

Determination of equilibrium isotherms and proper mathematical model for lime slices

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Abstract: Equilibrium moisture content (EMC) at different air relative humidity and temperatures is a vital parameter in optimization of drying and storage processes. In the present study amount of moisture content in lime (Local variety) slice at different ambient temperatures of 30 °C, 40 °C and 50 °C were measured using gravimetric method. The obtained E.M.C experimental values were employed to plot the isotherm curve at different environmental air temperatures. These data also were fed to fourteen popular mathematical models. The well-known statistical factors such as Coefficient of determination (R^2), Chi-square (χ^2), Root of mean square error (RMSE) and Mean of relative deviation (MRD) were calculated to validate the goodness of fit. The Peleg Model was selected as the best model for predicting the EMC at each three temperatures level. The result of present research displayed that the amount of EMC decreased with increasing the ambient temperature. The monolayer moisture content (m_0) was also determined using Brunauer, Emmett and Teller (BET) equation. The monolayer moisture content values were 0.023, 0.021 and 0.014 g.H₂O/g solid at 30 °C, 40 °C and 50 °C, respectively, and the corresponding constant values of the Brunauer, Emmett and Teller equation were found to be -4.925, -6.543 and -7.035, respectively.

Keywords: lime slice, equilibrium moisture content, modeling, monolayer

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1 Introduction

Lime (*Citrus aurantifolia*) has a lot of pharmaceutical and alimentary properties. In order to control the drying process and storage conditions, besides the knowledge of thin layer drying equations, the data on physical properties including equilibrium moisture content are needed which may be expressed in moisture desorption isotherms. To optimize storage stability, knowledge of the relationship between the equilibrium moisture content (EMC) in the plant material and the relative humidity (RH) of the surrounding air at the given temperature is required to avoid the deterioration by microbial and enzymatic activity (Karel and Yong, 1981). The moisture sorption isotherm of food graphically relates its equilibrium moisture content in either desorption or adsorption to the water activity (a_w) at a definite temperature. This

knowledge is required to stop the drying process at the aimed moisture content in order to save energy (Hamer et al., 2000). The EMC value may be related to air temperature. The effect of air temperature rise was investigated by Zomorodian and Tavakoli (2007). Fourteen mathematical models have been proposed for describing the water sorption of various foods and agriculture materials. Some of the mostly applied equations are Caurie, Chung-Pfost, GAB, Halsey, Henderson, Kuhn, Modified BET, Modified Chung-Pfost, Modified Halsey, Modified Henderson, Modified Oswin, Modified Smith, Oswin and Peleg. Researches on the temperature dependence of isotherms and mathematical models to represent desorption isotherms have been extensively reported in literatures (Zomorodian, 1979; Van den Berg and Bruin, 1981; Lee and Lee, 2008; Janjai et al., 2009; Al-Muhtaseb et al., 2010; Zomorodian et al., 2011; Argyropoulos et al., 2012). Lahsasni et al. (2002) found the BET and Henderson models as the most suitable for describing the sorption curves for prickly pear peel.

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They used the BET equation of monolayer moisture content values for the sorption at different temperature for determining the pertinent parameter for the product and optimum overall stability at the monolayer constant values. The desorption isotherms data are useful in the drying analysis while the adsorption isotherms data can be used for establishing a storage method (Cordeiro et al., 2006). The moisture sorption isotherm data of walnut kernels were determined at three different temperatures (25 °C, 35 °C and 45 °C) and of the models tested, the Peleg model gave the best fit to experimental data and also The BET equation was applied to the monolayer moisture content (Tog̃rul and Arslan, 2007). Using the static gravimetric method at three temperatures (40, 50 and 60 °C) and in wide range of water activity (0.109 – 0.891) desorption and adsorption data of orange peel and leaves were best fitted by the Peleg model (Bejar et al., 2012). Desorption and adsorption isotherms for *Melissa officinalis* L. (Lemon Balm) were determined at four different temperatures (25 °C, 40 °C, 50 °C and 60 °C), Experimental data were fitted using the mathematical models. Halsey's model was found to be the most suitable for describing the desorption curves at the temperatures of 25 and 40 and BET's model was most suitable for describing the data at the temperatures of 50 °C and 60 °C (Cuervo-Andrade and Hensel, 2013). The adsorption and desorption equilibrium moisture content/equilibrium relative humidity relationships of sunflower seeds with four different oil contents were obtained and the Modified Halsey model was the best models for predicting equilibrium moisture content/equilibrium relative humidity relationships (Macielet al, 2015). The monolayer moisture content shows the amount of water that adsorbed by a single layer to binding sites in the material. The water binding properties of active points in roasted seeds were probably reduced; hence, some water sorption active points disappeared (Martinez and Chiralt, 1996). Not much information is available about desorption isotherms of lime slice. As desorption isotherm is one of the basic

parameters of the drying process, this work will give an important contribution to the optimization of the drying of lime slice. The other objective was to determine desorption parameters such as monolayer moisture content values.

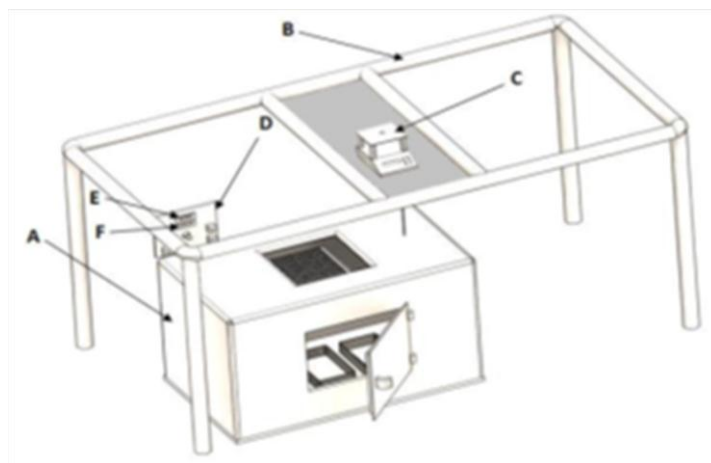
2 Materials and methods

2.1 Experimental procedure

The limes with average moisture content of 14.9 (% d.b) was harvested from a garden in Jahrom (Fars province, Iran). To perform the test, lime was become in a thin layer to a thickness of 4 mm by a digital caliper (MarCal 16 ER, Germany; maximum Measuring range of 300 mm; ± 0.01 mm). After sterilization, each of lime slices wrapped in paper towels and was kept in a cool, dry enclosure for preventing spoilage and any changes in the moisture content of the product during the trial. A controlled environment chamber (in terms of temperature and relative humidity) is required. To achieve this objective, a completely enclosed chamber, fully insulated and controlled in terms of thermodynamic conditions was employed (100cm \times 60cm \times 60 cm), Figure 1. In order to reduce the equilibrium time, a fan assisted by 1000 W thermostatic electrical heater was used to change the temperature of the air and circulate atmosphere inside the chamber is being used (Labuza, 1984). To carry out the test, the number of 25 lime slices were placed in a thin layer inside baskets. The basket was hung from the under hook of an electronic balance (GF-600, A&D Instruments Ltd., Oxon, UK, maximum capacity of 610 g; ± 0.001 g). The experiments were performed at three temperatures (30 °C, 40 °C and 50 °C, ± 0.5 °C), and seven air relative humidity (11% to 91%, ± 1 %), using super saturated salt solutions of LiCl, $\text{KC}_2\text{H}_3\text{O}_2$, CaCl_2 , MgCl_2 , KNO_2 , MgNO_3 , NaNO_2 , NaCl and KNO_3 . The experiments were conducted in three replications beginning with the highest environmental relative humidity and lime slices initial moisture content of 14.5 % db. Thus desorption process was continued by changing the proper saturated salt solutions at constant chamber air temperature. In order to

avoid any gain or loss during the sample weighing, the automatic balance was set to weight the samples periodically until the successive weigh records showed no significant changes (± 0.005 g). When equilibrium was

reached (ranging from 3 days to 2 weeks depending on the temperature and relative humidity), the samples were dried using oven method (at 105°C for 24 h) in order to obtain dry matter contents (Park et al., 2002).



A: Chamber test B: Desk C: Digital scale D: Electrical circuit boards
E: Moisture meter display F: Thermostat display

Figure 1 – The equipment for the desorption isotherm measurement

2.2 Data analyses

2.2.1 Determination of desorption isotherm model

The experimental desorption data of all the samples at three different temperatures was fitted to fourteen theoretical and empirical equations (Modified BET, Henderson, GAB, Caurie, Chung and Pfof, Modified Chung and Pfof, Halsey, Cohen, Oswin, Peleg, Modified Oswin, Modified Henderson, Modified Smith, Modified Halsey). Nonlinear regression analysis, using the statistical software SPSS 16.0 and the statistical factors (Coefficient of determination (R^2), chi-square (χ^2), Root of mean square error (RMSE) and Mean of relative deviation (MRD), was used for interpreting the experimental data. The R^2 , χ^2 , RMSE and MRD are defined as (Zomorodian, 1979):

$$R^2 = \frac{\left(\sum_{i=1}^N (MR_{\text{exp},i} - \overline{MR}_{\text{exp}}) (MR_{\text{pre},i} - \overline{MR}_{\text{pre}}) \right)^2}{\sum_{i=1}^N (MR_{\text{exp},i} - \overline{MR}_{\text{exp}})^2 \sum_{i=1}^N (MR_{\text{pre},i} - \overline{MR}_{\text{pre}})^2} \quad (1)$$

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

$$\text{RMSE} = \left[\frac{1}{N} \sum_{i=1}^N (MR_{\text{exp},i} - MR_{\text{pre},i})^2 \right]^{\frac{1}{2}} \quad (3)$$

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

Where EMC_{exp} is the equilibrium moisture content by experiment, EMC_{pre} is the predicted EMC due to models, N is the number of experimental data on relative humidity and n is the number of constants in each model.

2.2.2. Monolayer moisture content

The monolayer moisture content which is of significant importance to the physical and chemical stability of dehydrated materials with regard to lipid oxidation, enzyme activity, non-enzymatic browning, flavour components preservation, and structural characteristics (Labuza et al, 1970; Karel and Yong, 1981), can be determined from the equilibrium desorption

isotherms by means of the two parameter of BET (Brunauer-Emmett-Teller) equation (Brunauer, Emmett and Teller, 1938). To predict the desorption monolayer moisture content at different ambient temperatures, the experimental values of a_w (ERH/100) and m (EMC) were used to find the best fit regression line of the BET monolayer moisture content equation which can be a useful tool in predicting the monolayer value (Zomorodian et al, 2011).

$$\frac{a_w}{m(1 - a_w)} = \frac{1}{m_0 c} + \left(\frac{c - 1}{m_0}\right) a_w \quad (5)$$

Where a_w is water activity (decimal), m is the moisture content (gH₂O/g solid) at a given a_w , m_0 is the monolayer moisture content (gH₂O/g solid), and c is a constant showed the dependence between monolayer moisture content and temperature (Menkov et al. 1999). If a_w values, as independent variables, are plotted versus

the $[aw/m (1-aw)]$ values, as dependent variables, a unique linear regression line diagram displayed having $c-1/m_0c$ as its slope and $1/m_0c$ as its intercept (Pernille, 2007). The influence of temperature on monolayer moisture contentis required to be studied for moisture sorption characteristic of a food (Choudhury et al, 2011).

3 Results and discussion

3.1 Equilibrium isotherms and mathematical

The experiments were conducted on lime slice equilibrium moisture content at three air temperatures and relative humidity of 30 °C, 40 °C and 50 °C and 11% to 91%, respectively. Figure 3 presents the experimental data of desorption of lime slice at three temperatures. These curves provide a good representation of Type II (sigmoidal) according to the BET classification, typical of the most biological products (Brunauer, 1945).

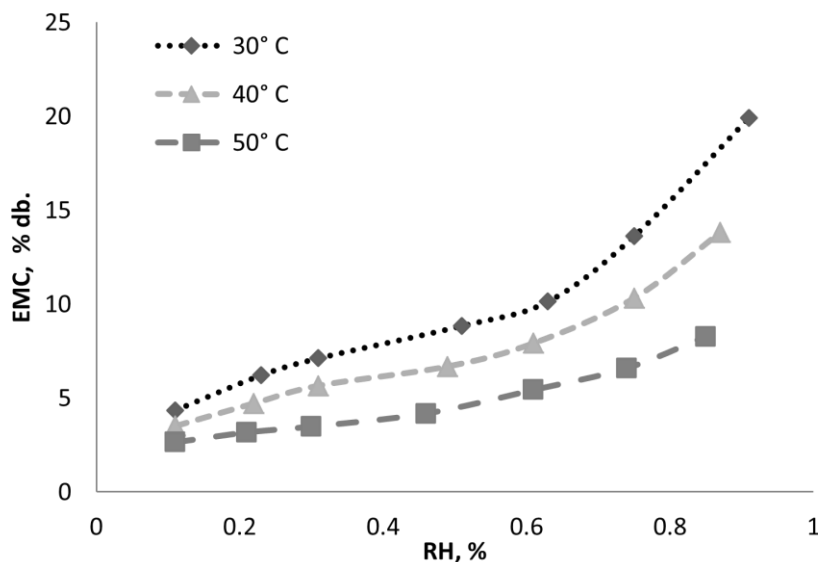


Figure 3 Desorption isotherm data of lime slice at three temperatures

At constant relative humidity, the EMC values decreased with an increase in temperature, while the EMC value increased with an increase in relative humidity at constant temperature. This behavior may be explained by considering excitation states of water molecules. At increased temperatures, molecules are in an increased state of excitation, thus increasing their distance apart and decreasing the attractive forces between them. This leads to a decrease in the degree of the EMC at a given water

activity with increasing temperature (Kouhila et al., 2002; Zomorodian et al., 2011). The equilibrium moisture content increases with increasing relative humidity (water activity) at a constant temperature. This can be explained in such a way that when relative humidity rises, the water vapor partial pressure reaches closer to saturation vapor pressure and thus the pressure gradient (water potential) between the material and the environment arise, thus more moisture is absorbed. In a general way, both temperature

and water activity have significant effect on experimental equilibrium moisture content values. The typical S-shape curves were found for all three temperatures. Similar trends have been observed in several other studies for agricultural and food products (Zomorodian, 1979, Ait Mohamed et al., 2004; Zanoelo, 2005; Jamali et al., 2006, Zomorodin and Tavakoli., 2007, Hii et al, 2009, Zomorodian et al., 2011). The selected models were fitted to the experimental data of the lime slice. The Coefficient of determination (R^2), chi-square (χ^2), Root of mean square error (RMSE) and Mean of relative deviation (MRD) of equations are listed in Tables 1, 2 and 3.

Table 1 Estimated values of statistical indicators and model parameters for desorption models (30 °C)

Model	R^2	MRD	χ^2	RMSE
Caurie	0.977	0.084	0.785	0.748
Chung and Pfof	0.980	0.080	0.662	0.687
GAB	0.996	0.043	0.179	0.320
Halsey	0.987	0.086	0.421	0.549
Henderson	0.969	0.115	1.023	0.425
Cohen	0.905	0.177	3.189	1.509
Oswin	0.994	0.038	0.185	0.363
Modified BET	0.340	0.443	27.610	3.972
Modified Chung and Pfof	0.980	0.080	0.828	0.687
Modified Halsey	0.987	0.086	0.527	0.548
Modified Henderson	0.969	0.117	1.666	0.975
Modified Oswin	0.994	0.038	0.231	0.364
Modified Smith	0.993	0.064	0.386	0.406
Peleg	0.998	0.028	0.128	0.235

According to the table 1, the Peleg model can be used to describe the desorption of lime slices at the 30o C, based on the levels of RMSE, χ^2 , MRD and R^2 .

Table 2 Estimated values of statistical indicators and model parameters for desorption models (40 °C).

Model	R^2	MRD	χ^2	RMSE
Caurie	0.982	0.062	0.227	0.445
Chung and Pfof	0.981	0.065	0.284	0.450
GAB	0.997	0.034	0.067	0.195
Halsey	0.994	0.059	0.089	0.252
Henderson	0.970	0.093	0.484	0.588
Cohen	0.938	0.165	0.937	0.818
Oswin	0.996	0.034	0.067	0.220
Modified BET	0.558	0.324	8.375	2.187
Modified Chung and Pfof	0.981	0.065	0.355	0.450
Modified Halsey	0.999	0.051	0.156	0.299

Modified Henderson	0.970	0.094	0.578	0.574
Modified Oswin	0.996	0.034	0.084	0.220
Modified Smith	0.994	0.177	2.116	0.952
Peleg	0.999	0.016	0.020	0.093

The results of the non-linear regression of desorption isotherms of lime slices, at 40° C, are shown in Table 2. The table 2 indicates that the Peleg model also showed a good fitting for the lime slices at given temperature.

Table 3 Estimated values of statistical indicators and model parameters for desorption models (50 °C).

Model	R^2	MRD	χ^2	RMSE
Caurie	0.992	0.040	0.046	0.182
Chung and Pfof	0.983	0.065	0.083	0.243
GAB	0.990	0.015	0.008	0.069
Halsey	0.995	0.025	0.023	0.128
Henderson	0.969	0.094	0.156	0.334
Cohen	0.942	0.108	0.293	0.458
Oswin	0.993	0.046	0.033	0.154
Modified BET	0.512	0.308	3.077	1.326
Modified Chung and Pfof	0.983	0.065	0.104	0.243
Modified Halsey	0.999	0.025	0.028	0.128
Modified Henderson	0.969	0.094	0.193	0.332
Modified Oswin	0.993	0.046	0.041	0.154
Modified Smith	0.998	0.019	0.013	0.076
Peleg	0.999	0.012	0.009	0.064

Models with the highest R^2 and the lowest χ^2 , MRD and RMSE values were selected to predict the EMC for lime slices. Examination of the results in these Tables indicated that the Peleg model suitably describes the experimental desorption data for lime slices throughout the entire range of water activity. In this way, the parameters of the Peleg model can be recommended for prediction of desorption isotherms of lime slices in the temperature and water activity ranging from 30 to 50 °C and from 11% to 91%, respectively. The comparison between the best fit (Peleg model) and the experimental values is shown in (Figures 4, 5 and 6).

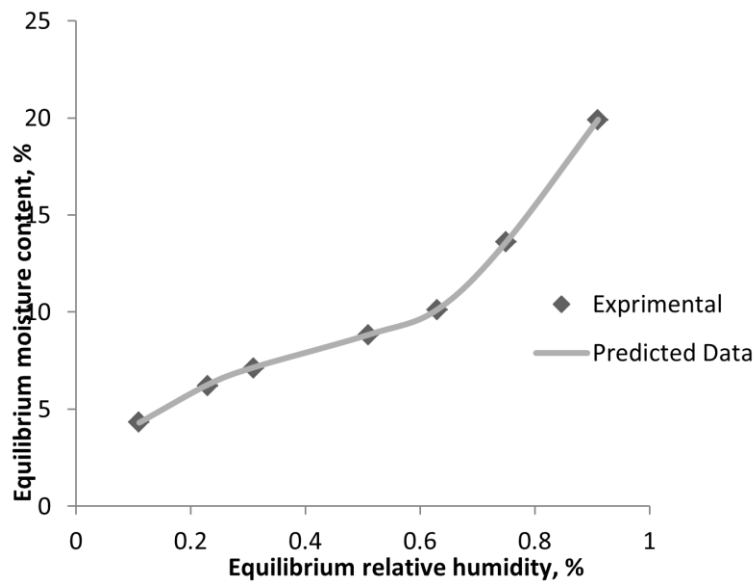


Figure 4 Experimental and predicted (Peleg model) desorption isotherms of lime slice at 30 °C

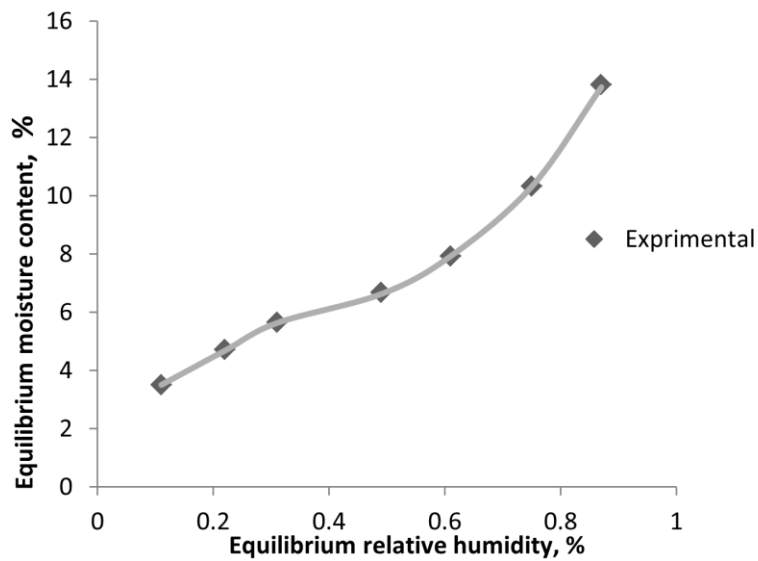


Figure 5 Experimental and predicted (Peleg model) desorption isotherms of lime slice at 40 °C

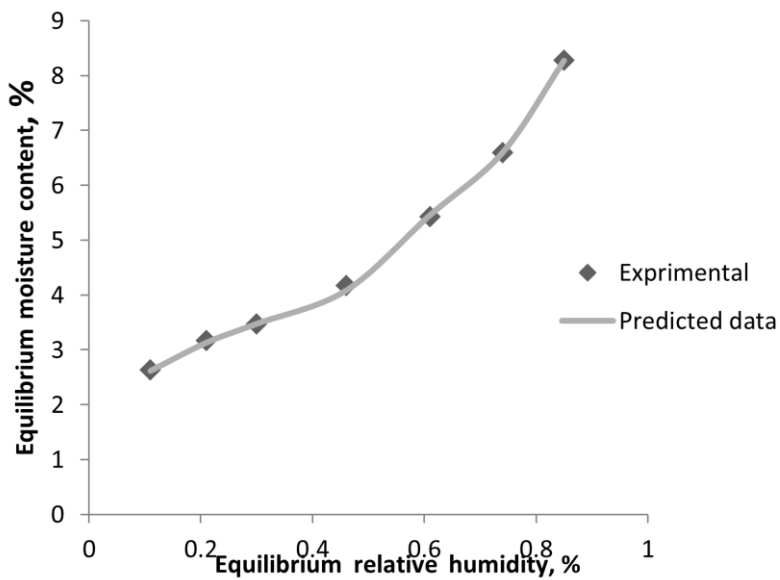


Figure 6 Experimental and predicted (Peleg model) desorption isotherms of lime slice at 50 °C

In Figures 4–6, the adjustment of experimental data with the Peleg model shows a good agreement between experimental and predicted data. Furthermore, this model provides a good representation of Type II (sigmoidal). Comparing the three figures shows that the EMC decreases by increasing in temperature at constant equilibrium relative humidity. Ethmane Kane et al. (2008) reported that the increase of temperature promotes the detachment of water molecules from the water binding sites. Subsequently increasing the temperature lessens the water desorption degree.

3.2 Monolayer moisture content

The values of monolayer moisture content of lime slices estimated with BET model can also be observed in Table 4 and are in the acceptable range for food products. The monolayer moisture content decreases with increasing temperature and the pertinent parameter c increases with increasing temperature (Figures 7, 8 and 9). This effect can be argued that as the chamber temperature changes, the excitation of molecules and the molecular distance

would be changed. (Chung and Pfof, 1967; Zomorodian and Tavakoli, 2007).

Table 4 Values of monolayer moisture content (m_0) and constant cat three micro-environmental air temperatures of 30 °C, 40 °C, 50 °C.

	m_0 (kg H ₂ O/ kg solid)	Constant c
BET equation at 30 °C	0.023	-4.925
BET equation at 40 °C	0.021	-6.543
BET equation at 50 °C	0.014	-7.035

The highest value of monolayer moisture content i.e. 0.023 kg H₂ O/ kg solid was found in the desorption process at 30 °C and the minimum value i.e. 0.014 kg H₂ O/ kg solid was found in the desorption process at 50 °C. As temperature increased from 30 to 50 oC, the monolayer value decreased in desorption processe. This may be due to higher water vapour pressure inside the lime slices at higher temperature. This behaviour has been ascribed to a reduction in the number of active sites due to physical and chemical changes induced by temperature (Rizvi, 1986).

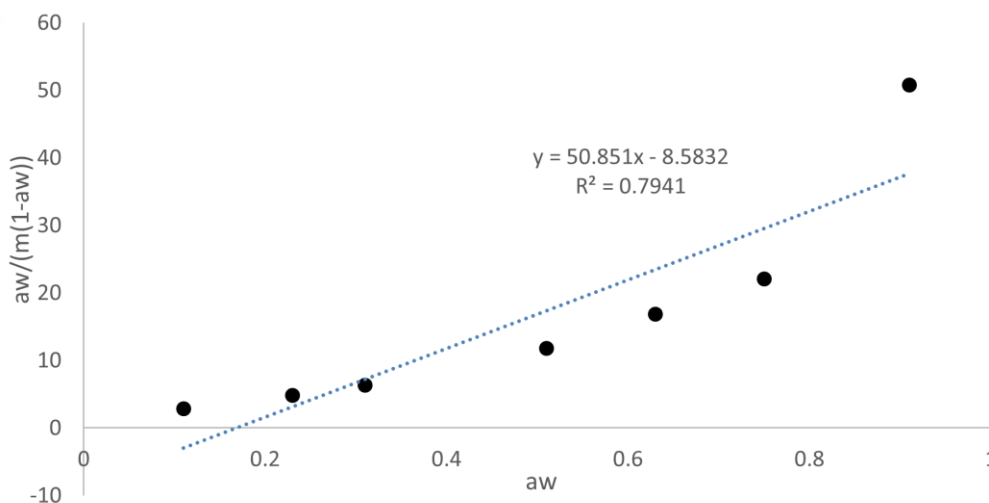


Figure 7 The BET monolayer moisture content plot at 30 °C

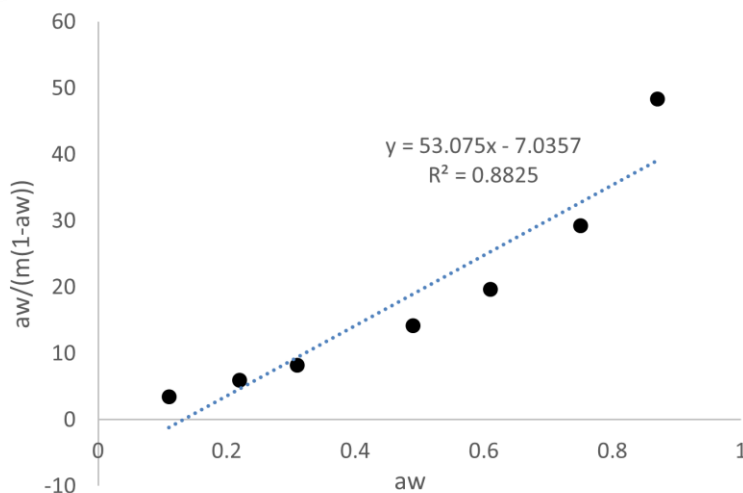


Figure 8 The BET monolayer moisture content plot at 40 °C

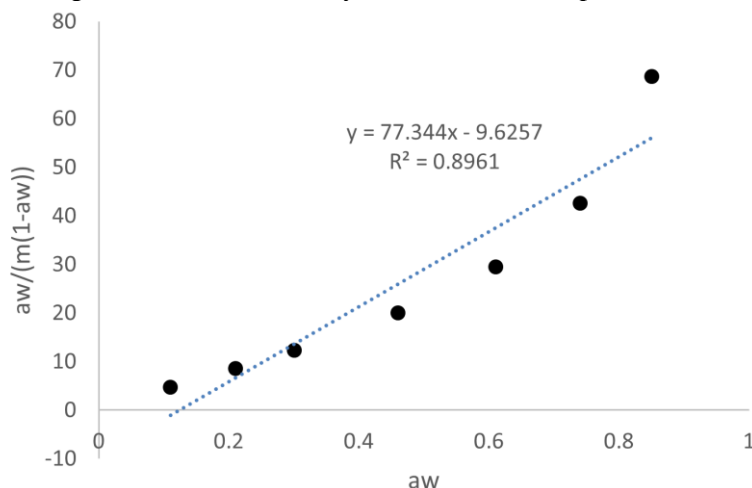


Figure 9 The BET monolayer moisture content plot at 50 °C

4 Conclusions

Based on the results, the following conclusions can be drawn from the study:

- a) The EMCs of lime slices increased directly proportionally to water activity and inversely proportionally to temperature.
- b) The Peleg model provided more accurate predictions about the desorption data.
- c) The monolayer moisture contents decreased as temperature increases.

Abbreviations

aw _ water activity (decimal); a, b, c, d, e, _ constants, EMC _ equilibrium moisture content (% d.b); MSE _

mean square error; R²_ determination coefficient; T_ environmental absolute temperature (K); χ^2 - Chi-Square; RMSE - Root Mean Square Error, MRD- Mean of relative deviation

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