

STATISTICAL EVALUATION OF FOUR-PARAMETER MOISTURE SORPTION ISOTHERM MODELS

STATISTIČKA OCENA NEKIH ČETIRI PARAMETARSKIH MODELA SORPCIONIH IZOTERMI

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ABSTRACT

In the scientific and engineering literature, there are various mathematical methods for modelling the equilibrium moisture content of agricultural and food materials. The objective of the present study was to statistically evaluate a total of thirty-two four-parameter moisture sorption isotherm models and subsequently compare their goodness of fit. The moisture sorption isotherm models considered were either originally developed or taken from the reference literature. The coefficient of determination and the graphical evaluation of residual randomness were utilized as the main assessment criteria for statistical evaluation of the moisture sorption isotherm models examined. The statistical performance of the models was tested according to the equilibrium moisture content of quinces. On the basis of the statistical analyses performed, the Popovski&Mitrevski model, i.e. the model with the reference numbers M25 and M30, and the McLaren&Rowen model were found to have the best statistical performance of all the models considered.

Key words: sorption isotherms, four-parameter models, statistical evaluation.

REZIME

Skladištenje vlažnog materijala može biti stabilno, ako je materijal u ravnoteži sa okolnim vazduhom. Ravnoteža zavisi od termodinamičkog stanja materijala i okolnog vazduha. Da bi se ovo poznavalo obavljaju se ispitivanja sorpcionih svojstava, odnosno istražuju se sorpcione krive za različite materijale. Slično razmatranje odnosi se na aktivnost vode. Za aproksimaciju sorpcionih podataka poljoprivrednih i prehranbenih materijala u naučnoj literaturi dostupni su brojni matematički modeli. Zavisno od broja parametara, modeli mogu biti jedno, dvo, tro, četvoro ili više parametaraskih. U ovom radu je prikazana statistička ocena trideset i dva četiri parametarska modela koji su korišćeni za aproksimaciju ravnotežne vlažnosti dunje. Analizirani modeli su preuzeti iz referentne naučne literature, a nekih su izvorno generisani. U cilju određivanja, koji model najbolje aproksimira eksperimentalne rezultate izoterme sorpcije dunje, urađeni su brojni numerički eksperimenti. Za svaki model i set podataka proračunata je vrednost koeficijenta determinacije i onda su modeli rangirani. Kako dopunski statistički kriterijum korišćena je grafička evaluacija ostataka regresione analize. Na osnovu dobijenih rezultata može se zaključiti da modeli Popovski&Mitrevski sa referentnim brojem M25 i M30 i model McLaren&Rowen, referentni broj M03, imaju najbolje statističke osobine, a za koje vrednosti koeficijenta determinacije iznose $R^2 = 0.99157$. Korišćene su metode minimuma greške sume kvadrata da bi se statistički procenilo slaganje računskih sa eksperimentalnim podacima ravnotežne vlažnosti dunje.

Ključne reči: izoterme sorpcije, četiri parametarski modeli, statistička ocena.

INTRODUCTION

In the scientific and engineering literature, there are numerous mathematical models for approximating the moisture sorption of agricultural and food materials. Depending on the number of parameters included, such models are referred to as one-parameter, two-parameter, three-parameter, or multi-parameter models.

The moisture sorption isotherm data, as a function of two or more temperatures, are important for the thermodynamic analysis, drying kinetics modelling and evaluation of food stability during storage (Vega-Galvez et al., 2007).

Over the past two decades, an increasing number of studies have been concerned with methods for sorption and/or desorption isotherm determination (Mitrevski et al., 2015; Mitrevski et al., 2018), sorption isotherm temperature dependence (Popovski&Mitrevski, 2004), sorption heat determination (Kaymak-Ertekin and Gedik, 2004) and the development of mathematical models for approximating

moisture sorption data (Mitrevski et al., 2012; Mitrevski et al., 2015a).

In engineering calculations, the simplicity of a mathematical model (namely a model with a smaller number of parameters) is of great importance. Provided a sorption isotherm model is incorporated into a mathematical model for calculating the drying process of certain products or used to predict the shelf-life of packaged dried products, approximations of the experimental data on the equilibrium moisture content of such products are highly significant (Boquet et al., 1978).

The objective of the present study was to statistically evaluate a total of thirty-two four-parameter sorption isotherm models for approximating the data on the equilibrium moisture content of quinces and to subsequently compare their goodness of fit using the coefficient of determination.

MATERIAL AND METHOD

In this study, the equilibrium moisture contents of the quince samples were determined at temperatures of 15, 30, 45 and 60 °C using the static gravimetric method (Mitrevski et al., 2018,

Mitrevski et al., 2019). A total of ten saturated salt solutions (namely LiCl , CH_3COOK , MgCl , K_2CO_3 , $\text{Mg}(\text{NO}_3)_2$, NaBr , SrCl_2 , NaCl , KCl and BaCl_2) were utilized to obtain the constant equilibrium relative humidity in the glass jars ranging from 0.110 to 0.920. Two dry samples were placed on holders in each of the ten glass jars and exposed to atmospheres of various relative humidity.

The glass sorption jars were placed and kept in the temperature controlled SANYO MCO-15AC cabinet (SANYO Electric Co., Ltd. Refrigeration Products Division 1-1-1, Sakata Oizumi-Machi, Ora-Gun, Gunma 370-0596 Japan) and maintained at temperatures 15, 30, 45 and 60 °C (with an accuracy of ± 0.1 °C). 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 the equilibrium moisture content were calculated (Mitrevski et al., 2018, Mitrevski et al., 2019). Changes in the sample masses were recorded every 7 days using the electrical balance KERN PLJ360-3M (Kern&Sohn GmbH, Ziegelei 1, 72336 Balingen, Germany) with a precision of 0.001 g.

The equilibrium between the samples and their environments was reached after 21 days as evidenced by the constant mass of the samples after two successive measurements. The equilibrium moisture content of the samples was determined gravimetrically by oven drying for 24 h at a temperature of 105 °C and atmospheric pressure.

RESULTS AND DISCUSSION

The equilibrium moisture content values of the quince slices X_{eq} (Table 1), obtained at different water activity values a_w and four different temperatures (Mitrevski et al., 2018, Mitrevski et al., 2019), were fitted with thirty-two four-parameter sorption isotherm models M01-M32 (Table 2).

Table 1. Equilibrium moisture content of the quince samples*

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

*mean and standard deviation based on $N = 3$ replications

Table 2. Mathematical models for approximating the sorption data

Num. model	Name of model	Model	References
M01	Enderby	$X_{eq} = a_w \left(\frac{A}{1 - Ba_w} + \frac{C}{1 - Da_w} \right)$	Enderby, 1955
M02	Kollmann	$X_{eq} = Aa_w^B + \exp[(a_w - C)^2 D]$	Kollmann, 1962
M03	McLaren & Rowen	$X_{eq} = \frac{a_w}{1 - a_w} \left(\frac{A}{1 - Ba_w} + \frac{C}{1 - Da_w} \right)$	McLaren & Rowen, 1951
M04	Peleg	$X_{eq} = Aa_w^B + Ca_w^D$	Peleg, 1993
M05	Polynomial equation	$X_{eq} = A + Ba_w + Ca_w^2 + Da_w^3$	Castillo et al., 2003
M06	Popovski & Mitrevski	$X_{eq} = \exp(Aa_w)B + \exp(Ca_w)D$	Popovski and Mitrevski, 2004
M07	Popovski & Mitrevski	$X_{eq} = A \left(\frac{a_w}{1 - a_w} \right)^B + C \left(\frac{a_w}{1 - a_w} \right)^D$	Popovski and Mitrevski, 2004
M08	Popovski & Mitrevski	$X_{eq} = \exp[A \ln(a_w)^2]B + \exp[C \ln(a_w)^2]D$	Popovski and Mitrevski, 2004
M09	Popovski & Mitrevski	$X_{eq} = \frac{A}{(1 - a_w)} + \frac{C}{(1 - Da_w)}$	Popovski and Mitrevski, 2004
M10	Popovski & Mitrevski	$X_{eq} = A(-\ln a_w)^B + C(-\ln a_w)^D$	Popovski and Mitrevski, 2004
M11	Popovski & Mitrevski	$X_{eq} = a_w(1 + a_w) + \left(\frac{1}{1 - Ba_w} + \frac{C}{1 - Da_w} \right)$	Popovski and Mitrevski, 2004
M12	Popovski & Mitrevski	$X_{eq} = A[-\ln(1 - a_w)]^B + C[-\ln(1 - a_w)]^D$	Popovski and Mitrevski, 2004
M13	Popovski & Mitrevski	$X_{eq} = \frac{A}{B - \ln a_w} + \frac{C}{D - \ln a_w}$	Popovski and Mitrevski, 2004
M14	Popovski & Mitrevski	$X_{eq} = \frac{A}{(1 - a_w)^B} + \frac{C}{(1 - a_w)^D}$	Popovski and Mitrevski, 2004a
M15	Popovski & Mitrevski	$X_{eq} = a_w \left[\frac{A}{(1 - Ba_w)^2} + \frac{C}{(1 - Da_w)^2} \right]$	Popovski and Mitrevski, 2004a
M16	Popovski & Mitrevski	$X_{eq} = \left[\frac{Aa_w}{(1 - Ba_w)(1 - Ca_w)} \right]^D$	Popovski and Mitrevski, 2004a
M17	Popovski & Mitrevski	$X_{eq} = \left[\frac{A}{1 - Ba_w} + C \right]^D$	Popovski and Mitrevski, 2004a
M18	Popovski & Mitrevski	$X_{eq} = \left[\frac{A}{1 - Ba_w} + Ca_w \right]^D$	Popovski and Mitrevski, 2004a
M19	Popovski & Mitrevski	$X_{eq} = \frac{Aa_w(1 - a_w^B)}{(1 - a_w)(1 - Ca_w)} + D$	Popovski and Mitrevski, 2005
M20	Popovski & Mitrevski	$X_{eq} = \frac{Aa_w}{(1 - Ba_w)(1 - Ca_w)} + D$	Popovski and Mitrevski, 2005
M21	Popovski & Mitrevski	$X_{eq} = \left(\frac{A}{B - \ln a_w} \right)^C + D$	Popovski and Mitrevski, 2005
M22	Popovski & Mitrevski	$X_{eq} = \frac{A}{1 - Ba_w} + Ca_w + D$	Popovski and Mitrevski, 2005
M23	Popovski & Mitrevski	$X_{eq} = \frac{Aa_w^3}{(1 - Ba_w)(1 - Ca_w)(1 - Da_w)}$	Popovski and Mitrevski, 2005a
M24	Popovski & Mitrevski	$X_{eq} = \left(\frac{A}{1 - a_w} + C \right) \frac{a_w}{1 - Da_w}$	Popovski and Mitrevski, 2005a
M25	Popovski & Mitrevski	$X_{eq} = a_w \left[\frac{A}{(1 - a_w)(1 - Ba_w)} + \frac{C}{1 - Da_w} \right]$	Popovski and Mitrevski, 2005b
M26	Popovski & Mitrevski	$X_{eq} = \frac{Aa_w}{(1 - a_w)(1 - Ba_w)} + Ca_w^D$	Popovski and Mitrevski, 2005b
M27	Popovski & Mitrevski	$X_{eq} = \frac{Aa_w}{(1 - Ba_w)} + Ca_w^D$	Popovski and Mitrevski, 2005b
M28	Popovski & Mitrevski	$X_{eq} = \frac{Aa_w}{(1 - Ba_w)} + \frac{C}{(1 - Da_w)}$	Popovski and Mitrevski, 2005b
M29	Popovski & Mitrevski	$X_{eq} = a_w \left[\frac{A(1 + a_w)}{(1 - Ba_w)} + \frac{C}{(1 - Da_w)} \right]$	Popovski and Mitrevski, 2005b
M30	Popovski & Mitrevski	$X_{eq} = a_w \left[\frac{A}{(1 - Ba_w)(1 - Ca_w)} + \frac{D}{(1 - a_w)} \right]$	Popovski and Mitrevski, 2005b
M31	Popovski & Mitrevski	$X_{eq} = \exp(Aa_w^B + Ca_w^D)$	Popovski and Mitrevski, 2006
M32	Riedel	$X_{eq} = \frac{a_w}{(Aa_w + B)} + \frac{Ca_w^D}{(1 - a_w)}$	van den Berg, and Bruin, 1981

The statistical evaluation of sorption isotherm models depends on the nature of the model itself. The following statistical criteria are used for determining the goodness of fit of an isotherm model: the coefficient of determination (R^2), the root-mean-squared error (RMSE) and the mean relative deviation (MRD).

Sorption isotherm models with the graphical evaluation of residual randomness are also commonly used (Mitrevski et al., 2012). Plotting the residuals against independent variables is a measure of error distribution. If the sorption model is correct, then the residuals should be random independent errors with a zero mean, constant variance and arranged in a normal distribution. If the residual plots indicate a clear pattern, the model should not be accepted (Basu et al., 2006; Ruiz-Lopez et al., 2009). In this study, the coefficient of determination (R^2) and the graphical evaluation of residual randomness were the main statistical indicators for selecting the best four-parameter sorption isotherm model.

As regression methods (both indirect nonlinear and direct nonlinear regression methods), estimation methods, initial step sizes, initial parameter values, convergence criteria and function forms exert significant effects on the accuracy of the parameters estimated, a large number of numerical experiments were performed (Mitrevski et al. 2015). The method of indirect nonlinear regression analysis and the estimation methods such as the 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 software Statistica (Statsoft Inc., Tulsa, OK, http://www.statsoft.com) were used to approximate the experimental equilibrium moisture content of quinces.

On the basis of the experimental data, the coefficient of determination (R^2) was calculated for each mathematical model shown in Table 2. The R^2 values obtained were used to rank the models considered (Table 3).

Table 3. Model Ranking

Model	R^2	Rank	Model	R^2	Rank
M01	0.99119	17	M17	0.99115	18
M02	0.99148	6	M18	0.98943	30
M03	0.99157	3	M19	0.99114	19
M04	0.99147	8	M20	0.99113	22
M05	0.98480	31	M21	0.97819	32
M06	0.99113	24	M22	0.99112	25
M07	0.99156	4	M23	0.99108	26
M08	0.99147	7	M24	0.99119	16
M09	0.99113	23	M25	0.99157	2
M10	0.99124	13	M26	0.99065	29
M11	0.99114	21	M27	0.99121	14
M12	0.99102	27	M28	0.99152	5
M13	0.99114	20	M29	0.99129	12
M14	0.99073	28	M30	0.99157	1
M15	0.99131	11	M31	0.99138	9
M16	0.99121	15	M32	0.99131	10

As can be seen in Table 3, the Popovski&Mitrevski models with the reference numbers M25 and M30 (Popovski&Mitrevski, 2005b) and the McLaren&Rowen model (McLaren and Rowen, 1951) had the highest coefficient of determination value ($R^2 = 0.99157$; rank 1). Consequently, these models correlate the experimental values of quince sorption better than other models. Moreover, the highest coefficient of determination value was obtained when two-parameter sorption isotherm models were

used for approximating quince sorption data (Mitrevski et al., 2019).

Of all the models considered, the model with the reference number M21 (Popovski&Mitrevski, 2005) had the lowest value of the coefficient of determination ($R^2 = 0.97819$; rank 32), i.e. the worst statistical performance.

The A, B, C and D parameter values of the models M03, M25 and M30 were estimated by fitting the models to the experimental equilibrium moisture content of quinces using estimation methods which minimize the sum squares errors. The estimated parameter values are shown in Table 4.

Table 4. Estimated parameter values

Model	A	B	C	D
$XEQ=AW/(1-AW)*((A/(1-B*AW)) + (C/(1-D*AW)))$	0.0209	1.0623	1.4456	0.0937
$XEQ=AW*((A/(1-AW)*(1-B*AW)) + (C/(1-D*AW)))$	0.1231	0.2102	-0.0098	1.0583
$XEQ=AW*((A/(1-B*AW)*(1-C*AW)) + (D/(1-AW)))$	0.0209	1.0623	1.4456	0.0937

XEQ - equilibrium moisture content, AW - water activity, A, B, C, D - parameters

The experimental and predicted values of the equilibrium moisture content of quinces at four different temperatures are shown in Figures 1a-1d.

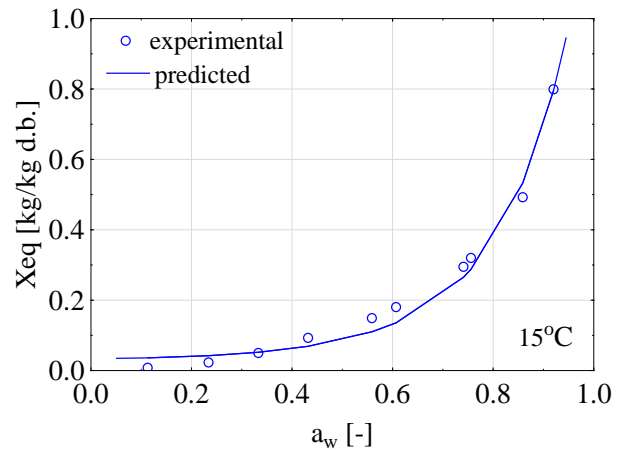


Fig. 1a Experimental and predicted sorption isotherms for the quince samples at 15 °C in the case of the M30 model

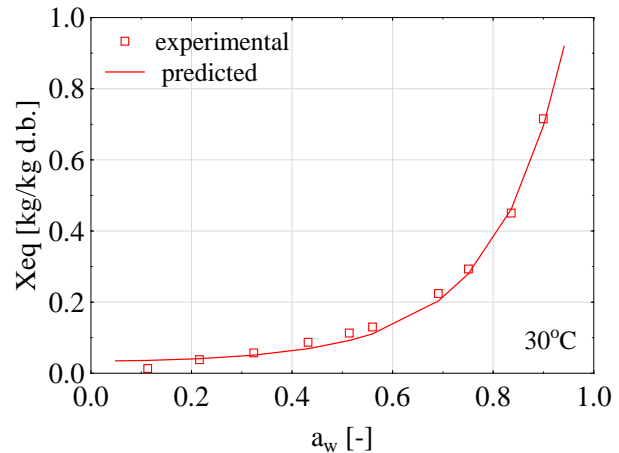


Fig. 1b Experimental and predicted sorption isotherms for the quince samples at 30 °C in the case of the M30 model

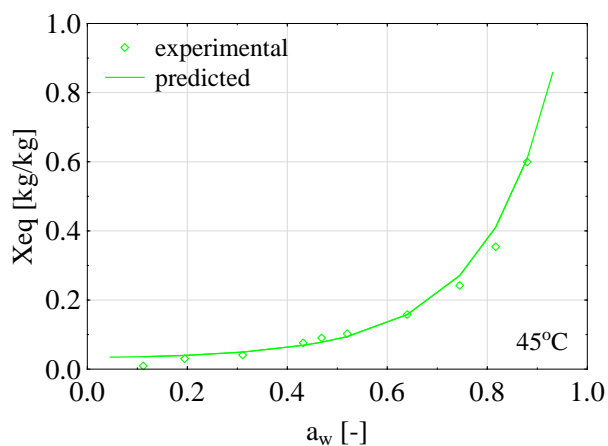


Fig. 1c Experimental and predicted sorption isotherms for the quince samples at 45 °C in the case of the M30 model

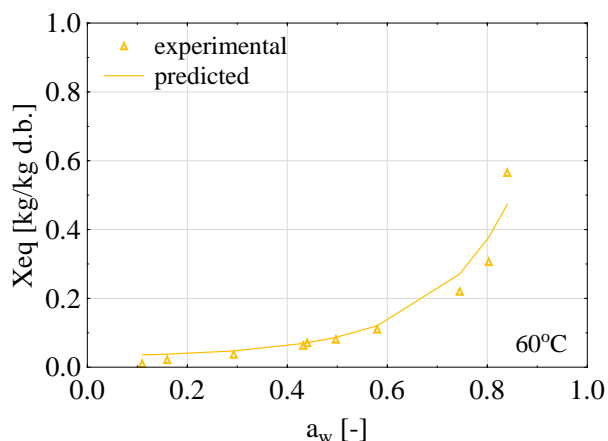


Fig. 1d Experimental and predicted sorption isotherms for the quince samples at 60 °C in the case of the M30 model

As can be seen in Figures 1a-1d, the M30 model exhibited a good agreement between the experimental and predicted values of the equilibrium moisture content of quinces.

The regression analyses for the models M03, M25 and M30 indicate that the residual plots obtained show no abnormal distribution relative to the predicted values. Figures 2a-2c show the residual plots for the M03, M25 and M30 models relative to the experimental values.

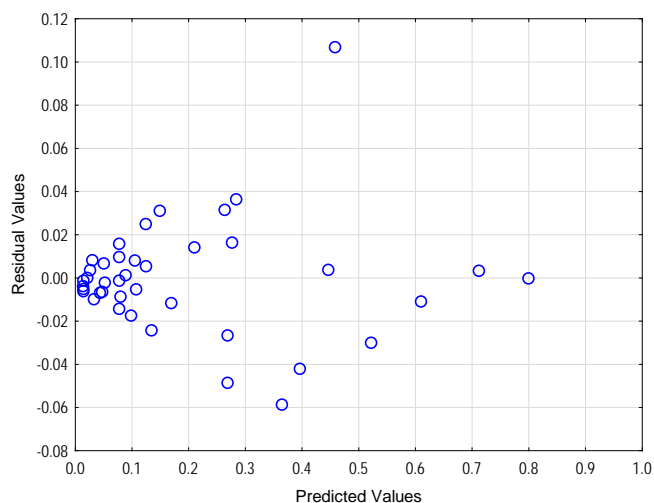


Fig. 2a Residual plot of quince sorption in the case of the M03 model

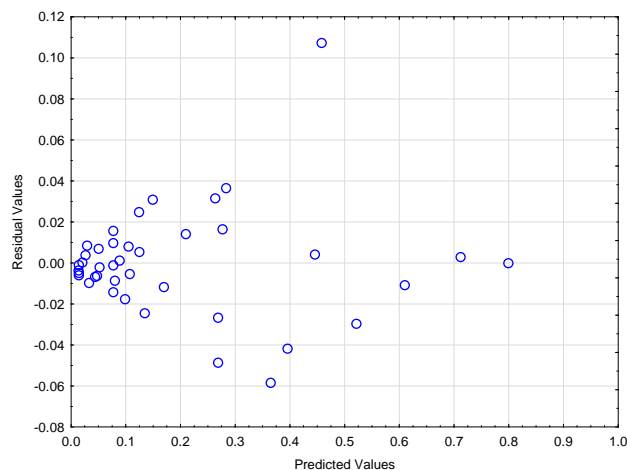


Fig. 2b Residual plot of quince sorption in the case of the M025 model

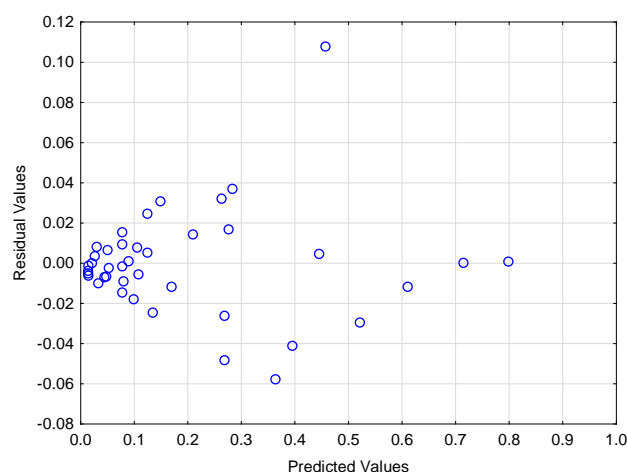


Fig. 2c Residual plot of quince sorption in the case of the M30 model

CONCLUSIONS

In this paper, the statistical performance of a total of thirty-two four-parameter sorption isotherm models was studied according to the equilibrium moisture content of quinces. The coefficient of determination and the graphical evaluation of residual randomness were utilized as the main assessment criteria for statistical evaluation of the sorption isotherm models considered. Of all the models considered, the Popovski&Mitrevski M25 and M30 models and the McLaren&Rowen M03 model exhibited the best statistical fit between the experimental and predicted equilibrium moisture contents of quinces at different water activity values.

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Received: 26.02.2020.

Accepted: 30.03.2020.