been used as diuretics, anti-hypertensive, muscle relaxants and as immunity enhancers in immune-compromised people. Importantly, the seeds have been reported to be safe when used orally with a moderate amount in food (Dermarderosian *et al.* 2005).

BC plant is grown in Mediterranean region and Western Asian countries including, India, Pakistan, Afghanistan, Saudi Arabia (Subhash *et al*. 2008) and Turkey, which is an annual flowering plant and about 20-30 cm tall. The flowers are delicate, and typically colored pale blue and white, with five to ten petals. The fruit is a large and inflated capsule composed of three to seven united follicles, each containing many seeds.

There are different methods of food preservation, such as: canning, freezing, pickling, curing (smoking or salting), and drying (en.wikipedia.org, 2011). Drying method is mass transfer process in which moisture from solid or nearly solid material is removed by evaporation, which is mostly used in a range of applications (Akpinar, 2005, Balbay *et al.* 2011, Kumar *et al.* 2012). It depends on different factors such as air temperature and air velocity, relative humidity of air, physical nature and initial MC of drying material (Akpinar *et al*. 2003). The applied energy for drying is important by means of energy costs and quality products (Balbay *et al*. 2011, Syahrul *et al*. 2002). Also, drying kinetics is important in the analysis, design, simulation and optimization of drying processes. Therefore, many mathematical models have been developed to calculate the drying time under the given operating conditions (Turner and Mujumdar 1996, Akal *et al*. 2007, Gazor and Mohsanimenesh 2010). Modeling of the drying kinetics of foods has been widely investigated in the recent studies, such as coconut (Madhiyanon 2009), strawberry (Doymaz 2008), olive pomace (Maziane 2011) and seeded grape (Cakmak and Yildiz 2011), canola (Gazor and Mohsanimenesh 2010). The objectives of this research were:

(i) to determine drying kinetics of BCs in terms of different air temperatures and air velocities in a fixed bed drying system;
(ii) to determine the effect of moisture diffusivities and activation energy at different temperatures in fixed bed drying system;
(iii) to compare experimental measurements with the different mathematical models available in the literature;
(iv) to determine the drying behaviour of BCs in thermo gravimetric analysis system.

Materials and Methods

BC plants used in the experiments were harvested in August in Batman province, Turkey. "Batman is located at 37º 52 N and 41 07 E and 550 m above sea level in the Southern East of the Anatolia in Turkey." Then, the initial MC of BCs was determined with a separate test. To determine initial MC, a weight of 100 g of BCs was put down in the high-temperature oven at 105 ˚C for six hours. Based on mass change, the initial MC of BCs was determined as 56.74 %(d.b). The same value of moisture content was also obtained from thermo gravimetric analysis and presented as wet basis in Fig. 9. (see, figure 9).

(1) Experimental Set-up

The experimental set-up was designed and fabricated in the laboratory in order to dry some fruits and vegetables. A three dimensional solid drawing of the dryer is presented in Figure 1.

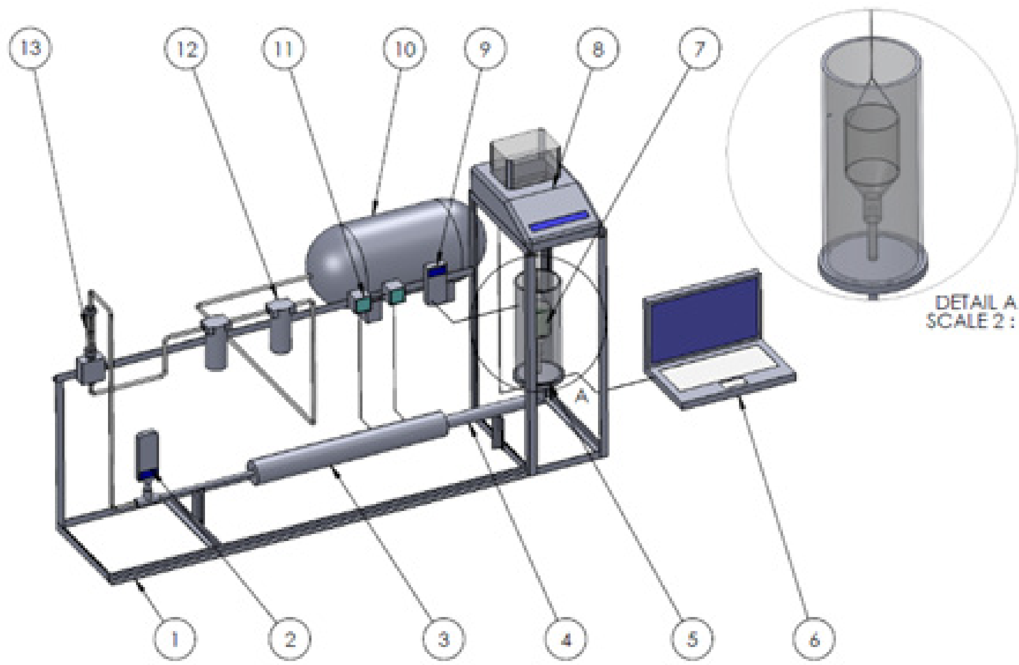

Figure1: The Schematic View of Experimental Set-up
(1.*Steel frame*, 2.*Thermometer&Hygrometer,* 3.*Electrical heater*, 4. *Isolated pipe*, 5.*Glass tube*, 6.*Notebook,* 7. *Drying cap*, 8. *Digital scale*, 9.*Thermometer,* 10. *Air compressor*, 11. *PID controller*, 12. *Air filters*, 13. *Flow meter*)

It consists of an air compressor, a steel frame, two air filters, a flow meter, a thermometer & hygrometer, an electrical heater, a thermometer, an insulated pipe, a drying cap, a glass tube, a digital scale, a PID controller, and a notebook. Air supplied from the compressor after being filtered by inlet and outlet filters with respectively was heated by electrical heater and fed to the drying cap. The temperature of the heating medium was controlled by using a PID controller having an accuracy of ±0.5 °C, which was connected to the electrical heater. Air velocity was measured by using a calibrated flow meter. In order to keep the temperature constant, the glass tube was insulated by rock wool.

The experiments were performed at different air temperatures (40, 60 and 80 ºC) and air velocity (0.25, 0.5 and 1 m/s). Generally, 20 g weight of samples (about 28 mm in bed height) was used in each experiment. Also, 10 g weight of samples (bed height: 14 mm) was used to determine the effect on BCs of bed height. Before each run was performed, the data acquisition system was switched on and air temperatures were then set to the desired temperature value. Then, the measured samples were put on the drying cap. Ambient temperature, relative humidity, air velocities, inlet and outlet temperature of the drying air in drying chamber and weight loss of BCs were recorded in short time periods (at the end of every 5 minutes) during the experiments.

(2) Mathematical Modeling of Drying Curves

Moisture ratio (MR) of BCs during the drying experiments is generally calculated using the following Equation 1. However, MR was simplified to M_t/M_0 since the value of equilibrium moisture content(M_e) is relatively small compared to M_t and M_0 (Midilli and Kucuk 2003, Kumar *et al*. 2012). The experimental drying data were converted to the dimensionless MR to compare with different models.

$$\mathrm{MR} = \frac{\mathrm{M_t} - \mathrm{M_e}}{\mathrm{M_0} - \mathrm{M_e}} \qquad (1)$$

The analysis for non-linear regression was performed by using MATLAB (version 2009a). In order to select a suitable drying curve, each model given in Table-1 was fitted to the experimental MR data. The values of R^2, SSE and RMSE were considered to select the best model. R^2 is one of the primary criteria to select the fit quality of these models (Midilli and Kücük, 2003), which ranges from 0 to 1. In addition to R^2 values, RMSE values are used to determine the quality of the fit too (Sobukola *et al*. 2007, Meziane 2011). The higher values for R^2 and the lower value for RMSE indicate better goodness of fit (Doymaz 2008, Maziane 2011). The RMSE and SSE can be calculated as follows:

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}(\mathrm{MR_{ei}} - \mathrm{MR_{pi}})^2\right]^{1/2} \qquad (2)$$

$$SSE = \frac{1}{N}\sum_{i=1}^{N}(\mathrm{MR_{ei}} - \mathrm{MR_{pi}})^2 \qquad (3)$$

(3) Analysis of Drying Rates

The drying rate of BCs was calculated by the following equation:

$$\mathrm{DR} = \frac{\mathrm{M_{t+dt}} - \mathrm{M_t}}{\mathrm{dt}} \qquad (4)$$

(4) Calculation of Activation Energy

The effective moisture diffusivity is an important transport property needed in the modeling of various drying food process. The analytical solution of Fick's second law of unsteady state diffusion in spherical body can describe the transport of moisture during the process that is occurred in the falling rate period, by assuming that the effective moisture diffusivity is constant during the drying process and is calculated by the following equation(Crank 1975): Fick's 2^{nd} law of diffusion describes the rate of accumulation (or depletion) of concentration within the volume as proportional to the local curvature of the concentration gradient.

$$\frac{\mathrm{M_t} - \mathrm{M_e}}{\mathrm{M_0} - \mathrm{M_e}} = \frac{8}{\pi^2}\sum_{n=0}^{\infty}\frac{1}{(2n+1)^2}\exp\left(-\frac{(2n+1)^2\pi^2\mathrm{D_e}t}{4L^2}\right) \qquad (5)$$

$$\ln\mathrm{MR} = \ln\left[\frac{8}{\pi^2}\right] - \left[\frac{\pi^2\mathrm{D_e}}{4L^2}\right]t \qquad (6)$$

The effective moisture diffusivity is calculated using the method of slopes obtained fitting experimental data to drying model. Diffusion coefficients are typically determined by plotting experimental drying data in terms of ln (MR) versus time (as given in Equation 6) (Tutuncu *et al*. 1996). From Equation (6), a plot of ln (MR) versus time gives a straight line with a slope as seen below:

$$\mathrm{Slope} = -\left[\frac{\pi^2\mathrm{D_e}}{4L^2}\right] \qquad (7)$$

The activation energy (7) can be obtained from the slope of the Arrhenius plot of ln (D) versus 1/T as follows:

$$\mathrm{D_e} = \mathrm{D_0}\exp\left(-\frac{\mathrm{E}_a}{\mathrm{R}_g\mathrm{T}_a}\right) \qquad (8)$$

Where, D_e is effective moisture diffusivity (m^2/s), E_a is the energy of activation (kJ/mol), R_g is the universal gas constant (8.3143 J/mol K), T_a is absolute air temperature (K) and D_0 is constant. From Equation 7, a plot of ln D_e versus 1/Ta gives a straight line whose slope is K, which can be found as below:

$$K = \frac{E_a}{R_g} \qquad (9)$$

(5) Thermo-gravimetric Analysis

Thermo gravimetric analysis measures weight change in a material as a function of temperature or time in a controlled atmosphere and so provides a convenient method for predicting optimum drying conditions. The method demonstrates weight loss in material due to decomposition or drying.

In this study, Perkin Elmer-Thermo gravimetric/Differential Thermal analyzer instrument were used for thermo gravimetric and differential thermal analysis (non-isothermal conditions). Thermo-gravimetric tests were performed in nitrogen flow of 0.850 ml/s and of constant heating rate of 5 °C/min towards 250 °C. Isothermal analyses can't be performed at this temperature, because BCs decompose before this temperature is attained.

Results and Discussion

(1) Analysis of Drying Kinetics

The variations of MR as a function of drying time at temperatures of 40, 60 and 80 ºC are shown in figure. 2. As can be seen figure 2, the MC decreases continuously with drying time. High temperature drying accelerates moisture removal as compared to low temperature drying. The MR falls rapidly in 50 minutes, on the other hand the rate of drying slows, as the BCs lose moisture. The temperature of drying air has a significant effect on the drying behaviors of BCs.

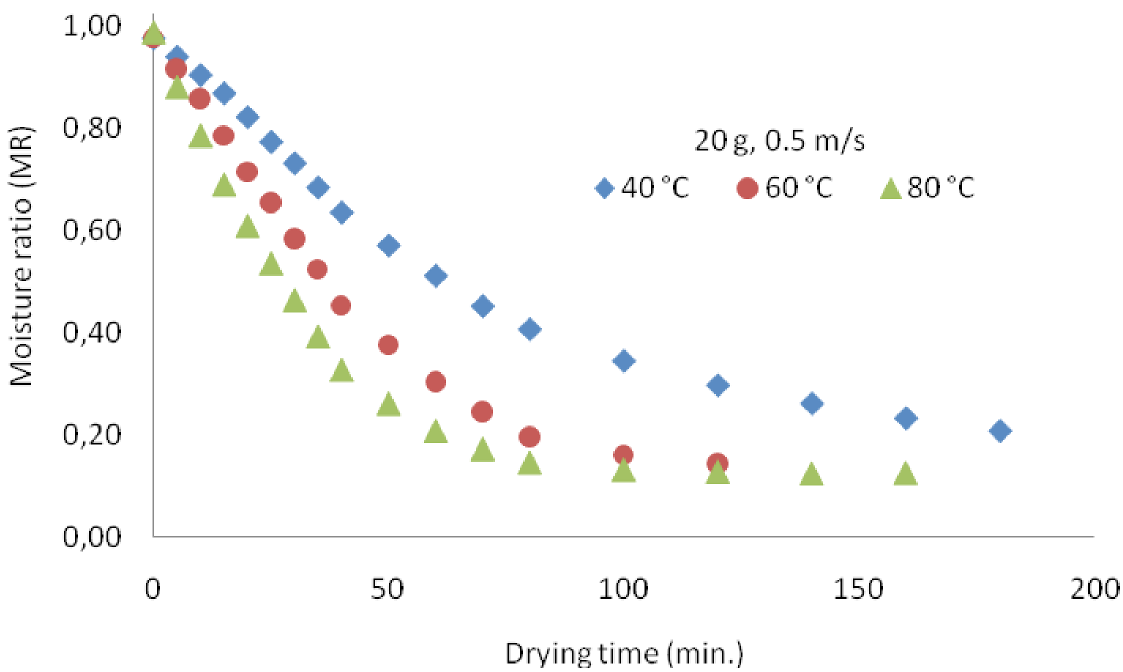

Figure 2. Variation of Moisture Ratio with Drying Time at Different Temperatures.

Figure 3 shows the effect of air velocities (0.25, 0.5 and 1 m/s) and constant air temperature of 60 ºC. The air velocity of drying air does not have effect on the increase of MR as much as temperature. Since, the capacity of air to remove moisture is principally dependent on its operating temperature. And also, it is seen that the effect of air velocity varying from 0.25 m/s to 0.5 m/s is higher than 0.5 to 1 m/s.

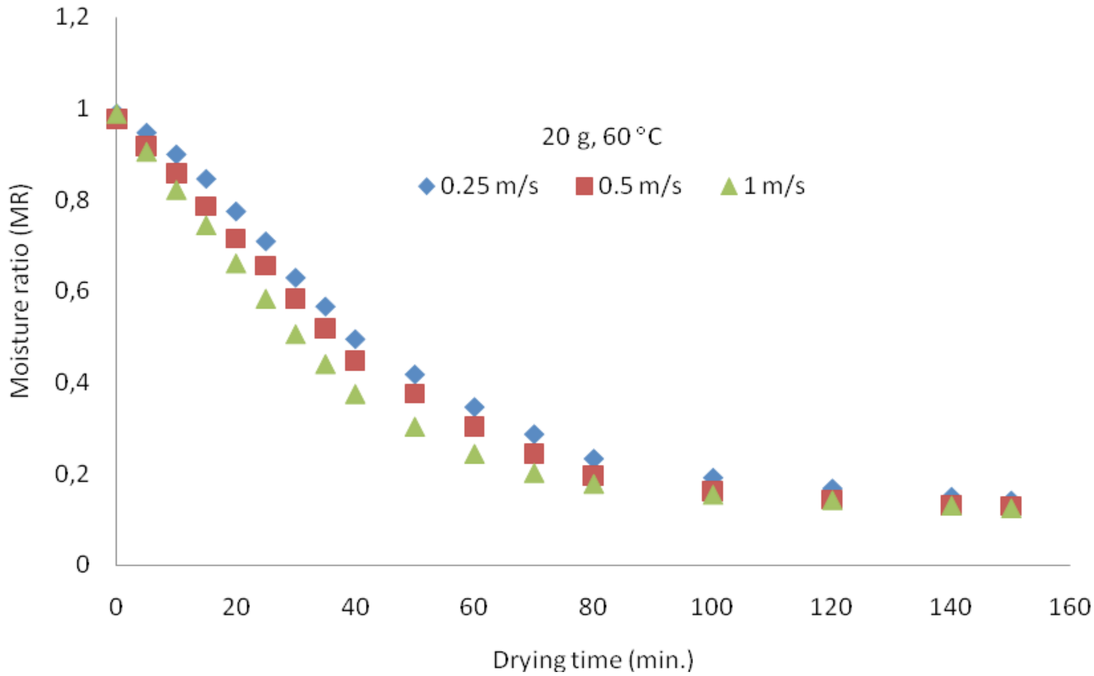

Figure 3: Variation of Moisture ratio with Drying Time at Different Air Velocities.

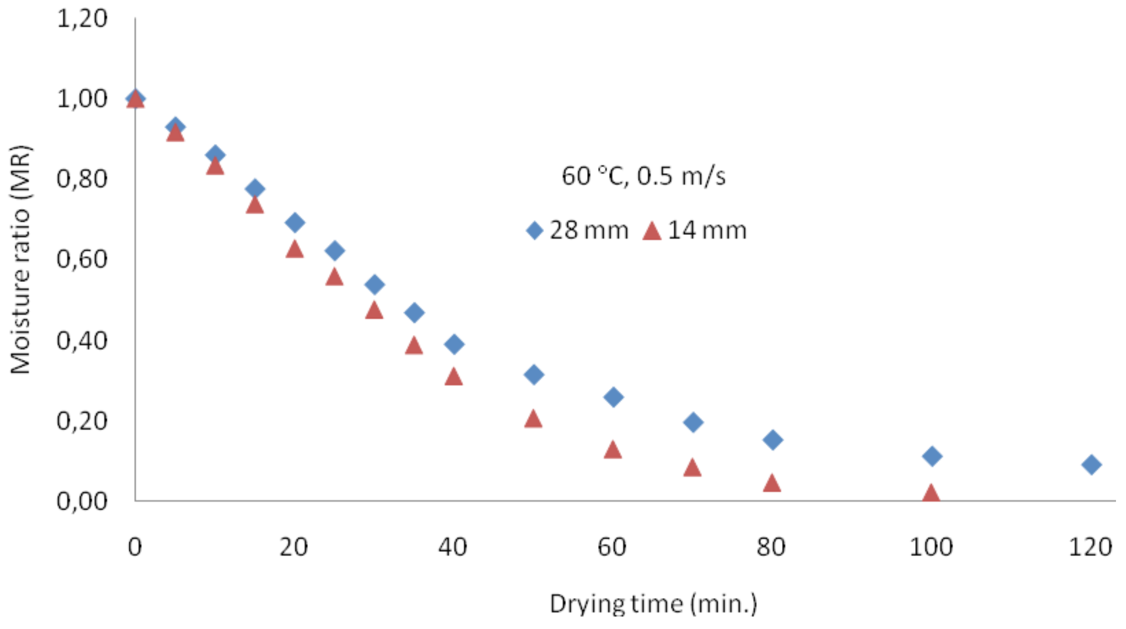

Figure 4: Variation of Moisture ratio with Drying Time at Different Bed Heights.

The effect of bed heights on drying behaviors is presented in figure 4. The lower value for bed height is to provide shorter drying time. However, the drying rates for bed heights are nearly equal and very low about in 120 minutes.

(2) Modelling of Drying Curves

The data of MR obtained from the drying experiments were fitted with 4 mathematical models listed in Table 1.

Table 1: Mathematical models applied to drying curves in this work

#	Model Name	Model	References
1	Newton	$MR = \exp(-kt)$	(Togrul and Pehlivan 2003)
2	Henderson and Pabis	$MR = a\exp(-kt)$	(Henderson and Pabis 1961)
3	Logarithmic	$MR = a\exp(-kt) + c$	(Midilli and Kücük 2003)
4	Two term	$MR = a\exp(-k_0 t) + b\exp(-k_1 t)$	(Henderson 1974)

The regression analysis was performed using the MATLAB software. The values of R^2, SSE and RMSE were determined by non-linear regression analysis for different air temperatures (40 °C, 60 °C and 80 °C). The calculated values were presented in Table 2.

Table 2; Selected drying models for describing black cumin seeds drying data

General model	T °C	Coefficients	Goodness of fit R^2	SSE	RMSE
Newton	40	k = 0.0126	0.9864	0.001034	0.02821
	60	k =0.02134	0.9885	0.01329	0.03197
	80	k =0.03247	0.9908	0.01234	0.0308
Henderson and Pabis	40	a = 1.041, k = 0.01355	0.9934	0.005034	0.02048
	60	a = 1.048, k = 0.02273	0.9933	0.00704	0.02534
	80	a = 1.039, k = 0.03393	0.993	0.009327	0.02788
Logarithmic	40	a = 1.251, c = -0.2251, k=0.009923	0.996	0.003016	0.01656
	60	a = 1.095, c = -0.05732, k=0.02038	0.9943	0.006535	0.02437
	80	a = 1.062, c = -0.03126, k=0.03162	0.9939	0.008141	0.0272
Two term	40	a=-0.06169, b=1.062 k_0=4.854, k_1=0.01401	0.9967	0.002488	0.01577
	60	a=-0.1572 , b=1.154, k_0=0,1459, k_1=0.02533	0.9985	0.001785	0.01336
	80	a=-0,08342 , b=1.083 , k_0=4.954, k_1=-0.03559	0.9954	0.006076	0.02465

As can be seen in Table 2, the two term model has a good agreement with the experimental data and gave the best results for BCs. The R^2, SSE and RMSE values range from 0.9954 to 0.9985, 0.001785 to 0.006076 and 0.01336 to 0.02465, respectively. Figure5 shows the graph of variation of experimental and predicted moisture ratio by two term model with drying time at different temperatures and an air velocity of 0.5 m/s. As can be seen in Figure 5, two term model has a good agreement with the experimental data for different temperatures.

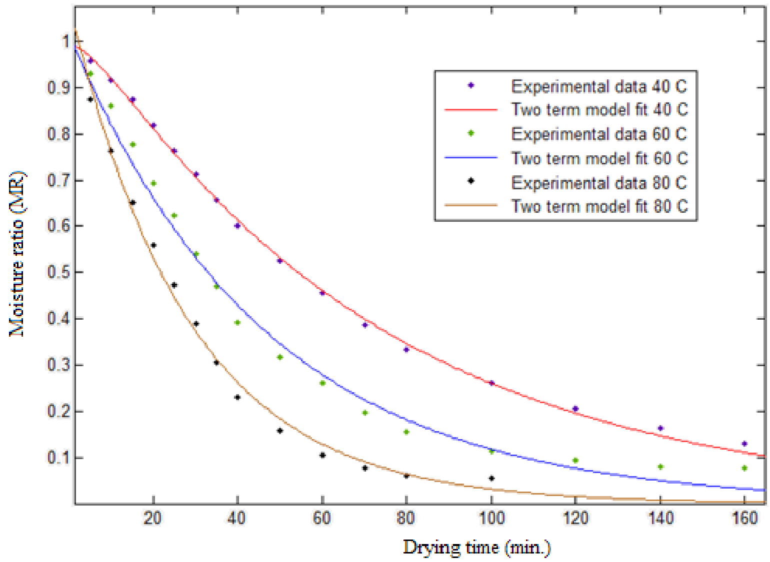

Figure 5: Variation of Experimental and Predicted Moisture Ratio with Drying Time by Two Term Model.

Figure 6 shows the graph of drying rate versus drying time depending on different air temperatures and constant air velocity (0.5 m/s). As can be seen in fig.6, the values of drying rate at 80 °C are higher than 60 and 40 °C, respectively and decrease with time. It increases with the increase in air temperature. About 120 minutes, the drying rates are very low for all temperatures.

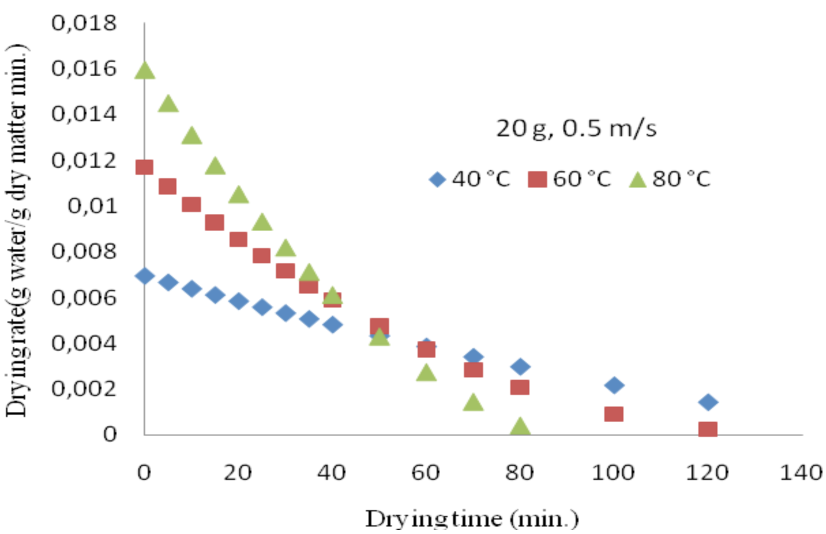

Figure 6: Variation of Moisture ratio with Drying Time at Different Temperatures.

Figure 7 shows the graph of drying rate versus drying time depending on different bed heights at air temperature of 60 °C and air velocity of 0.5 m/s. As can be seen in figure. 7, the drying rate depending on bed height (14 mm) is faster than bed height (28 mm) during the drying period. The drying rates are equal to each other after 120 minutes. During the drying period, there is no constant rate period. All the drying rate process is seen to occur in falling rate period. It is clear that the drying rate decreases continuously with drying time.

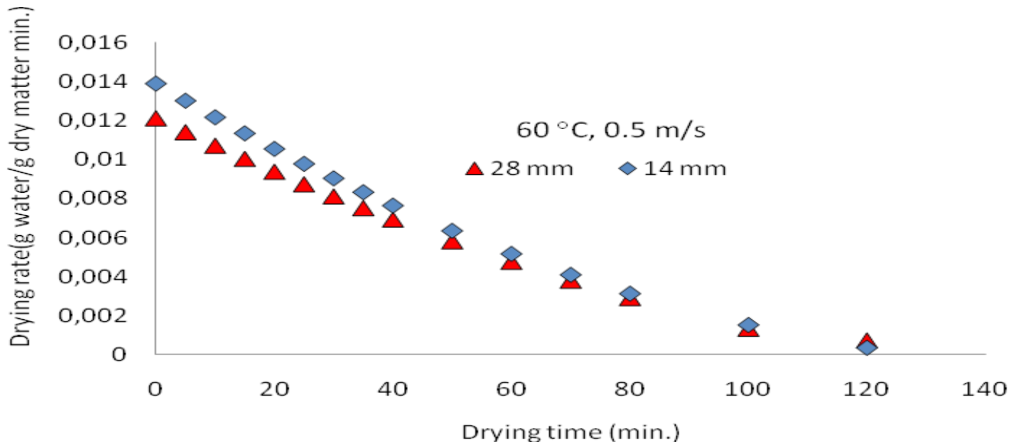

Figure 7: Variation of Moisture ratio with Drying Time at Different Bed Heights.

The effective moisture diffusivity and activation energy were calculated from the slope (figure 8). The value of activation energy depending on different temperatures was calculated as 19.92 kJ/mol and the effective moisture diffusivity values were calculated between 2.85×10^{-10} and 6.77×10^{-10} m^2/s.

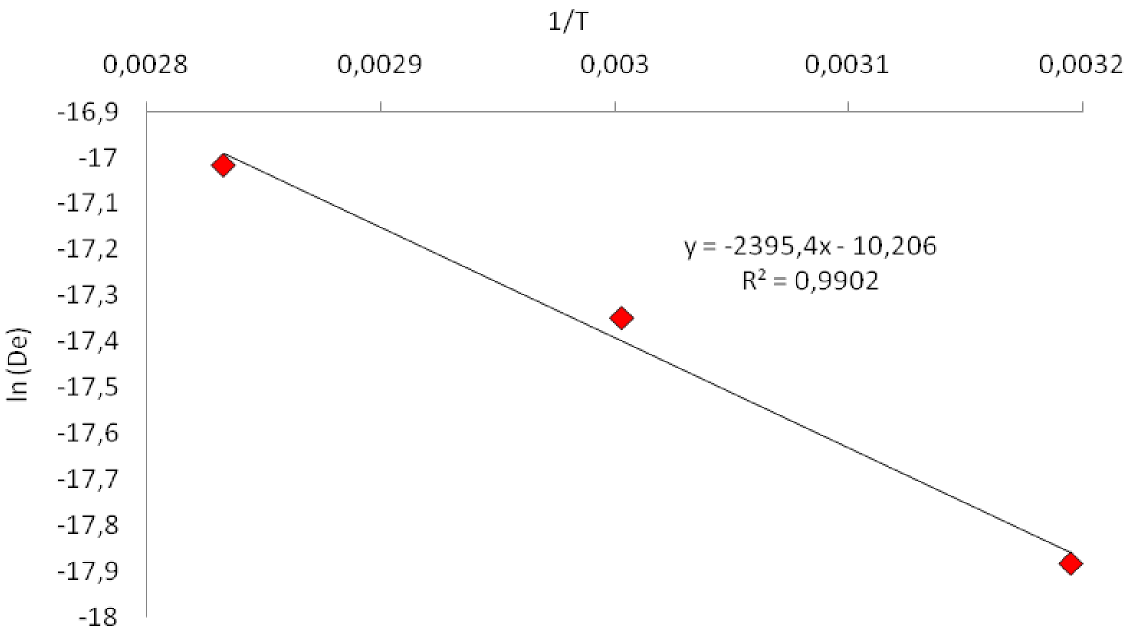

Figure 8: ln (De) Versus 1/T as Function Different Temperatures

Figure9 shows the TG plots in nitrogen atmosphere of the BCs of heating rate of 5 °C min^{-1} and the typical TG-DTA curves. The probable decomposition or drying process of the BCs has been determined by using TG technique. As can be seen in fig.9, the thermal decomposition of BCs was realized in one step at the temperature range of 25-250 °C. The observed mass loss (56.74 % (d.b)) of this drying stage is attributed to the removing of water in the structure of BCs. Also the DTA curve is identical to the TG curve and in both curves for heating rate of 5 °C/min do not provide the formation of any intermediate stage in the process of drying. The corresponding mass loss obtained by TG in this stage is consistence with the mass loss in the initial MC determined at the temperature of 105 °C.

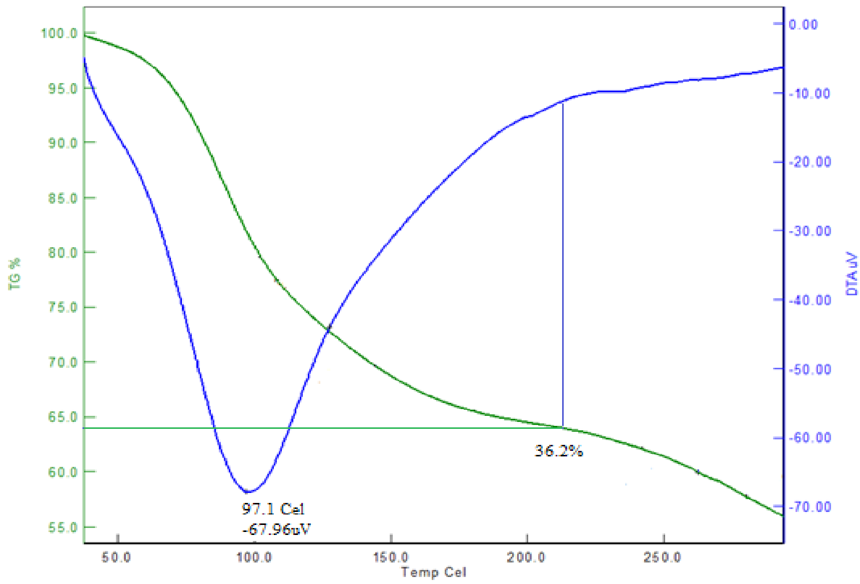

Figure 9: The Typical TG-DTA Curves in Nitrogen Atmosphere of the BC Seeds in Terms of Heating Rate of 5 °C/min.

Conclusions

The drying characteristics of BCs were investigated in a newly designed fixed bed drying system at different temperatures of drying air (40, 60 and 80 ºC) and air velocities (0.25, 0.5 and 1 m/s). Also, the behaviours of BCs were investigated in a thermo gravimetric analysis system in nitrogen flow of 0.850 ml/s and of constant heating rate of 5 °C/min towards 250 °C. The obtained results are summarized as follows:

(i) The initial moisture content of BCs was determined as 56.74% (d.b) in the high-temperature oven at 105 ºC for 6 hours.

(ii) The mass loss obtained by TG is consistence with the mass loss in the initial MC determined at the temperature of 105 °C.

(iii) TG corresponds to drying behaviours of BCs in nitrogen flow of 0.850 ml/s and of constant heating rate of 5 °C/min towards 220 °C.

(iv) Increasing the temperature gradually increases the MR of BCs for the same period of drying time and decreases the total drying time since heat transfer is increased.

(v) The drying rate in shorter bed height is faster than in higher bed height during the drying period.

(vi) Two term models showed a good agreement with the experimental data with higher coefficient of determination value.

(vii) The R^2, SSE and RMSE values range from 0.9954 to 0.9985, 0.001785 to 0.006076 and 0.01336 to 0.02465, respectively.

(viii) All the drying rate process was seen to occur in falling drying rate period.

(ix) The activation energy was calculated as 19.92 kJ/mol and also effective diffusivity values were calculated between 2.85×10^{-10} and 6.77×10^{-10} m^2/s depending air temperatures.

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