A BASIC APPROACH TO THE VERIFICATION AND VALIDATION OF SORPTION ISOTHERM MODELS

BAZNI PRISTUP VERIFIKACIJI I VALIDACIJI MODELA IZOTERME SORPCIJE

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ABSTRACT

In this paper, a procedure for the statistical verification and validation of a newly generated sorption isotherm M01 model is defined. A basic approach to the evaluation of a model for approximating experimental sorption data is presented. Based on the accuracy within an acceptable range, a model is considered valid for a set of experimental conditions conducive to its intended purpose. The matching of experimental data to the data obtained in the calculations was examined. Several statistical criteria, proposed in the literature, and indicators (the coefficient of determination (R^2), the root mean squared error (RMSE) and the mean relative deviation (MRD)) were used for the quantitative verification of the newly generated sorption isotherm model. A qualitative statistical analysis was performed by the validation of statistical assumptions relative to the residuals obtained from the regression analysis. This was achieved by creating a normal quantile-quantile diagram with a normal residual distribution, and a Scatter plot diagram with a random residual distribution. Fitted with the newly generated three-parameter sorption isotherm model, the validation of the experimental data on the sorption isotherms of pear flesh tissue (determined at four temperatures 15°C, 30°C, 45°C and 60°C, and over a range of water activity from 0.110 to 0.920) was performed relative to the results of the Guggenheim, Anderson and De Boer (GAB) model. The success of the newly generated model was confirmed in relation to this reference model.

Key words: verification, validation, sorption isotherm model.

REZIME

U ovom radu definisan je postupak statističke verifikacije i validacije novogeneriranog modela izoterme sorpcije. Prikazan je osnovni pristup evaluacije uspeha modela za aproksimaciju eksperimentalnih sorpcionih podataka. Tačnost modela u okviru prihvatljivog opsega je osnova da bi se jedan model smatrao validnim za set eksperimentalnih uslova, potrebnih za njegovu namenu. Ispitivani su eksperimentalno dobijeni podaci sa izračunatim podacima. Korišćeno je nekoliko statističkih kriterijuma predloženih u naučnoj literaturi i više statističkih pokazatelja (koeficijent determinacije (R^2), srednja kvadratna greška (RMSE) i srednja relativna devijacija (MRD)), za kvantitativnu verifikaciju novogenerisanog modela izoterme sorpcije. Kvalitativna statistička analiza izvršena je proverom nekoliko statističkih pretpostavki u vezi sa rezidualima dobijenim iz regresione analize. Ovo je realizovano stvaranjem normalnog dijagrama quantile-quantile koji izražava normalnu raspodelu i Scatter plot dijagrama koji izražava slučajnu raspodelu ostataka. Validacija eksperimentalnih podataka izotermnih sorpcija kruške (određivanja na četiri temperature 15°C, 30°C, 45°C i 60°C, u opsegu aktivnosti vode od 0.110 do 0.920), aproksimirana novogeneriranim triparametarskim modelom izoterme sorpcije, poređena je sa rezultatima referentnog Andersonovog modela, poznatijeg u naučnoj literaturi pod imenom GAB (Guggenheim - Anderson - de Boer) model. Uspeh aproksimacije novoformiranog modela u odnosu na referentni model je potvrđen.

Ključne reči: verifikacija, validacija, model izoterme sorpcije.

INTRODUCTION

The verification and validation processes are essential for every newly generated sorption isotherm model. Such model can be successfully used for the approximation of sorption data on food materials. The development of a new mathematical model is always associated with determining whether the model and its results are “correct” for a specific use or purpose [1]. Admittedly, the term “successful” with regard to models is largely subjective. However, from the modeling performance perspective, this criterion is based on the matching of the experimentally obtained data to the data calculated. When this comparison produces results close to each other, the model is considered to achieve a verified simulation, as well as a valid presentation of the results. Model verification is defined as “ensuring that the computer program of the computerized model and its implementation are correct” [2]. The verification process in fact involves testing, finding and fixing the errors that occur during the implementation of the model. Various techniques are used to check the suitability of a particular model: comparison with a

model that has been previously checked, creating diagrams and evaluating the output data depending on the change in the input parameters [3]. Model validation is defined as the “substantiation that a computerized model, within its domain of applicability, possesses a satisfactory range of accuracy consistent with the intended application of the model” [2]. Determining a specific purpose of the model and its validity are of particular importance. A model is considered valid for a set of experimental conditions if its accuracy is within the acceptable range pertinent to the model. This model may be valid for one set of experimental conditions and invalid for another [1]. The required accuracy should be specified prior to starting the development of a model, or very early in the model development process, so several versions have been previously developed in order to obtain a model with satisfactory validity.

The following types of validation are generally used:

- conceptual validation - in the case when the expected confidentiality of the model is assessed, and
- validation of the results - the results or outputs of the newly generated model validated are compared to the results of an appropriate reference model.

Fig. 1. Adsorption isotherms of pear tissue

The following objectives were established for this study:

1. to define a procedure for the statistical verification and validation of a newly generated sorption isotherm model, and
2. to confirm the success of the newly generated sorption isotherm model in relation to the Anderson (GAB) reference model.

MATERIALS AND METHODS

A sorption isotherm describes the thermodynamic relationship between water activity and the equilibrium of moisture content at a constant temperature and pressure. The moisture sorption isotherms of different food materials are extremely important in the modeling of the drying process, the design and optimization of drying equipment, the prediction of shelf-life stability, the calculation of moisture changes which may occur during storage, and the selection of appropriate packaging material [4]. Furthermore, the knowledge of the sorption data is essentially useful for predicting the microbiological, enzymatic and chemical stability of food

materials. The experimental determination and modeling of the sorption isotherms of food materials have inspired numerous researches due to their valuable industrial uses. Several researches have reported the sorption data on pear tissues obtained at different temperatures and water activities. Kiranoudis et al. [5] determined the sorption isotherms of pear tissue at two temperatures (25 °C and 45 °C), whereas Guine et al. [6] determined the sorption isotherms of pear tissue at 20, 25, and 30 °C. Moreover, Lahsasni et al. [7] determined the sorption isotherms of prickly pear fruit tissue at three temperatures (30 °C, 40 °C, and 50 °C) over a range of relative humidity from 0.05 to 0.90. New researches conducted by Guine [8] determined the desorption isotherms of two pear tissue varieties at three temperatures (20, 30, and 40 °C), whereas Djendoubi et al. [9] determined the desorption isotherms of pear tissue at three temperatures (30, 45, and 60 °C) and water activities ranging from 0.07 to 0.98. For the purpose of this paper, the experimental data base of the equilibrium pear moisture content was created at four different temperatures (15°C, 30°C, 45°C, 60°C) and water activities (0.110-0.920) (Table 1) [3].

The sorption isotherms of pear tissue at four temperatures were determined by using the static gravimetric method, developed and standardized in the European COST 90 Project [10]. Ten saturated salt solutions (LiCl, CH₃COOK, MgCl, K₂CO₃, Mg(NO₃)₂, NaBr, SrCl₂, NaCl, KCl, and BaCl₂), prepared according to the Greenspan recommendation [11], were used for a defined constant water activity ranging from 0.112 to 0.920 [12].

The adsorption isotherms of pear tissue at temperatures of 15, 30, 45 and 60°C are shown in Figure 1 [13].

Sorption isotherm models

Many sorption isotherm models are available in the scientific literature for approximating the

Table 1. Equilibrium moisture content for pear tissue*

15°C		30°C		45°C		60°C	
a_w [-]	u_r [kg/kg]	a_w [-]	u_r [kg/kg]	a_w [-]	u_r [kg/kg]	a_w [-]	u_r [kg/kg]
0.113	0.011±0.000	0.113	0.007±0.000	0.112	0.008±0.000	0.110	0.009±0.001
0.234	0.018±0.000	0.216	0.016±0.001	0.195	0.018±0.000	0.160	0.014±0.002
0.333	0.040±0.001	0.324	0.040±0.000	0.311	0.040±0.000	0.293	0.031±0.003
0.432	0.083±0.001	0.432	0.080±0.001	0.432	0.074±0.001	0.432	0.070±0.002
0.559	0.159±0.000	0.514	0.119±0.000	0.469	0.091±0.002	0.440	0.074±0.002
0.607	0.197±0.002	0.560	0.145±0.002	0.520	0.107±0.000	0.497	0.090±0.003
0.741	0.325±0.001	0.691	0.250±0.000	0.640	0.172±0.001	0.580	0.130±0.002
0.756	0.350±0.002	0.751	0.320±0.003	0.745	0.265±0.000	0.745	0.235±0.001
0.859	0.600±0.001	0.836	0.495±0.001	0.817	0.395±0.002	0.803	0.335±0.001
0.920	0.904±0.003	0.900	0.813±0.002	0.880	0.653±0.002	0.840	0.597±0.001

* mean and standard deviation based on N = 3 replication

experimental data on the sorption isotherms of food materials [14; 15; 16; 17]. Popovski and Mitrevski [18; 19; 20; 21; 22; 23; 24] proposed seven methods for generating new sorption models. These models have been developed on the basis of different rational, exponential, logarithmic, trigonometric or cyclometric functions. Each of those generated models has proven successful to various extents in the approximation of the equilibrium moisture data of a given type of food material within an appropriate range of a_w . Van den Berg and Bruin (1981) [25] conducted a survey of 77 models classified into three groups: theoretical, partially theoretical and fully empirical. The power series of Jaroniec (1975) [26], which represents a family of water sorption isotherm models, was also included:

$$u_r = \exp\left(\sum_{i=1}^n P_i \ln^{i-1} a_w\right) \quad (1)$$

Instead of the logarithmic function $\ln(a_w)$, any monotonous function $F(a_w)$ can be used with a constant sign into the interval (0,1). Consequently, the Jaroniec power series can be generalized as follows:

$$u_r = \exp\left[\sum_{i=1}^n P_i F^{i-1}(a_w)\right] \quad (2)$$

One of the families, which were obtained by using a different monotonous functions $F(a_w)$ in Eq. (1), is expressed as follows:

$$u_r = \exp\left[\sum_{i=1}^n P_i \left(\frac{\sin a_w}{a_w}\right)^{i-1}\right] \quad (3)$$

The following three-parameter model was created for this family [27]:

$$\textbf{M01 Model} \quad u_r = \exp\left[P_1 + P_2 \frac{\sin a_w}{a_w} + P_3 \left(\frac{\sin a_w}{a_w}\right)^2\right] \quad (4)$$

To examine the statistical efficiency of the M01 model, a comparison with the Anderson (GAB) model [28] was made:

$$u_r = \frac{P_1 a_w}{(1 - P_2 a_w)(1 - P_3 a_w)} \quad (5)$$

One of the objectives of this study was to make an evaluation of the suitability of this newly generated three-parameter model for approximating the equilibrium moisture data of pear tissue, as well as to compare its goodness of fit based on several statistical criterions.

Basic procedure for statistical verification and validation

Statistical verification and validation are equally important when developing a new mathematical model. Although the evaluation of a sorption model seems very simple and precisely determined, a number of difficulties may occur in a correct interpretation of the results obtained when comparing the experimental with the calculated data. These differences can be caused by various reasons:

- deficiencies in the structure of the model and the assumptions used,
- errors and deviations in the input data of the model,
- irregularities in the measurement procedure,
- uncertainties related to the measurement procedure and input values,
- incorrect interpretation of the output values.

In accordance with the model specifics, different evaluation procedures are applied to different models. Prior to evaluating a particular model, it is necessary to accurately define the objectives to be achieved. Specific objectives impose the need to evaluate different parameters and apply different criteria for estimating the performance of the model [29].

In the scientific literature, the following statistical criteria are used for the goodness of fit of the experimental sorption data and the selection of the best isotherm model: the coefficient of determination (R^2), the root mean squared error (RMSE) and the mean relative deviation (MRD). The selection of a sorption isotherm model with a graphical evaluation of the residual randomness is also frequently utilized [30; 31].

In this paper, quantitative and qualitative statistical analyses of the results were performed, and several statistical criterions or statistical parameters were used. The value of the performance index (ϕ), which is calculated on the basis of the R^2 , RMSE and MRD values, is **the first statistical criterion** for the estimation of a sorption isotherm model [31]:

$$\phi = \frac{R^2}{\text{RMSE} \cdot \text{MRD}} \quad (6)$$

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

The second statistical criterion for a successful approximation of the sorption model is the validation of certain statistical assumptions relative to the residuals obtained from the regression analysis. *The first statistical assumption* is that the population of the residuals obtained from the regression analysis is distributed according to the normal distribution. The validation of this assumption, with a normal error distribution, is performed graphically by creating a normal quantile-quantile diagram. Provided equilibrium moisture content errors are close to a straight line, the distribution of the population does not deviate from the normal distribution. However, due to the subjective character of graphic evaluation, the interpretation of the diagram results should be underpinned by the results obtained from a formal test. Several tests are used in the scientific literature [32]:

- single-sample test, which is used to evaluate the skewness and kurtosis of the residual population,
- Shapiro-Wilk test,
- G-kurtosis test,
- D'Agostino-Pearson test [33],
- Kolmogorov-Smirnov test.

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 the residual population of skewness (z_1) and kurtosis (z_2), and is the second adequate statistical criterion for the selection of sorption models. The test statistic for the D'Agostino-Pearson's test of normality is computed using the following equation, [32]:

$$\chi^2 = z_1^2 + z_2^2 \quad (7)$$

The χ^2 statistics has a chi-squared distribution with 2 degrees of freedom (df). The tabled critical 0.05 chi-square value for $df = 2$ is $\chi_{0.05}^2 = 5.99$. 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 [32]. Because the χ^2 statistics is not recommended

individually as an adequate measure of the effectiveness of a sorption model to describe the experimental sorption data, an additional statistical criterion must be used.

The second statistical assumption of regression analysis refers to the random distribution of the residuals. The validation of this assumption is performed by drawing a Scatter plot diagram, showing the dependence of the equilibrium moisture content residuals from water activity as an independent variable [3]. The single-sample run test is one of the numerous statistical procedures that have been developed for evaluating the randomness of series distribution. This test is a statistical criterion for the effectiveness of a sorption model. The test evaluates the number of runs in a series in which, on each trial, the outcome must be one out of $k = 2$ alternatives. 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 [32]:

$$z_r = \frac{|g - g_1| - 0.5}{\sigma_r}; \quad g_1 = \frac{2n_1n_2}{n_1 + n_2} + 1; \quad \sigma_r = \sqrt{\frac{2n_1n_2(2n_1n_2 - n_1 - n_2)}{(n_1 + n_2)^2(n_1 + n_2 - 1)}}$$

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 [32].

The third statistical criterion for the selection of an sorption isotherm model is the evaluation of the significance and precision of the model constant. This is performed by constructing the individual confidence intervals (CI), accompanied by the calculation of the two-tailed P-values of the parameters estimated. Provided the parameters values obtained are outside the 95 % CI or the estimated two-tailed P-value, according to the (t) test of statistic ($p < 0.05$), the model contains irrelevant parameters for the approximation of experimental sorption data, i.e. the sorption isotherm model should be rejected.

Accordingly, a procedure for statistical verification of a particular sorption model is defined [31]:

1. Selecting a model that has the greatest value of the performance index, ϕ .
2. Provided the equilibrium moisture content residuals show a significant deviation from the normal distribution, the model should be rejected, i.e. should not be taken into account in further statistical evaluation.
3. Provided significant deviations of the equilibrium moisture content residuals from the random distribution are recorded, the model should be rejected by further statistical evaluation due to the subjectivity of the assessment.
4. The use of the D-optimum criterion enables obtaining a smaller confidence region and a better precision of the parameters defined. Provided the calculated parameters have no statistical significance, the sorption model should be rejected by further statistical evaluation. Consequently, the parameters values obtained are inadequate, or the experimental data of the equilibrium moisture content are inadequate for determining the parameters in the model. In some instances, the simplification of the model can be applied.
5. Testing the statistical significance and precision of the parameters determined by constructing the individual confidence intervals (CI).

RESULTS AND DISCUSSION

In accordance with the defined procedure, the statistical verification of the newly generated triparametric sorption model was conducted. The model verification consisted of quantitative and qualitative statistical analyses of the performance of the generated model and the reference model. The values of the coefficient of determination (R^2), root mean squared error (RMSE), mean relative deviation (MRD), and performance index (ϕ) computed for the newly generated sorption isotherm model and the Anderson (GAB) reference model are presented in Table 2, [3].

Table 2. Calculated values of statistical indicators for pear tissue

Model	R^2	RMSE	MRD	ϕ	Range
M01	0.9819	0.0321	0.5555	55.052	2
GAB	0.9860	0.0282	0.1526	229.18	1

The models were ranked on the basis of the calculated values of the performance index. It is noteworthy that the Anderson reference model, i.e. the GAB model, has a higher value of the performance index ($\phi = 229.18$). Therefore, in accordance with the first statistical criterion, the Anderson model best approximates the experimental data on the equilibrium moisture content of pear tissue. The computed average values for χ^2 and z_r are shown in Table 3.

Table 3. Rejection criteria for sorption models

Model	χ^2	z_r	Rejection criteria
M01	0.8907	1.7155	–
GAB	7.1785	3.0708	χ^2, z_r

“–” the model is not rejected by further statistical analysis

Table 3 clearly indicates that the newly generated M01 model has smaller values of χ^2 and z_r than the tabled critical values ($\chi_{0.05}^2 = 5.99$; $z_{0.05} = 1.96$). For these reasons and in accordance with the second statistical criterion, only the M01 model is appropriate for further statistical verification.

In Table 4, the estimated values of parameters, 95 % CI and two-tailed p-value of estimated parameters for each of the two models are given. The nlparci (beta, resid, “jacobian”, J) function of the Statistics Toolbox of Matlab 8.3 (The MathWorks Inc., Natick, MA) was used for the 95 % CI determination, whereas the significance of each of the estimated parameters (P_1, P_2, P_3) was evaluated through the t-test statistic.

Table 4. Estimated parameters values, 95 % CI and p values

Model	Parameter	Value	95% CI	p
M01	P_1	23.371	(-10.059 56.800)	1.65E-01
	P_2	-29.494	(-103.41 44.421)	4.24E-01
	P_3	2.7145	(-38.118 43.545)	8.94E-01
GAB	P_1	0.0861	(0.0559 0.1160)	1.20E-06
	P_2	0.3752	(-0.0621 0.8099)	9.07E-02
	P_3	0.9432	(0.9000 0.9903)	1.01E-17

* Bold values show that calculated parameters have no statistical significance

Table 4 clearly shows that the calculated two-tailed p-values for all the parameters of the GAB reference model are extremely small ($p < 0.05$). In accordance with the third statistical criterion, the values of these parameters have no statistical significance. Conversely, all the parameters of the M01 model are larger than the critical ones ($p > 0.05$).

Given that only the M01 model "survived" the elimination statistical criteria defined in the procedure for statistical verification of a sorption model, further qualitative analyses refer only to this model.

The quantile-quantile diagram in Figure 2 shows that the errors of the equilibrium moisture content regression are close to a straight line, which means that they do not deviate from the normal distribution.

Fig. 2. Normal quantile-quantile diagram of the equilibrium moisture content residuals - Model M01 (pear)

The Scatter plot diagram in Figure 3 shows the dependence between the equilibrium moisture content residuals and water activity.

Fig. 3. Scatter plot diagram of the equilibrium moisture content residuals - Model M01 (pear)

According to the second assumption of the regression analysis, the M01 model successfully approximates the experimental data of the equilibrium moisture content for pear tissue due to the lack of a schematic distribution of the regression residuals.

The experimentally obtained data and the calculated data of the pear equilibrium moisture content are shown in Figure 4, approximated using the M01 model at temperatures of 15, 30, 45 and 60 °C.

In Figure 4, a good matching of the experimental values to the calculated values of the pear equilibrium moisture content is evident in approximation to the M01 model.

Fig. 4. Experimental and calculated values of the equilibrium moisture content of pear tissue

CONCLUSIONS

Every simulation of sorption processes presents a new and unique challenge with regard to the verification and validation of the model employed. Therefore, a basic approach to evaluating the success of the model is defined in this paper, as well as a procedure for statistical model verification and validation. In accordance with this procedure, the verification of a newly generated triparametric sorption isotherm model for approximating the experimental sorption data of the pear equilibrium moisture content was conducted. Several statistical criteria and indicators were used for the quantitative verification performed, whereas the qualitative verification included a number of statistical assumptions relative to the regression residuals. The validation of the results obtained for the newly generated model was made by comparing these results to the GAB reference model results, thus the success of the new model was confirmed. The comparison of the calculated and experimental values of the pear equilibrium moisture content shows a high degree of matching, provided they are approximated using the M01 model, in keeping with the defined statistical verification procedure.

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