# An experimental investigation on drying kinetics of calcium carbonate

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HIGHLIGHTS

- Drying behavior of calcium carbonate was investigated experimentally.

- Drying kinetics was modeled by several empirical correlations.

- Effective moisture diffusion coefficient was calculated in different temperatures.

GRAPHICAL ABSTRACT
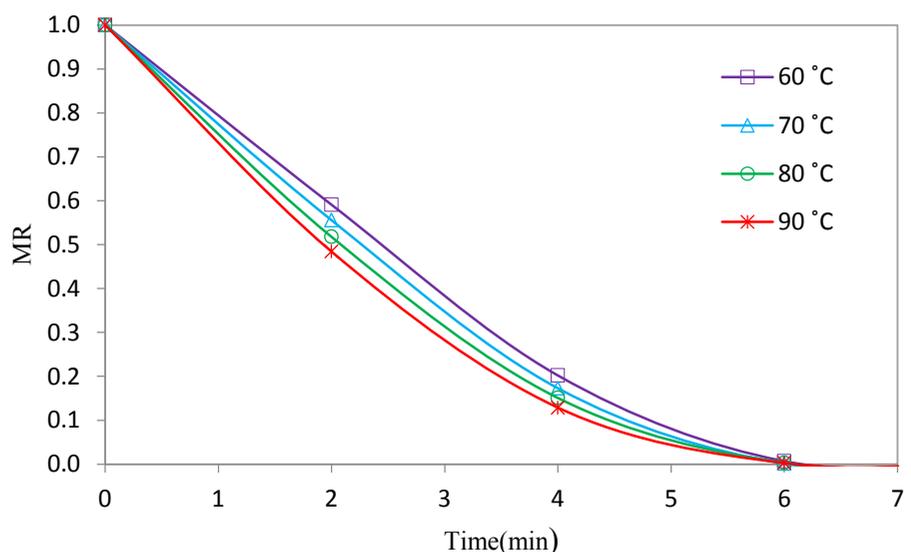
ABSTRACT

Calcium carbonate is a versatile mineral with diverse industrial applications. Drying is a necessary processing step in preparation of calcium carbonate product. Deep knowledge of drying kinetics is required in order to design an appropriate dryer. The main aim of the present study was to investigate the drying kinetics of calcium carbonate, experimentally. Thirteen empirical correlations with temperature-dependent constants were used to describe the thin-layer drying behavior of calcium carbonate. The correlation fitting results revealed that all examined empirical correlations were acceptable. But, the Page correlation was in the best agreement with experimental data. The effective moisture diffusivity was calculated within $8.61 \times 10^{-9}$ to $10.40 \times 10^{-9} m^2 s^{-1}$ in the temperature range of 60 to 90°C. The drying activation energy was also calculated to be 6.306 kJmol$^{-1}$. The fitted correlations for the drying kinetics of calcium carbonate can be used to design an industrial dryer and also to estimate drying time in various drying conditions.

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# 1. Introduction

Calcium carbonate is one of the most versatile minerals with very diverse industrial applications in the chemical [1], pharmaceutical [2, 3], food[4], plastics [5, 6], paper [7], paints [8], ceramic materials [9, 10], and etc. These applications made calcium carbonate an important inorganic chemical in industries.

There are several methods for calcium carbonate production such as (1) intensive milling or grinding of natural calcium carbonate (GCC) (2) reaction of aqueous calcium hydroxide suspension with carbon dioxide leading to the production of precipitate calcium carbonate (PCC). The product is conventionally dewatered and dried in a known manner [11-13]. For instance, the second production method has four processing steps including: (1) calcination, (2) lime slaking, (3) carbonation, (4) drying [14]. Therefore, drying operation is necessary in preparation of final market product. In some applications, the nanoparticles of calcium carbonate as a final product is required [5, 15]. The selection and design of a suitable dryer is dependent on the required form of final product and drying kinetics of calcium carbonate [16].

Thus, deep knowledge of drying kinetics is required in order to design an appropriate calcium carbonate dryer [16]. The drying kinetics of calcium carbonate can be specified by the related transport properties such as effective moisture diffusivity and empirical correlations commonly used to estimate drying time in various drying conditions [17].

There are several empirical correlations describing the thin layer drying kinetics [18-22]. In the empirical correlations, it is assumed that the moisture in the particle surface reaches in equilibrium with the surrounding air, immediately [23-25].

The main purposes of the present work were:

(1) To determine drying kinetics data of calcium carbonate in an oven dryer at different temperatures, experimentally.

(2) To model drying kinetics of calcium carbonate using empirical correlations with temperature-dependent constant.

(3) To determine effective moisture diffusivity and activation energy of calcium carbonate drying.

# 2. Experimental

## 2.1. Materials and Methods

Calcium Carbonate particles were provided from local corporation (Zagros Powder Co., Iran). In the beginning, 15 g of 20 wt% calcium carbonate slurry was filtered by Buchner funnel that connected to the vacuum pump for 5 min duration. Then, the thin layer of pastelike filter cake with 2 mm thickness was shed in the crystallizing dish and weighted. The area of crystallizing dish was approximately 0.00283 $m^2$. Afterwards, the crystallizing dish was put in the oven. The oven temperature was set in each experiment. The temperature was controlled by adjusting the air flow in the oven convection channel. Drying experiments were conducted at different four temperatures (i.e., 60, 70, 80, and 90°C).

In each experimental run, the weight loss of solid sample was measured every two minutes. The experimental run was terminated when the sample weight became constant between two measurements. Afterwards, the crystallizing dish was put in the oven at 110°C for 24 h in order to find the sample dry weight. It is important to note that for each data point, the experimental runs were repeated three times and the average value was reported. Then, the solid moisture ratio (MR) was calculated as follows:

$$MR = \frac{X - X_{eq}}{X_0 - X_{eq}} \tag{1}$$

Where, MR, X, $X_0$ and $X_{eq}$ are the moisture ratio, the solid moisture content at time $t$, initial, and equilibrium moisture content, respectively. Moisture content of calcium carbonate was calculated according to dry basis (d.b.) with the following equation:

$$X_{db} = \frac{w_t - w_k}{w_k} \tag{2}$$

Where, $w_t$ is the sample weight at specific time, $w_k$ is the dry sample weight.

It should be noted that $X_{eq}$ was assumed to be zero because the values of equilibrium moisture content are negligible in comparison with X and $X_0$ [26, 27]. Therefore, the moisture ratio was simplified to X/$X_0$ instead of the Eq. (1). The variation of MR versus drying time was considered according to the obtained experimental data.

It should be noted that all experimental tests were carried out in one day with the same ambient moisture of air. So, in the present study, the effect of ambient moisture of air can be neglected.

## 2.2 Mathematical modeling on drying kinetics

In order to describe relationships between moisture ratios with time, different empirical correlations have been proposed. Thirteen empirical correlations were used in order to describe the drying kinetics under isothermal conditions. These correlations are as follows:

Lewis correlation[28]:

$$MR = exp(-kt) \tag{3}$$

Henderson and Pabis correlation[29]:

$$MR = a.exp(-kt) \tag{4}$$

Page correlation[30]:

$$MR = exp\left(-kt^n\right) \tag{5}$$

First modified Page correlation[31]:

$$MR = exp\left(-(kt)^n\right) \tag{6}$$

Logarithmic correlation[32]:

$$MR = a.exp(-kt) + b \tag{7}$$

Midilli and Kucuk correlation[33]:

$$MR = a.exp\left(-kt^n\right) + bt \tag{8}$$

Balbay and Sahin correlation[24]:

$$MR = (1-a)exp\left(-kt^N\right) + b \tag{9}$$

Two-term correlation[34]:

$$MR = a_1.exp(-k_1t) + a_2.exp(-k_2t) \tag{10}$$

Two term exponentialcorrelation[35]:

$$MR = a.exp(-kt) + (1-a).exp(-k.a.t) \tag{11}$$

Diffusion approach correlation[36]:

$$MR = a.exp(-kt) + (1-a).exp(-kbt) \tag{12}$$

Verma et al. correlation[37]:

$$MR = a.exp(-k_1t) + (1-a).exp(-k_2t) \tag{13}$$

Simplified Fick's diffusion correlation[38]:

$$MR = a.exp\left(-k\left(\frac{t}{L^2}\right)\right) \tag{14}$$

Second modified Page correlation[38]:

$$MR = a.exp\left(-k\left(\frac{t}{L^2}\right)^n\right) \tag{15}$$

In order to take into account the effect of temperature on drying kinetics in these correlations, an Arrhenius type is considered between k constant and temperature[39]:

$$k = k_0.exp(-E/RT) \tag{16}$$

The regression analysis was carried out using Levenberg-Marquardt algorithm (LMA), also known as the damped least-squares (DLS) method [40]. This method is used to solve non-linear least squares problems[40]. The LMA interpolates between Gauss–Newton algorithm (GNA) and gradient descent method [40]. Three statistical criteria such as the coefficient of determination ($R^2$), root mean square error (RMSE), and Chi-square ($\chi^2$) were applied to evaluate the adequacy of fitted correlations under different conditions:

$$SS_{error} = \sum_{i=1}^{N}\left(M_{exp,i} - M_{pre,i}\right)^2 \tag{17}$$

$$SS_{tot} = \sum_{i=1}^{N}\left(M_{exp,i} - \overline{M}_{exp,i}\right)^2 \tag{18}$$

$$R^2 = 1 - \frac{SS_{error}}{SS_{tot}} \tag{19}$$

$$RMSE = \left[\frac{1}{N}\sum_{i=1}^{N}\left(M_{exp,i} - M_{pre,i}\right)^2\right]^{1/2} \tag{20}$$

$$\chi^2 = \frac{\sum_{i=1}^{N}\left(M_{exp,i} - M_{pre,i}\right)^2}{N-Z} \tag{21}$$

Where $M_{pre,i}$, $M_{exp,i}$, $N$, and $z$ are predicted moisture ratio, experimental moisture ratio, number of experimental data, and the number of constants in the drying model, respectively. It should be noted that the fitted correlations with higher values of coefficient of determination and lower values of the root mean square error, and chi-square were preferred[41, 42].

## 2.3. Effective moisture diffusivity and activation energy

The Fick's second law (Eq. 22) is a well-known mass diffusion equation applied to describe the drying kinetics.

This model is based on the following assumptions[43]:

• The moisture is transferred from inside to the surface of by diffusion mechanism.
• The moisture evaporation is carried out on the surface of particles.
• All particles are in the same size.
• The physical properties of solid particles do not change with time.
• The temperature and moisture content of all the particles are the same.
• Shrinkage and temperature gradient in the solid particles are neglected.
• Diffusion coefficient is constant.

$$\frac{\partial X}{\partial t} = D_{eff} \frac{\partial^2 X}{\partial z^2} \tag{22}$$

Initial and boundary conditions are as follows:

$$t = 0; \; 0 < z < L; \; X = X_0 \tag{23}$$

$$t > 0; \; z = 0; \; \frac{\partial X}{\partial z} = 0 \tag{24}$$

$$t > 0; \; z = L; \; X = X_{eq} \tag{25}$$

In the present case, the slab geometry is considered. According to the initial condition, it is assumed that the moisture is distributed in a uniform manner throughout the sample at the beginning of drying. According to the first boundary condition, it is assumed that the mass transfer is carried out in a symmetric domain. According to the second boundary condition, it is assumed that the sample surface is in equilibrium with the surrounding air. In other words, the mass transfer resistance on the sample surface is neglected. The analytical solution of the above-mentioned partial differential equation for the slab geometry has been presented by Crank[44]:

$$MR = \frac{8}{\pi^2} \sum_{n=0}^{\infty} \frac{1}{(2n+1)^2} exp\left(-\frac{(2n+1)^2 \pi^2 D_{eff} \; t}{4L^2}\right) \tag{26}$$

Where $D_{eff}$, $L$, and t are the effective moisture diffusivity, the thickness of the drying layer, and drying time, respectively. For each temperature, the effective moisture diffusivity was calculated by considering experimental drying data versus drying time. Eq. (26) can be simplified as follows considering only the first term for thin-layer drying (by setting n=0):

$$MR = \frac{8}{\pi^2} exp\left(-\frac{\pi^2 D_{eff} \; t}{4L^2}\right) \tag{27}$$

It should be noted that the first term has the highest contribution in MR in comparison with the other terms of eq. 26. The effective moisture diffusivity depends on the temperature by simple Arrhenius equation [45]:

$$D_{eff} = D_0 \; exp\left(\frac{-E_a}{RT}\right) \tag{28}$$

Where $D_0$, $E_a$, and $R$ are the pre-exponential factor, the activation energy, and the universal gas constant, respectively. The activation energy can be easily determined through the following linear fitting:

$$ln\left(D_{eff}\right) = ln\left(D_0\right) - \frac{E_a}{R}\left(\frac{1}{T}\right) \tag{29}$$

## 3. Results and Discussion

### 3.1. Drying kinetics analysis

The drying correlations (eq. 3 to eq. 15) were fitted to the experimental data one by one and the correlation parameters were determined by regression analysis. Figs. 1-2 show the drying of calcium carbonate samples under isothermal condition.
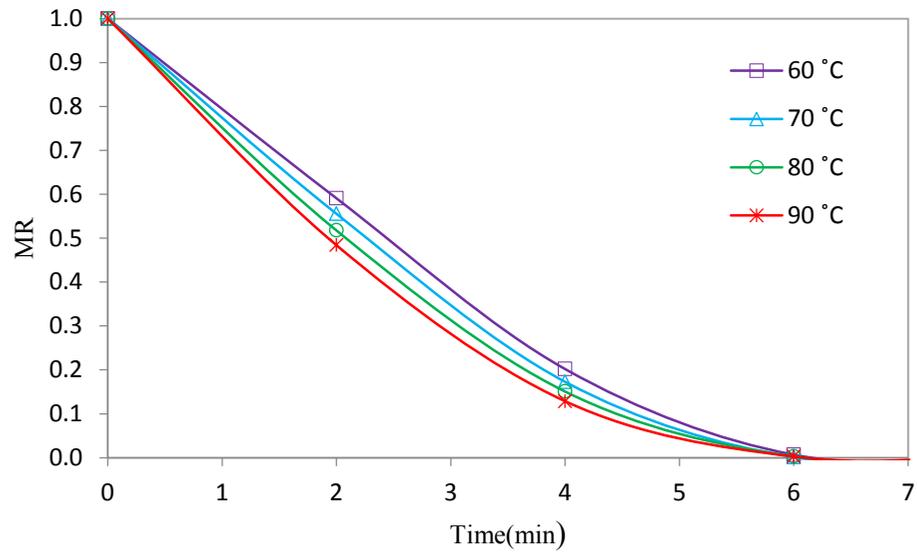
**Fig. 1.** Variation of moisture ratio versus drying time for calcium carbonate samples at different drying temperature.
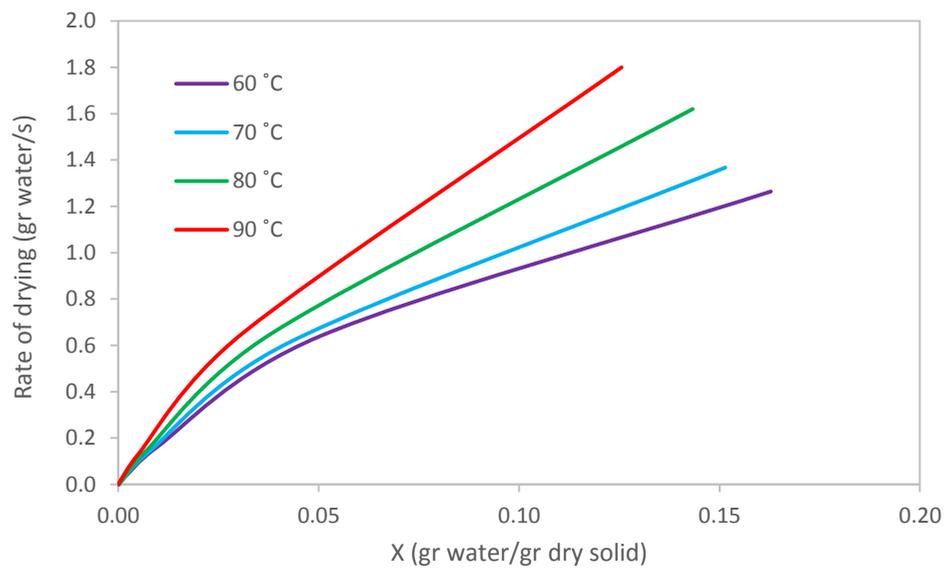


**Fig. 2.** Variation of drying rate versus moisture content of calcium carbonate samples at different drying temperature.

As expected from these drying curves, the drying rate and required drying time are affected by temperature, significantly. It should be noted that higher amount of moisture can be removed in high temperature drying because temperature is the main driving force for moisture evaporation. As can be observed in Fig. 2, the solid moisture content and the drying rate are high at the beginning of the drying process. The moisture content and the drying rate were gradually decreased with the drying time. The main portion of the sample moisture is removed in the early stages of drying, but the removal of the remaining moisture is carried out in a longer time period.

In the drying process, the amount of evaporated moisture on the sample surface is influenced by the internal mass transfer resistance related to liquid and vapor diffusion, and capillary forces. By passing drying time, the particle surface moisture is evaporated and the internal moisture starts to evaporate with lower rate due to the heat and mass transfer limitations. The parameters of different correlations and the results of fitting statistical analysis are summarized in Table 1. It should be noted that all the experimental data points obtained in different temperature were used for fitting of each empirical correlation.

**Table 1.**

Fitting statistical results and parameters of the examined empirical correlations for calcium carbonate drying[a].

| Models | $k_{01}$ (s$^{-1}$) | $k_{02}$ (s$^{-1}$) | $E_1$ (kJmol$^{-1}$) | $E_2$ (kJmol$^{-1}$) | $a_1$ | $a_2$ | $b$ | $N$ | $L$ | Number of constants | $R^2$ | RMSE | $X^2$ |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Lewis | 0.057 | — | 6.148 | — | — | — | — | — | — | 1 | 0.9789 | 0.0542 | 0.0026 |
| Henderson and Pabis | 0.057 | — | 6.083 | — | 1.025 | — | — | — | — | 2 | 0.9796 | 0.0543 | 0.0031 |
| Page | 0.006 | — | 9.453 | — | — | — | — | 1.637 | — | 2 | 0.9991 | 0.0114 | 0.0001 |
| First modified Page | 0.045 | — | 5.773 | — | — | — | — | 1.637 | — | 2 | 0.9991 | 0.0114 | 0.0001 |
| Logarithmic | 0.048 | — | 5.835 | — | 1.059 | — | -0.036 | — | — | 3 | 0.9833 | 0.0501 | 0.0031 |
| Midilli and Kucuk | 0.006 | — | 9.436 | — | 0.999 | — | -2.47e$^{-7}$ | 1.633 | — | 4 | 0.9991 | 0.0118 | 0.0002 |
| Balbay and Sahin | 0.006 | — | 9.328 | — | -0.004 | — | -0.004 | 1.621 | — | 4 | 0.9992 | 0.0115 | 0.0002 |
| Two term | 0.009 | 0.011 | 2.557 | 2.926 | -5.223 | 6.243 | — | — | — | 4 | 0.9880 | 0.0444 | 0.0028 |
| Two term exponential | 0.076 | — | 5.422 | — | 2.378 | — | — | — | — | 2 | 0.9982 | 0.0160 | 0.0002 |
| Diffusion approach | 0.096 | — | 5.579 | — | 6.958 | — | 1.228 | — | — | 3 | 0.9985 | 0.0148 | 0.0022 |
| Verma et al. | 0.030 | 0.028 | 5.804 | 5.880 | 7.999 | — | — | — | — | 3 | 0.9877 | 0.0440 | 0.0022 |
| Simplified Fick's diffusion | 0.064 | — | 6.087 | — | 1.025 | — | — | — | 1.063 | 3 | 0.9796 | 0.0554 | 0.0038 |
| Second modified Page | 0.006 | — | 9.337 | — | — | — | — | 1.636 | 1.001 | 4 | 0.9991 | 0.0117 | 0.0001 |

a.The fitted coefficient can be used in the correlation with time in second unit.

As can be observed, the fitting results are acceptable for all correlations but the best correlations describing the drying kinetics of calcium carbonate should be chosen as the correlation with the highest value of $R^2$ and lowest values of $X^2$, and RMSE. In this regard, Page, first modified Page, Midilli and Kucuk, Balbay and Sahin, and Diffusion approach correlations are in better agreement with experimental data in comparison with the other correlations. The experimental moisture ratio versus the predicted values using Page correlation is presented in Fig. 3.

This graph confirms the validity of the selected correlation, again.

### 3.2 Effective moisture diffusivity ($D_{eff}$) and activation energy

As can be observed in Figures 1-2, the internal moisture transfer mechanism dominates the drying process. According to the eq. 27, effective moisture diffusivity can be obtained by fitting MR versus drying time. Table 2 shows the calculated values of effective moisture diffusivity in different temperature.
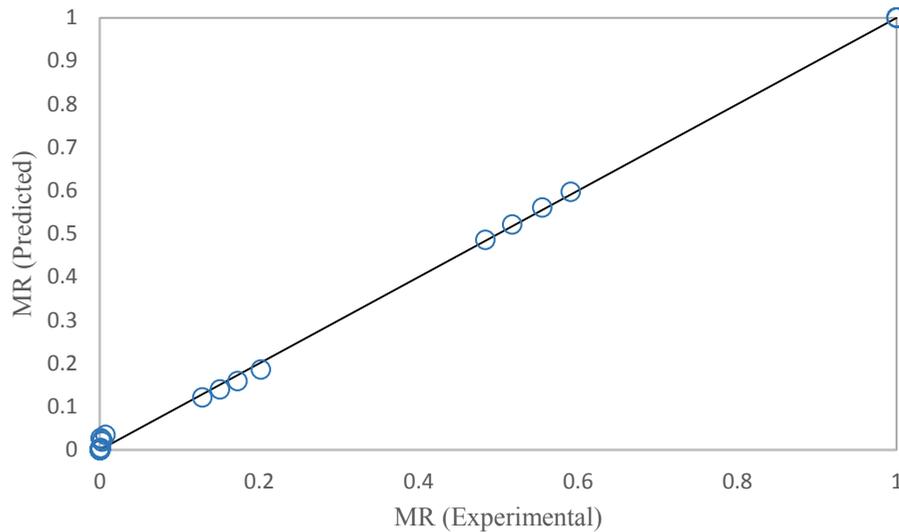


**Fig. 3.** Comparison of predicted moisture ratio using Page correlation versus experimental moisture ratio at different temperatures.

**Table 2.**
Effective moisture diffusivity in different temperatures.

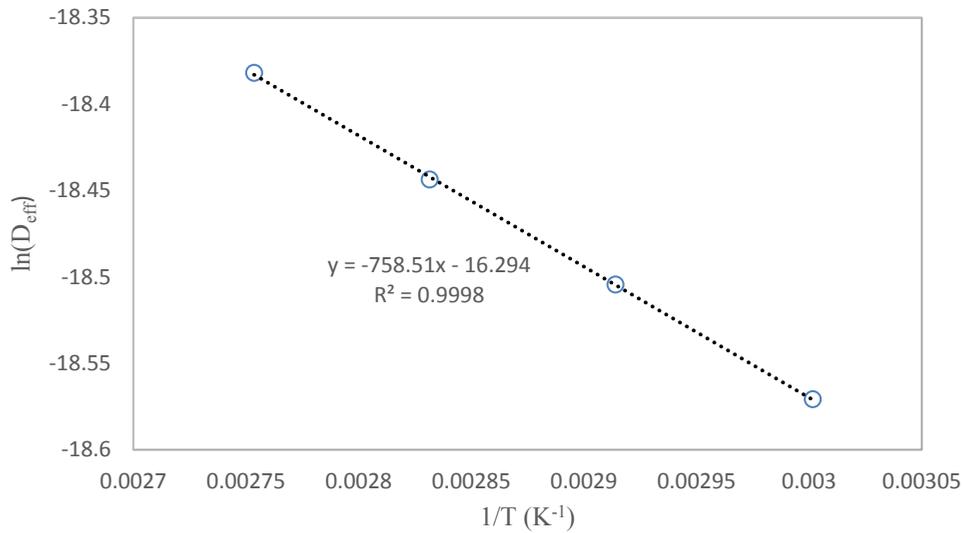| Temperature (°C) | $D_{eff}$ ($10^{-9}$m$^2$s$^{-1}$) | $R^2$ |
|:---:|:---:|:---:|
| 60 | 8.61 | 0.9144 |
| 70 | 9.20 | 0.9213 |
| 80 | 9.77 | 0.9297 |
| 90 | 10.40 | 0.9353 |

**Fig. 4.** Arrhenius-type relationship between the effective moisture diffusivity and temperature.

Activation energy was determined using Eq. (28). Fig. 4 shows the plot of $\ln(D_{eff})$ versus the reciprocal of temperature (1/T). The values of activation energy and Arrhenius factor ($D_0$) were found to be 6.306 kJ mol$^{-1}$ and 8.387×10$^{-8}$ m$^2$ s$^{-1}$, respectively.

## 4. Conclusions

In the present study, drying kinetics of calcium carbonate was studied under isothermal conditions. Several available drying correlations were examined by fitting the experimental data. It was found that Page correlation is in the best agreement with the experimental data. The values of effective moisture diffusivity of calcium carbonate were found to be within 8.61×10$^{-9}$ to 10.40 ×10$^{-9}$ m$^2$s$^{-1}$ in temperature range of 60 to 90 ˚C. The effective moisture diffusivity values of calcium carbonate were increased by an increase in the temperature. The order of reported values of moisture diffusivity of calcium carbonate were similar to those reported in the literature for other mineral materials such as 2.2337×10$^{-10}$ to 22.0648×10$^{-10}$ m$^2$ s$^{-1}$ for sodium percarbonate drying at 60-80 ˚C[45]; 1.26×10$^{-9}$ to 1.71×10$^{-9}$ m$^2$ s$^{-1}$ for paper sludge drying at 180-240 ˚C[46]; 4.5×10$^{-9}$ to 6.7×10$^{-9}$ m$^2$ s$^{-1}$ for cellular concrete drying[47]; 2.0×10$^{-9}$ to 3.5×10$^{-9}$ m$^2$ s$^{-1}$ for lime binder drying[47]; 1.90×10$^{-9}$ to 3.05× 10$^{-9}$ m$^2$ s$^{-1}$ for masonry clay drying[48]; 1.481 ×10$^{-8}$ to 5.098×10$^{-9}$ m$^2$ s$^{-1}$ for lignite drying[49]; Temperature dependence of the effective moisture diffusivity was considered using the Arrhenius relationship. The activation energy and Arrhenius factor for moisture diffusion is found to be 6.306 kJ mole$^{-1}$ and 8.387×10$^{-8}$ m$^2$ s$^{-1}$, respectively.

## Nomenclature

**Symbol**

| | |
|---|---|
| $\chi^2$ | Reduced chi-square |
| $R^2$ | Coefficient of determination |

**Letter**

| | |
|---|---|
| $a, b, c, g, k0, n$ | Correlation parameters |
| $D$ | Diffusion coefficient, m$^2$s$^{-1}$ |
| $Ea$ | Activation energy, kJ mole$^{-1}$ |
| $L$ | Half thickness, m |
| $MR$ | Moisture ratio |
| $N$ | Number of observation |
| $R$ | Universal gas constant, 8/314 J K$^{-1}$ mole$^{-1}$ |
| $REMS$ | Root mean square error |
| $t$ | Drying time |
| $T$ | Drying temperature |
| $X$ | Moisture content, gwater gdry solid$^{-1}$ |
| $z$ | Number of constants in the correlations |

**Subscript**

| | |
|---|---|
| $d$ | Dry weight |
| $eff$ | Effective |
| $eq$ | Equilibrium |
| $exp$ | Experimental |
| $pre$ | Predicted |

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