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STATISTICAL EVALUATION OF TWO-PARAMETER SORPTION ISOTHERM MODELS

STATISTIČKA OCENA DVOPARAMETARSKIH MODELA SORPCIONIH IZOTERMI

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ABSTRACT

Numerous mathematical models for approximating the water sorption data of food materials are available in the scientific literature. Depending on the number of parameters included, such models are referred to as one-parameter, two-parameter, three-parameter, or multi-parameter models. The objective of this study was to evaluate a total of sixty two-parameter sorption isotherm models for approximating the equilibrium moisture content data of quince and to compare the goodness of fit between the models examined. On the basis of the statistical analyses performed, it can be concluded that the Popovski&Mitrevski model has the best statistical performance of all the models examined. The coefficient of determination was the main criterion for assessing the model performance, totalling $R^2 = 0.9913$ for the best model performance.

Key words: *sorption isotherms, two-parameter model, statistical evaluation*

REZIME

Krive sorpcije i desorpcije su veoma važne pri definisanju ravnotežne vlažnosti i aktivnosti vode. Predviđanje promena ovih veličina u zavisnosti od uslova okolnog vazduha su veoma važne za sušenje i skladištenje poljoprivredg proizvoda. Izoterme sorpcije vlage opisuju odnos između ravnotežnog sadržaja vlage i aktivnosti vode pri konstantnim temperaturama i pritiscima. Za preradu prehraambenih materijala važne su sorpcione izoterme u modelovanju procesa sušenja, projektovanju i optimizaciji opreme za sušenje, predviđanju stabilnosti roka trajanja , izračunavanju promena vlažnosti , koje se mogu pojaviti tokom skladištenja i izborodgovarajućeg materijala za pakovanje . U radu su korišćeni esperimentalni podaci ispitvanja sorpcionih osobina dunje, odnosno sorpcionih krivih. Ove krive predstavljaju se matematičkim funkcija – modelima, čiji parametri se ispituju eksperimentima. Za aproksimaciju sorpcionih podataka prehrambenih materijala u naučnoj literaturi dostupni su brojni matematički modeli. Zavisno od broja parametara, modeli mogu biti jedno, dvo, tro ili više parametarski. U radu je prikazana statistička ocena šezdeset dvoparametarskih modela koji su korišćeni za aproksimaciju ravnotežne vlažnosti dunje. Analizirani modeli su preuzeti iz refrentne svetske literature. Na osnovu dobijenih rezultatata može se zaključiti da model Popovski & Mitrevski ima najbolje statističke performanse. Glavni kriterijum ocene bio je koeficijent determinacije čija je vrednost za najbolje ocenjeni model bila vrednost od R2 = 0,9913. Vrednosti statističkih parametara modela Popovski & Mitrevski procenjene su slaganjem modela sa eksperimentalnim podacima ispitivanja ravnotežne vlažnosti dunje korišćenjem Quasi-Newton metod procene koji minimizira greške sume kvadrata.

Ključne reči: izoterme sorpcije, dvoparametarski modeli, statistička ocena.

INTRODUCTION

Moisture sorption isotherms describe the relationship between the equilibrium moisture content and the water activity at constant temperatures and pressures. With regard to food materials, sorption isotherms are of great importance to modelling the drying process, designing and optimising the drying equipment, predicting the shelf-life stability, calculating changes in the moisture content during storage, and selecting the appropriate packaging material (*Gal, 1987*). Over the last two decades, an increasing number of studies have been reported in the scientific and engineering literature addressing the following issues: methods for determining sorption or desorption isotherms (*Mitrevski et al., 2015*; *Mitrevski et al., 2018*), temperature dependence between sorption isotherms (*Popovski&Mitrevski, 2004*), determination of sorption heat (*Kaymak-Ertekin and Gedik, 2004*), and mathematical models for approximating the moisture sorption of food materials (*Mitrevski et al., 2012*; *Mitrevski et al., 2015a*). Depending on the number of parameters included, models for approximating the moisture sorption data of materials are referred to as one-parameter, two-parameter, three-parameter, or multi-parameter models. In engineering calculations, the simplicity of a mathematical model, i.e. a model with a smaller number of parameters, is of great importance. When the sorption isotherm model is incorporated into the mathematical model for calculating the drying process or predicting the shelf-life of the packaged dried product, the approximation of the experimental data on the equilibrium moisture content is of particular significance (*Boquet et al., 1978*). The objective of the present study was to evaluate a total of sixty two-parameter sorption isotherm models for approximating the equilibrium moisture content data of quince and to compare the goodness of fit between the models examined on the basis of the coefficient of determination. The results obtained argue that the Popovski&Mitrevski model, i.e. the model M52 (*Popovski&Mitrevski, 2007*), has the best statistical performance.

MATERIAL AND METHOD

Quince samples for the determination of sorption isotherms were predried to the final moisture content in a convective dryer at an air drying temperature of 60 °C and an air drying velocity of 1 m/s for a period of 7 hours.The equilibrium moisture contents of the quince samples were determined at temperatures of 15, 30, 45 and 60 ^{o}C, using the static gravimetric method (*Mitrevski et al., 2018*). Ten saturated salt solutions L$_i$Cl, CH_3COOK, MgCl, K_2CO_3, $Mg(NO_3)_2$, 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 of 15, 30, 45 and 60 ^{o}C (with an accuracy of ± 0.1 ^{o}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*). Changes in the sample masses were recorded every 7 days using the electrical balance KERN PLJ360-3M with a precision of 0.001 g (Kern&Sohn GmbH, Ziegelei 1, 72336 Balingen, Germany). The equilibrium between the samples and their environments was reached after 21 days as evidenced by the constant weight of the samples after two successive measurements. The equilibrium moisture content of the samples was determined gravimetrically by drying in an oven at a temperature of 105^{o}C and atmospheric pressure for 24 h.

RESULTS AND DISCUSSION

The equilibrium moisture content values (X_{eq}) of quince slices (Table 1), obtained at different water activity values (a_w) and four different temperatures (*Mitrevski et al., 2018*), were fitted with sixty two-parameter sorption isotherm models M01-M60 (Table 2). In the scientific literature, the following statistical critera are used for the goodness of fit of a sorption isotherm model, i.e. the selection of the best sorption isotherm model: the coefficient of determination (R^2), the root mean squared error (*RMSE)*, and the mean relative deviation (*MRD)*. In this study, the coefficient of determination (R^2) was used as a guide for selecting the best two-parameter sorption isotherm model. Furthermore, a large number of numerical experiments were conducted because the accuracy of the estimated parameter values is greatly affected by the regression methods (indirect non-linear or direct non-linear), the estimation method, the initial step size, the initial parameter values, the convergence critera, and the function form. The method of indirect non-linear regression and the 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 (Statsoft Inc., Tulsa, OK, http://www.statsoft.com) were used to approximate the data obtained on the experimental equilibrium moisture content of quince. On the basis of the experimental data and each mathematical model in Table 2, the values of the coefficient of determination (R^2) were calculated. Subsequently, the models were ranked on the basis of the R^2 values obtained (Table 3).

*Table 1. Equilibrium moisture contents of the quince samples**

15^{o}C		30^{o}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^{o}C		60^{o}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 deviations were based on N = 3 replications

Table 2. Mathematical models for approximating the sorption data

Num. model	Name of model	Model	References
M01	BET modified	$X_{eq}=\frac{ABa_w}{[(1-a_w)(1-B\ln(1-a_w))]}$	Al-Muhtaseb et al., 2004
M02	Bradley	$X_{eq}=A\ln(-\ln a_w)+B$	Bradley, 1936
M03	Brunauer	$X_{eq}=\frac{Aa_w}{(1-a_w)(1-Ba_w)}$	Brunauer et al., 1938
M04	Caurie ≡ Miniowitsch	$X_{eq}=\exp(Aa_w+B)$	Caurie, 1970
M05	Chen and Clayton ≡ Bradley (for T=const)	$X_{eq}=A\ln(-\ln a_w)+B$	Chen and Clayton, 1971
M06	Chung-Pfost ≡ Bradley (for T=const)	$X_{eq}=A\ln(-\ln a_w)+B$	Chung and Pfost, 1967
M07	Chung-Pfost	$X_{eq}=A+B(\ln a_w)$	Chung and Pfost, 1967
M08	Day-Nelson ≡ Henderson (for T = const)	$X_{eq}=A\ln[-\ln(1-a_w)]+B$	Van den Berg and Bruin, 1981
M09	Dubinin-Radushkevich	$X_{eq}=\exp[A\ln(a_w^2)]B$	Dubinin and Radushkevich, 1947
M10	Freundlich ≡ McGavack-Patrick	$X_{eq}=Ba_w^A$	Freundlich, 1926
M11	Halsey	$X_{eq}=A(-\ln a_w)^B$	Halsey, 1948
M12	Halsey modified	$X_{eq}=A+B(\frac{a_w}{1-Ba_w})$	Iglesias and Chirife, 1981
M13	Harkins-Jura	$X_{eq}=\sqrt{\frac{A}{B-\ln a_w}}$	Jura and and Harkins, 1943
M14	Henderson	$X_{eq}=A[-\ln(1-a_w)]^B$	Henderson, 1952
M15	Hoover-Mellon ≡ Bradley	$X_{eq}=A\ln(-\ln a_w)+B$	Van den Berg and Bruin, 1981
M16	Hüttig	$X_{eq}=\frac{Aa_w(1+a_w)}{1-Ba_w}$	Hüttig,1948
M17	Iglesias-Chirife ≡ Halsey (for T=const)	$X_{eq}=B(-\ln a_w)^A$	Iglesias and Chirife, 1976

M18	Iglesias-Chirife	$X_{eq} = A + \frac{Ba_w}{1-a_w}$	Iglesias and Chirife, 1981
M19	Isse	$X_{eq} = \exp[\ln(\frac{A}{1-a_w}) + B]$	Isse et al., 1993
M20	Kühn	$X_{eq} = A + B / \ln a_w$	Kuhn, 1964
M21	Langumir	$X_{eq} = \frac{Aa_w}{1-Ba_w}$	Langumir, 1918
M22	Linear equation	$X_{eq} = A + Ba_w$	Castillo et al., 2003
M23	McGavack-Partric	$X_{eq} = Aa_w^B$	McGavack and Partric, 1920
M24	Miniowitsch	$X_{eq} = \exp(Aa_w + B)$	Lykow, 1958
M25	Mizrahi	$X_{eq} = \frac{A}{1-a_w} + B$	Mizrahi et al.,1970
M26	Mizrahi modified	$X_{eq} = A\frac{a_w}{1-a_w} + B\frac{1}{1-a_w}$	Castillo et al., 2003
M27	Oswin	$X_{eq} = A(\frac{a_w}{1-a_w})^B$	Oswin, 1946
M28	Pierce	$X_{eq} = A\ln(1-a_w) + B$	Pierce and Inst, 1929
M29	Polanyi	$X_{eq} = A(-\ln a_w)^{-1/3} + B$	Polanyi, 1928
M30	Popovski-Mitrevski	$X_{eq} = \frac{Aa_w}{(1-Ba_w)^2}$	Popovski and Mitrevski, 2003
M31	Popovski-Mitrevski	$X_{eq} = A\ln[-\ln(1-a_w)] + B$	Popovski and Mitrevski, 2006
M32	Popovski-Mitrevski	$X_{eq} = A\ln\frac{a_w}{1-a_w} + B$	Popovski and Mitrevski, 2006
M33	Popovski-Mitrevski	$X_{eq} = \frac{A\sin a_w}{1-a_w} + B$	Popovski and Mitrevski, 2007
M34	Popovski-Mitrevski	$X_{eq} = \frac{A\cos a_w}{\sin(1-a_w)} + B$	Popovski and Mitrevski, 2007
M35	Popovski-Mitrevski	$X_{eq} = \frac{Atga_w}{1-a_w} + B$	Popovski and Mitrevski, 2007
M36	Popovski-Mitrevski	$X_{eq} = \exp\left[\frac{A}{\sin a_w} + B\right]$	Popovski and Mitrevski, 2007
M37	Popovski-Mitrevski	$X_{eq} = \exp\left[\frac{A\sin(1-a_w)}{a_w} + B\right]$	Popovski and Mitrevski, 2007
M38	Popovski-Mitrevski	$X_{eq} = \exp\left(\frac{A}{tga_w} + B\right)$	Popovski and Mitrevski, 2007
M39	Popovski-Mitrevski	$X_{eq} = \frac{1}{\frac{A}{\sin a_w} + B}$	Popovski and Mitrevski, 2007
M40	Popovski-Mitrevski	$X_{eq} = \frac{a_w}{A\sin(1-a_w) + Ba_w}$	Popovski and Mitrevski, 2007
M41	Popovski-Mitrevski	$X_{eq} = \frac{1}{\frac{A}{tga_w} + B}$	Popovski and Mitrevski, 2007
M42	Popovski-Mitrevski	$X_{eq} = A\left(\frac{\sin a_w}{1-a_w}\right)^B$	Popovski and Mitrevski, 2007
M43	Popovski-Mitrevski	$X_{eq} = Atg^B a_w$	Popovski and Mitrevski, 2007
M44	Popovski-Mitrevski	$X_{eq} = A\left(\frac{tga_w}{1-a_w}\right)^B$	Popovski and Mitrevski, 2007
M45	Popovski-Mitrevski	$X_{eq} = \frac{A}{\arcsin(1-a_w)} + B$	Popovski and Mitrevski, 2007
M46	Popovski-Mitrevski	$X_{eq} = \frac{A}{\arccos a_w} + B$	Popovski and Mitrevski, 2007
M47	Popovski-Mitrevski	$X_{eq} = \frac{Aa_w}{arctga_w} + B$	Popovski and Mitrevski, 2007
M48	Popovski-Mitrevski	$X_{eq} = \exp(\frac{A}{\arcsin a_w} + B)$	Popovski and Mitrevski, 2007
M49	Popovski-Mitrevski	$X_{eq} = \exp\left(\frac{A}{arctga_w} + B\right)$	Popovski and Mitrevski, 2007
M50	Popovski-Mitrevski	$X_{eq} = \exp\left[\frac{Aarctg\ (1-a_W)}{a_W} + B\right]$	Popovski and Mitrevski, 2007
M51	Popovski-Mitrevski	$X_{eq} = \frac{1}{\frac{A}{\arcsin a_w} + B}$	Popovski and Mitrevski, 2007
M52	Popovski-Mitrevski	$X_{eq} = \frac{1}{\frac{A}{arctga_w} + B}$	Popovski and Mitrevski, 2007
M53	Popovski-Mitrevski	$X_{eq} = \frac{a_W}{Aarctg \cdots (1-a_W) + Ba_W}$	Popovski and Mitrevski, 2007
M54	Popovski-Mitrevski	$X_{eq} = A\arcsin^B a_w$	Popovski and Mitrevski, 2007
M55	Popovski-Mitrevski	$X_{eq} = A(\frac{a_w}{\arccos a_w})^B$	Popovski and Mitrevski, 2007
M56	Popovski-Mitrevski	$X_{eq} = A(\frac{a_w}{arctga_w})^B$	Popovski and Mitrevski, 2007
M57	Posnow	$X_{eq} = \frac{A}{B - \ln a_w}$	Lykow, 1958
M58	Smith ≡ Pierce	$X_{eq} = A\ln(1-a_w) + B$	Smith, 1947
M59	Thompson ≡ Henderson (for T=const)	$X_{eq} = A\ln[-\ln(1-a_W)] + B$	Thompson et al., 1968
M60	White-Eyring	$X_{eq} = \frac{A}{1-Ba_w}$	White and Eyring, 1947

Table 3.Statistical summary of the regression analysis

Model	R^2	Rank	Model	R^2	Rank
M01	0.9897	8	M31	0.8121	59
M02	0.9278	50	M32	0.8796	55
M03	0.9867	18	M33	0.9860	21
M04	0.9855	22	M34	0.9861	20
M05	0.9278	51	M35	0.9785	32
M06	0.9278	52	M36	0.9593	47
M07	0.7251	60	M37	0.9644	43
M08	0.8121	58	M38	0.9670	41
M09	0.9749	34	M39	0.8297	57
M10	0.9749	35	M40	0.9910	3
M11	0.9853	25	M41	0.9905	7
M12	0.9088	54	M42	0.9892	15
M13	0.9375	48	M43	0.9832	31
M14	0.9897	9	M44	0.9896	12
M15	0.9280	49	M45	0.9769	33
M16	0.9907	5	M46	0.9879	17
M17	0.9853	26	M47	0.9183	53
M18	0.9844	27	M48	0.9699	40
M19	0.9702	38	M49	0.9603	46
M20	0.9854	24	M50	0.9656	42
M21	0.9911	2	M51	0.9893	14
M22	0.8593	56	M52	0.9913	1
M23	0.9749	36	M53	0.9908	4
M24	0.9855	23	M54	0.9841	30
M25	0.9844	28	M55	0.9907	6
M26	0.9844	29	M56	0.9880	16
M27	0.9894	13	M57	0.9866	19
M28	0.9642	44	M58	0.9642	45
M29	0.9702	39	M59	0.9897	11
M30	0.9897	10	M60	0.972	37

As can be seen in Table 3, the highest coefficient of determination vlaue (R^2 = 0.9913; rank 1) was recorded in the instance of the Popovski&Mitrevski model (M52) (*Popovski&Mitrevski, 2007*). Consequently, this model correlates the experimental values of quince sorption better than other models. Of all the models examined, the model of Chung and Pfost (M07) (*Chung and Pfost, 1967*) had the lowest value of the coefficient of determination (R^2 = 0.7251; rank 60), i.e. the worst statistical performance.

The parameter values A and B for the Popovski&Mitrevski model (M52) were estimated by fitting the model to experimental data obtained on the equilibrium moisture content of quince using the quasi-Newton estimation method, which minimizes the sum squares errors. The estimated values of the parameters A and B are shown in Table 4.

Table 4. Estimated values of the parameters A and B

Model	A	B
XEQ=1/(A/ARCTAN(AW)+B)	11.12	-13.72

XEQ - equlibrium moisture content, AW- water activity

The experimental and predicted values of the quince equilibrium moisture content at four temperatures are shown in Figure 1.

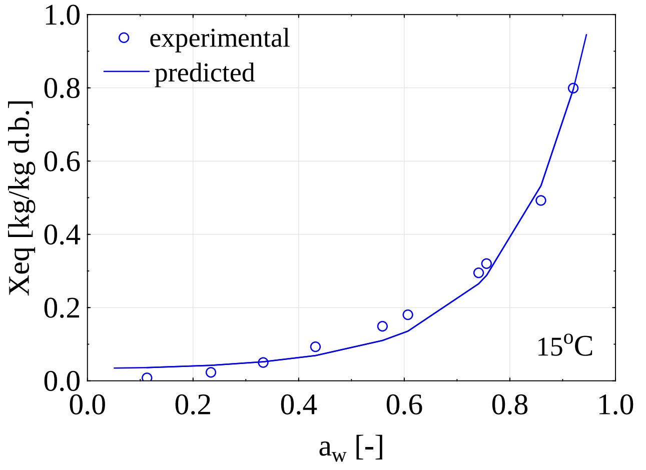

Fig. 1a Experimental and predicted quince sorption isotherms at 15 °C for the model M52

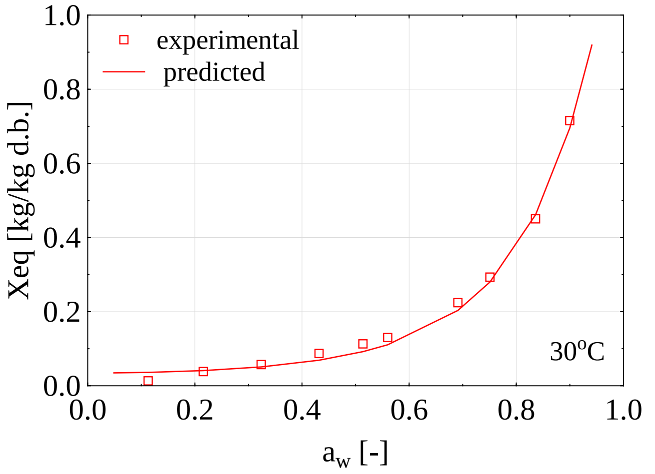

Fig. 1b Experimental and predicted quince sorption isotherms at 30 °C for the model M52

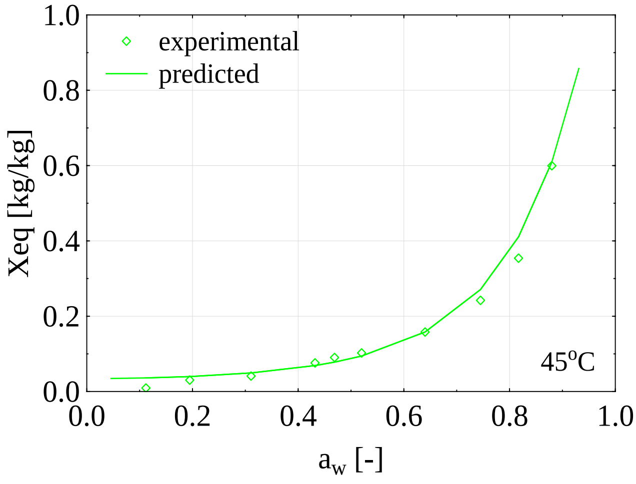

Fig. 1c Experimental and predicted quince sorption isotherms at 45 °C for the model M52

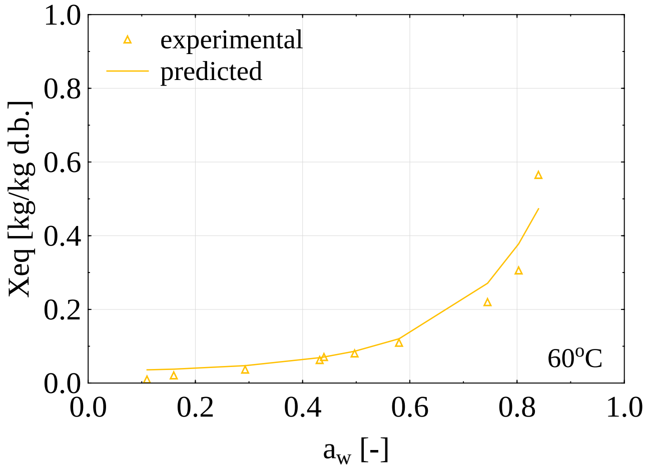

Fig. 1d Experimental and predicted quince sorption isotherms at 60 °C for the model M52

As can be seen in Figures 1a-1d, the model M52 exhibits a good agreement between the experimental and predicted values of the equilibrium moisture content of quince.

CONCLUSION

The experimantal data obtained on the equilibrium moisture content of quince were fitted with sixty two-parameter sorption isotherm models. On the basis of the statistical criteria proposed in the present study, it can be concluded that the Popovski&Mitrevski model (M52) exhibits the best agreement between the experimental and predicted values of the equilibrium moisture content of quince throughout the entire range of water activity observed.

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