MATHEMATICAL MODELLING OF THE SORPTION ISOTHERMS OF QUINCE

by

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The moisture adsorption isotherms of quince were determined at four temperatures 15, 30, 45, and 60 °C over a range of water activity from 0.110 to 0.920 using the standard static gravimetric method. The experimental data were fitted with generated three parameter sorption isotherm models on Mitrevski et al., and the referent Anderson model known in the scientific and engineering literature as Guggenheim-Anderson-de Boer model. In order to find which models give the best results, large number of numerical experiments was performed. After that, several statistical criteria for estimation and selection of the best sorption isotherm model was used. The performed statistical analysis shows that the generated three parameter model M11 gave the best fit to the sorption data of quince than the referent three parameter Anderson model.

Key words: *adsorption isotherms, quince, statistical analysis*

Introduction

Increasing demand for dry fruits have given a new initiative for food manufactures to produce dried fruit products. Dried fruit informal in human diet and nutrition is sources of vitamins, minerals, and dietary fibers. The quince is a fruit that is a good source of minerals such as Cu, iron, potassium, and Mg as well as vitamin C and B-complex vitamins such as thiamin, riboflavin, and pyridoxine (vitamin B-6). Because fresh quince has short shelf life, preservation after harvesting is necessary by using of different processes such as storing, drying, and canning. The moisture sorption isotherms are important practical tool in modelling the drying process, in design and optimization of drying equipment, in predicting shelf-life stability, in calculating moisture changes which may occur during storage and in selecting appropriate packaging material [1]. In scientific and engineering literature, published data for sorption isotherms of quince are scarce and for that reasons, its determination is interesting for the researchers. Only several researches have reported sorption data for quince at different temperatures and water activities. Kaya *et al*. [2] using static gravimetric method determined sorption isotherms of quince at four temperatures 25, 35, 45, and 55 °C for a range of water activity from 0.12 to 1, while Noshad *et al.* [3] determined the effect of osmotic-ultrasonic dehydration on the desorption isotherms of quince at three temperatures 30, 45, and 60 °C over a range of water activity

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from 0.11 to 0.90. For approximation of the experimental data of sorption isotherm on food materials, numerous mathematical models are available [4-14]. However, due to complex composition and structure of food materials, it is difficult to have a unique mathematical model that describes accurately the sorption isotherms in whole range of water activity [5]. A detailed research of the scientific literature showed that moisture sorption isotherms of foods can be described by more than one sorption model [15]. Generally, criteria used to select most appropriate sorption model are the degree of fit to the equilibrium moisture data and the simplicity of the model. In scientific literature, for the goodness of fit of experimental sorption data and selection of the best isotherm model, following statistical criterions are used: coefficient of determination, R^2, root mean squared error, *RMSE*, and the mean relative deviation, *MRD*. The selection of a sorption isotherm model with graphical evaluation of the residual randomness is also popular [16, 17].

The objectives of this study were: experimental determination of adsorption isotherms of quince at temperatures 15, 30, 45, and 60 ºC for a range of water activity from 0.110 to 0.920 and evaluation of three parameter sorption isotherm models for approximation of equilibrium moisture data of quince and to make comparison on their goodness of fit based on several statistical criteria.

Material and methods

The material used in the experimental part of the research was fresh quince, cultivar *Champion*. Until the processing time, the quince was stored in cold chamber at temperature of 4 ºC and relative air humidity of 75%. To prepare samples, the quinces were washed, peeled and sliced from the mesocarp manually in order to obtain uniform samples with thickness of 2 ± 10^{-1} mm, before being reduced to a cylinder form with diameter of 29 ± 10^{-1} mm. Several measurements were made using a caliper and only samples with a tolerance of ± 5% were used. The representative samples taken for determination of sorption isotherms were predried to final moisture content in a convective dryer at air drying temperature of 60 ºC, and air drying velocity of 1 m/s for a period of 7 hours. The equilibrium moisture content of quince was determined at temperatures of 15, 30, 45, and 60 ºC using static gravimetric method [18, 19]. Ten saturated salt solutions LiCl, CH_3COOK, MgCl, K_2CO_3, $Mg(NO_3)_2$, NaBr, $SrCl_2$, NaCl, KCl, and $BaCl_2$ prepared according to the recommendation of Greenspan [20], were used to give defined constant equilibrium relative humidity in the glass jars from 0.110 to 0.920. Two dry samples were placed on holder into each of the ten glass jars and exposed to atmospheres of various relative humidity. At water activities, $a_w > 0.60$, small quantity of crystalline thymol was placed in the glass jar in order to prevent microbial spoilage of the sample of quinces. The glass sorption jars were placed and kept in the temperature controlled cabinet type SANYO MCO-15AC (SANYO Electric Co., Ltd. Refrigeration Products Division 1-1-1, Sakata Oizumi-Machi, Ora-Gun, Gunma 370-0596, Japan), maintained at temperatures 15, 30, 45, and 60 ºC with an accuracy of ±0.1ºC (fig. 1). Three replications were made at each temperature and equilibrium relative humidity in the glass jars, using two samples per replication and the average values of equilibrium moisture content were calculated. The change of samples mass was determined by electrical balance type KERN PLJ360-3M (Kern&Sohn GmbH, Balingen, Germany), with precision of 0.001 g every 7 days. The equilibrium between samples and their environment was reached after 21 days as evidenced by the constant weight after two successive weighing of samples. The equilibrium moisture content of the samples was determined gravimetrically by drying in an oven at temperature of 105 ºC and atmospheric pressure for 24 hours.

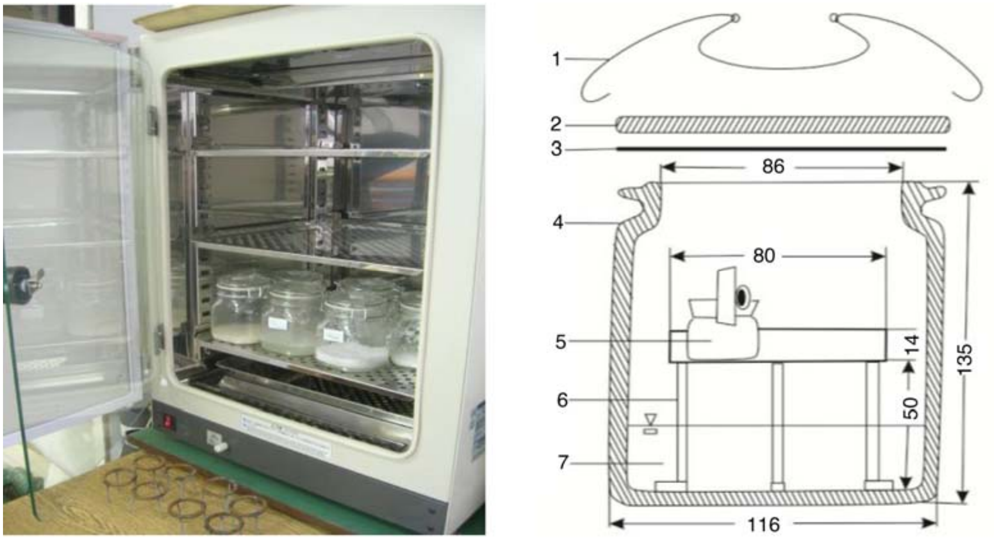

Figure 1. Experimental apparatus for determination of sorption isotherms; (a) temperature controlled cabinet and (b) preserving jar;
1 – locking ring, 2 – glass lid, 3 – rubber ring, 4 – glass container, 5 – measuring container, 6 – Petri-dish on tripod, and 7 – saturated salt solution

Statistical criterion for selection of sorption isotherm model

Statistical analysis is a very useful tool in many engineering problems in order to obtain certain statistical parameters. Generally, several statistical criterions or statistical parameters are used for selection of sorption isotherm models. For selection of the most appropriate sorption model several statistical criteria proposed by Ruiz-Lopez, and Herman-Lara, [17] were used.

The value of performance index, ϕ, is the first statistical criterion for selection of isotherm sorption model [17]:

$$\phi = \frac{R^2}{RMSE \times MRD} \tag{1}$$

Higher values of performance index, ϕ, indicate that the sorption model better approximates the experimental sorption data.

The D'Agostino-Pearson's test of normality is the most effective procedure for assessing a goodness of fit for a normal distribution. This test is based on the individual statistics for testing of the residual population of skewness, z_1, and kurtosis, z_2, and is the second statistical criterion as adequate of sorption model:

$$\chi^2 = z_1^2 + z_2^2 \tag{2}$$

The χ^2 statistics has a chi-squared distribution with two degrees of freedom (*df*). The tabled critical 0.05 chi-square value for $df = 2$ is $\chi^2_{0.05} = 5.99$. Therefore, if the computed value of chi-square is equal to, or greater than, either of the aforementioned values, the null hypothesis can be rejected at the appropriate level of significance ($p > 0.95$), *i. e.* the sorption model should be rejected [21].

The single-sample run test is one of numerous statistical procedures that have been developed for evaluating whether or not the distribution of series is random. This test is the third statistical criterion for effectiveness of sorption model. In this test, the number of positive and negative residuals, n_1 and n_2, and the number of times the sequence of residuals changes sign, g, are used to calculate the following test statistic [21]:

$$\left.\begin{array}{l} z_{\mathrm{r}} = \dfrac{|g - g_1| - 0.5}{\sigma_{\mathrm{r}}}, \qquad g_1 = \dfrac{2n_1 n_2}{n_1 + n_2} + 1, \qquad \sigma_{\mathrm{r}} = \sqrt{\dfrac{2n_1 n_2 (2n_1 n_2 - n_1 - n_2)}{(n_1 + n_2)^2 (n_1 + n_2 - 1)}} \end{array}\right\} \tag{3}$$

If the computed value of, z_{r}, is greater than the tabled critical two-tailed value $z_{0.05} = 1.96$, the null hypothesis should be rejected ($p > 0.95$), *i. e.* the sorption model should be rejected [21].

A fourth statistical criterion for selection of sorption isotherm model is the evaluation of significance and precision of the model constant. That can be done by constructing individual confidence intervals, CI, and with calculated two-tailed p-value of estimated parameters. If the estimated value of parameters is out of the 95% confidence interval or estimated two-tailed p-value according to, t-test of statistic is ($p < 0.05$) the model contains non--significant parameters for approximation of experimental sorption data *i. e.* sorption isotherm model should be rejected.

Results and discussion

The experimental values for the equilibrium moisture content on the slices of the quince from the mesocarp at each water activity for the four different temperatures given in tab. 1 were fitted with three parameters model (M01-M11) generated of Miterevski *et al.*, [14], and plus referent Anderson model (M12), tab. 2.

Table 1. Equilibrium moisture content of quince*

15 °C		30 °C		45 °C		60 °C	
a_{w}	X_{eq} [kg/kg d. b.]	a_{w}	X_{eq} [kg/kg d. b.]	a_{w}	X_{eq} [kg/kg d. b.]	a_{w}	X_{eq} [kg/kg d. b.]
0.113	0.008 ± 0.000	0.113	0.013 ± 0.001	0.112	0.009 ± 0.002	0.110	0.010 ± 0.003
0.234	0.023 ± 0.000	0.216	0.038 ± 0.001	0.195	0.030 ± 0.002	0.160	0.021 ± 0.001
0.333	0.050 ± 0.001	0.324	0.057 ± 0.003	0.311	0.041 ± 0.003	0.293	0.037 ± 0.001
0.432	0.093 ± 0.001	0.432	0.087 ± 0.002	0.432	0.076 ± 0.002	0.432	0.063 ± 0.003
0.559	0.149 ± 0.000	0.514	0.113 ± 0.002	0.469	0.090 ± 0.002	0.440	0.071 ± 0.002
0.607	0.180 ± 0.002	0.560	0.130 ± 0.000	0.520	0.102 ± 0.002	0.497	0.081 ± 0.001
0.741	0.295 ± 0.001	0.691	0.224 ± 0.002	0.640	0.158 ± 0.002	0.580	0.110 ± 0.001
0.756	0.320 ± 0.002	0.751	0.293 ± 0.003	0.745	0.242 ± 0.003	0.745	0.220 ± 0.001
0.859	0.492 ± 0.001	0.836	0.450 ± 0.001	0.817	0.354 ± 0.001	0.803	0.306 ± 0.001
0.920	0.799 ± 0.003	0.900	0.715 ± 0.002	0.880	0.599 ± 0.002	0.840	0.565 ± 0.003

* Mean and standard deviation based on $N = 3$ replications
d. b. – day basis

Table 2. Mathematical models for fitting the equilibrium moisture data

Model	Equation	Ref.
M01	$x_{\mathrm{eq}} = \exp\left(P_1 + P_2 \ln a_{\mathrm{w}} + P_3 \ln^2 a_{\mathrm{w}}\right)$	[14]
M02	$x_{\mathrm{eq}} = \exp\left[P_1 + \dfrac{P_2}{1+a_{\mathrm{w}}}\right] + \left[\dfrac{P_3}{\left(1+a_{\mathrm{w}}\right)^2}\right]$	[14]
M03	$x_{\mathrm{eq}} = \exp\left[P_1 + P_2 \dfrac{1+a_{\mathrm{w}}}{1+2a_{\mathrm{w}}} + P_3\left(\dfrac{1+a_{\mathrm{w}}}{1+2a_{\mathrm{w}}}\right)^2\right]$	[14]
M04	$x_{\mathrm{eq}} = \exp\left(P_1 + P_2 \exp a_{\mathrm{w}} + P_3 \exp^2 a_{\mathrm{w}}\right)$	[14]
M05	$x_{\mathrm{eq}} = \exp\left[P_1 + P_2 \exp\left(1-a_{\mathrm{w}}\right) + P_3 \exp^2\left(1-a_{\mathrm{w}}\right)\right]$	[14]
M06	$x_{\mathrm{eq}} = \exp\left[P_1 + P_2 \ln\left(1+a_{\mathrm{w}}\right) + P_3 \ln^2\left(1+a_{\mathrm{w}}\right)\right]$	[14]
M07	$x_{\mathrm{eq}} = \exp\left\{P_1 + P_2 \dfrac{a_{\mathrm{w}}}{\ln\left(1+a_{\mathrm{w}}\right)} + P_3\left[\dfrac{a_{\mathrm{w}}}{\ln\left(1+a_{\mathrm{w}}\right)}\right]^2\right\}$	[14]
M08	$x_{\mathrm{eq}} = \exp\left[P_1 + P_2 \dfrac{a_{\mathrm{w}}}{\sin a_{\mathrm{w}}} + P_3\left(\dfrac{a_{\mathrm{w}}}{\sin a_{\mathrm{w}}}\right)^2\right]$	[14]
M09	$x_{\mathrm{eq}} = \exp\left[P_1 + P_2 \dfrac{\sin a_{\mathrm{w}}}{a_{\mathrm{w}}} + P_3\left(\dfrac{\sin a_{\mathrm{w}}}{a_{\mathrm{w}}}\right)^2\right]$	[14]
M10	$x_{\mathrm{eq}} = \exp\left[P_1 + P_2 \dfrac{\arcsin a_{\mathrm{w}}}{a_{\mathrm{w}}} + P_3\left(\dfrac{\arcsin a_{\mathrm{w}}}{a_{\mathrm{w}}}\right)^2\right]$	[14]
M11	$x_{\mathrm{eq}} = \exp\left[P_1 + P_2 \dfrac{a_{\mathrm{w}}}{\arctan a_{\mathrm{w}}} + P_3\left(\dfrac{a_{\mathrm{w}}}{\arctan a_{\mathrm{w}}}\right)^2\right]$	[14]
M12	$x_{\mathrm{eq}} = \dfrac{P_1 a_{\mathrm{w}}}{\left(1-P_2 a_{\mathrm{w}}\right)\left(1-P_3 a_{\mathrm{w}}\right)}$	[22]

Because the regression methods (indirect non-linear or direct non-linear), estimation method, the initial step size, the start values of parameters, convergence criterion and form of the function have significant influence on accuracy of estimated parameters [19], a large number of numerical experiments were performed. The method of indirect non-linear regression and estimation methods of Quasi-Newton, Simplex, Simplex and quasi-Newton, Hooke-Jeeves pattern moves, Hooke-Jeeves pattern moves and quasi-Newton, Rosenbrock pattern search, Rosenbrock pattern search and quasi-Newton, Gauss-Newton and Levenberg-Marquardt from computer program STATISTICA [23], were used to approximate the experimental equilibrium moisture content data of quince. On the basis of experimental data, and each mathematical model from tab. 2, the values of: R^2, *RMSE*, *MRD*, and ϕ, were calculated. After that, the models were ranked on the basis of values of the performance index, ϕ, tab. 3. From tab. 3 it is evident that the Anderson model *i. e.* GAB model (M12), has the highest value of average performance index, $\phi = 260.77$ (rank 1). In agreement with the first statistic criterion, this model correlates the experimental values of sorption data of quince better than other models. From all models, the generated model in [14] (M03) has the smallest value of performance index, $\phi = 56.319$ (rank 12). So, this model exhibited the worst ability to correlate the experimental equilibrium moisture data of quince according to first statistical criterion.

Table 3. Statistic summary of the regression analysis

Model	R^2	*RMSE*	*MRD*	ϕ	Rank
M01	0.9672	0.0380	0.3972	64.147	9
M02	0.9721	0.0350	0.3865	71.874	7
M03	0.9655	0.0389	0.4405	56.319	12
M04	0.9805	0.0293	0.3318	100.95	2
M05	0.9737	0.0340	0.3649	78.512	4
M06	0.9769	0.0319	0.3653	83.891	3
M07	0.9776	0.0314	0.5104	61.024	11
M08	0.9788	0.0305	0.4525	70.850	8
M09	0.9788	0.0305	0.4305	74.571	6
M10	0.9776	0.0314	0.5105	61.024	10
M11	0.9788	0.0305	0.4212	76.202	5
M12	0.9823	0.0279	0.1350	260.77	1

In tab. 4, the computed values for, χ^2, and, z_r, are given. From the same table, it is obvious that only Mitrevski *et al.*, [14] and models M02, M05, M09, and M011, have value of, χ^2 and z_r, smaller than the tabled critical value. In accordance with the proposed statistical criteria, these models are able to correlate the experimental values of sorption isotherms of quince with 3.05--3.50% average RMSE. From tab. 4 it can be see that Anderson model have value of, χ^2, and, z_r, greater than the tabled critical value. In accordance with the proposed statistical criteria, this model was rejected in further statistical evaluation.

Table 4. Rejection criteria for sorption models

Model	χ^{2a}	z_r^{b}	Rejection criteria
M01	**7.0094**	1.6589	χ^2
M02	5.7162	1.7972	–
M03	**6.4803**	1.7972	χ^2
M04	**8.7018**	**2.6866**	χ^2, z_r
M05	5.0774	1.6589	–
M06	3.3503	**1.9840**	z_r
M07	5.8120	**3.2957**	z_r
M08	**6.6080**	**2.6399**	χ^2, z_r
M09	5.9579	1.7155	–
M10	5.8120	**3.2957**	z_r
M11	5.1826	1.6561	–
M12	**18.768**	**2.0392**	χ^2, z_r

"–" The model is not rejected
[a] Bold numbers indicate a significant lack of normality of the moisture residuals ($p > 0.05$)
[b] Bold numbers indicate a significant lack of randomness of the moisture residual series ($p > 0.05$)

Table 5. Estimated values of parameters, 95% confidence intervals (CI) and *p*-value

Model	Parameter	Value*	95% CI	p
M02	P_1	19.289	(15.452 , 22.883)	**1.36E-12**
	P_2	–54.399	(–68.292 , –42.086)	**6.09E-11**
	P_3	32.349	(22.603 , 42.113)	**6.69E-08**
M05	P_1	8.1009	(6.5748 , 9.4609)	**1.65E-13**
	P_2	–10.081	(–12.082 , –7.9758)	**6.06E-12**
	P_3	2.1811	(1.4852 , 2.9033)	**2.70E-07**
M09	P_1	27.604	(–5.6610 , 60.870)	1.01E-01
	P_2	–40.061	(–113.46 , 33.338)	2.76E-01
	P_3	9.1069	(–31.352 , 493.565)	6.56E-01
M11	P_1	–5.8999	(–24.687 , 12.868)	5.28E-01
	P_2	– 6.1358	(–38.387 , 26.150)	7.03E-01
	P_3	8.6669	(–5.1900 , 22.504)	2.13E-01

* Bold numbers indicate that estimated parameters have a lack of significance ($p > 0.05$)

The values of model parameters for models M02, M05, M09, and M011 were estimated by fitting the models to experimental equilibrium moisture content data of quince using Gauss-Newton estimation method which minimizes the sum squares errors.

The 95% confidence intervals of the estimated parameters were determined by using the *nlparci* (beta, resid,'jacobian', J) function of the Statistics Toolbox of Matlab®8.3, [24], while the significance of each of the estimated parameters, P_1, P_2, P_3 was evaluated through, t-test statistic. The estimated values of parameters, 95% confidence intervals and two tailed *p*-value of estimated parameters are given in tab. 5.

From tab. 5 it can be see that the calculated two tailed *p*-values for models M02 and M05 for all parameters are extremely small (much less than $p < 0.05$). So, these models contain nonsignificant parameters for approximation of experimental sorption data of quince. In accordance with the proposed statistical criteria, these models were rejected in further statistical evaluation. The calculated two tailed *p*-values for all parameters for models M09 and M11 for each of estimated parameters has statistical significance, tab. 5. In accordance to proposed statistical criterion these models are able to approximate moisture equilibrium content data of quince in the whole range of water activity. The statistical superiority of these models over the referent model of Anderson has been confirmed on experimental data from pear and potatoes [14]. But, from the first statistical criterion, the model M11 have higher value of performance index, $\phi = 76.202$ than model M09, $\phi = 74.571$. Thus, the model M11 on Mitrevski *et al*., [14] better approximate experimental sorption data of quince.

The experimental and predicted values for equilibrium moisture content for quince at four temperatures are shown on fig. 2. The random pattern of the moisture residual series can be observed in fig. 3, while the normal distribution of the residuals is verified in fig. 4.

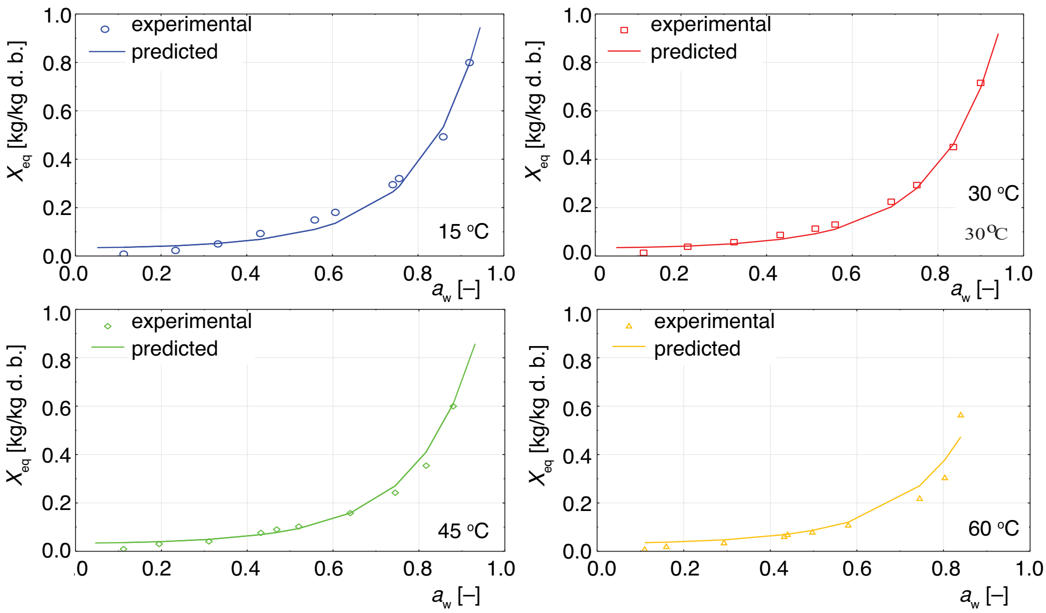

Figure 2. Experimental and predicted sorption isotherms for quince at 15, 30, 45, and 60 °C for the model M11

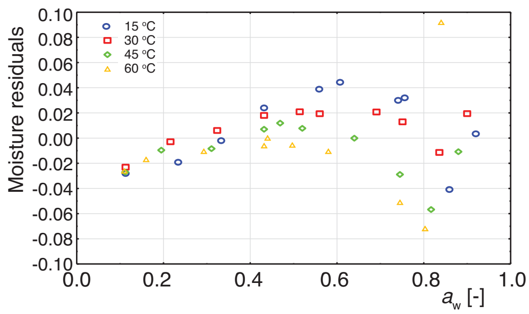

Figure 3. Residuals plot of sorption data for quince-Model M11

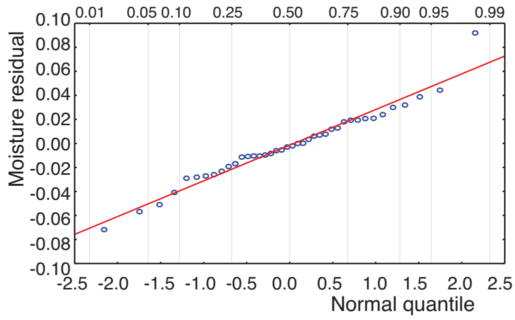

Figure 4. Normal quantile plot of the residuals for quince data-Model M11

Conclusion

The moisture adsorption isotherms of quince at four temperatures 15, 30, 45, and 60 ºC and ten different water activities were experimentally determined with the gravimetric static method. The experimental equilibrium moisture content data of quince were fitted with eleven generated three-parameter sorption isotherm models on Mitrevski *et al.,* [14] and the referent Anderson model. In accordance to proposed statistical criterion it was concluded that the generated model of Mitrevski *et al*., [14], M11, have a better statistical fit on experimental equilibrium moisture data of quince in whole range of water activity than referent Anderson model and others generated models. So, this model can be successfully used in practical calculations of the equilibrium moisture content data, which is important parameter in storage conditions of dry food materials. With incorporation of the [14] M11 model in the drying model more accurate values of temperature profiles, transient moisture content, mid-plane temperature, and the volume averaged moisture content changes for given drying regime on the dried food materials will be obtained.

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Nomenclature

a_w – water activity
g – residual change
g_1 – mean of the sampling distribution of runs in a random series
MRD – mean relative deviation
n_1, n_2 – number of positive and negative residuals
P_1, P_2, P_3 – parameters
R^2 – coefficient of determination
$RMSE$ – root mean squared error
z_1, z_2 – statistic for testing the skewness and kurtosis of the residual population
z_r – statistic for testing the randomness of the moisture residual series
X – moisture content, [kgkg^{-1} d. b.]

Greek symbols

χ^2 – statistic for testing the normality of the moisture residuals
ϕ – measure for the goodness of fit
σ_r – expected standard deviation of the sampling distribution of runs in a random series

Subscripts

eq – equilibrium

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