

Modelling Drying Time of *Candesartan Cilixetil* Powder Using Computational Intelligence Technique

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Abstract

The aim of this work was to use two computational intelligence techniques, namely, artificial neural network (ANN) and support vector regression (SVR), to model the drying time of a pharmaceutical powder *Candesartan Cilixetil*, which is used for arterial hypertension treatment and heart failure. The experimental data set used in this work has been collected from previously published paper of the drying kinetics of *Candesartan Cilixetil* using vacuum dryer and under different operating conditions. The comparison between the two models has been conducted using different statistical parameters namely root mean squared error (RMSE) and determination coefficient (R^2). Results show that SVR model shows high accuracy in comparison with ANN model to predict the non-linear behaviour of the drying time using pertinent variables with $\{R^2 = 0.9991, RMSE = 0.262\}$ against $\{R^2 = 0.998, RMSE = 0.339\}$ for SVR and ANN, respectively.

Keywords

Candesartan Cilixetil, response surface methodology, vacuum drying, artificial neural networks, support vector regression

1 Introduction

Different studies have been proposed recently in literature concerning the use of different drying technologies and equipment to preserve the final quality of diverse products such as pharmaceuticals.¹ The main objective of the drying process is to remove the impregnated humidity in the products to prevent any type of contamination and alleviate their weight to facilitate their transportation and caking. Taking into account that pharmaceutical powders are heat sensitive, the choice of an appropriate dryer depends on the properties of the powders. Numerous researchers have investigated the effect of diverse operating conditions, namely, vacuum pressure and temperature, on the drying time of various powders.^{2–4} Since drying experiments are generally costly and tedious, the exact mathematical representation of such complex and highly non-linear behaviour of the drying phenomena, which is usually established based on a lot of hypothesis and multivariable interaction is difficult. Some computational models show their ability to alleviate above limitations and fit and control the drying processes accurately.^{5,6} The use of artificial neural networks and support vector machines to model different governing parameters of the drying process of different products and dryers has gained growing interest.⁷ For instance, ANNs have been used successfully to model the moisture content of quince slices,⁸ also used to study the effect of different drying techniques on different types of root vegetables,⁹ another ANN model predicts the dehydration kinetics of pineapple,¹⁰ and an ANN has been used to optimise the drying process of kiwifruit slices in pulsed vacuum dry-

ing.¹¹ In comparison to ANN modelling, SVR is known for its simplicity and optimisation adaptability and handling the complex parameters,¹² for instance, polynomial SVM was employed to estimate the experimental drying performance parameters,¹³ also it was applied successfully to describe the drying kinetics of persimmon fruit (*Diospyros kaki*) during vacuum and hot air drying process.¹⁴

To our knowledge, few works have been focused on modelling of quality indicators of pharmaceutical powders using machine learning techniques ANN and SVR. Therefore, the novelty of this work is to model the drying time of an active ingredient “*Candesartan Cilixetil*” using artificial neural networks and support vector regression.

2 Results and discussion

2.1 Design of experiments

The experimental data for vacuum oven drying kinetics of a thin-layer of active ingredient was reported in our previously published article.¹ MODDE software was used to schedule experiments and generate a second-order fitting model with optimised coefficients. The equation of the response surface methodology (RSM) can be written in the following form:

$$\begin{aligned} \text{Drying time (min)} = & 84.5402 - 10.679m_0 + \\ & + 0.7556X_0 + 61.2037p - 2.91914T + \\ & + 6.02469m_0^2 + 0.02247X_0^2 - 2.9321p^2 - 0.0273T^2 + (1) \\ & + 0.2111X_0m_0 + 10.5556m_0p - 0.08333m_0T - \\ & - 0.08333X_0p + 0.003889X_0T - 0.08472pT \end{aligned}$$

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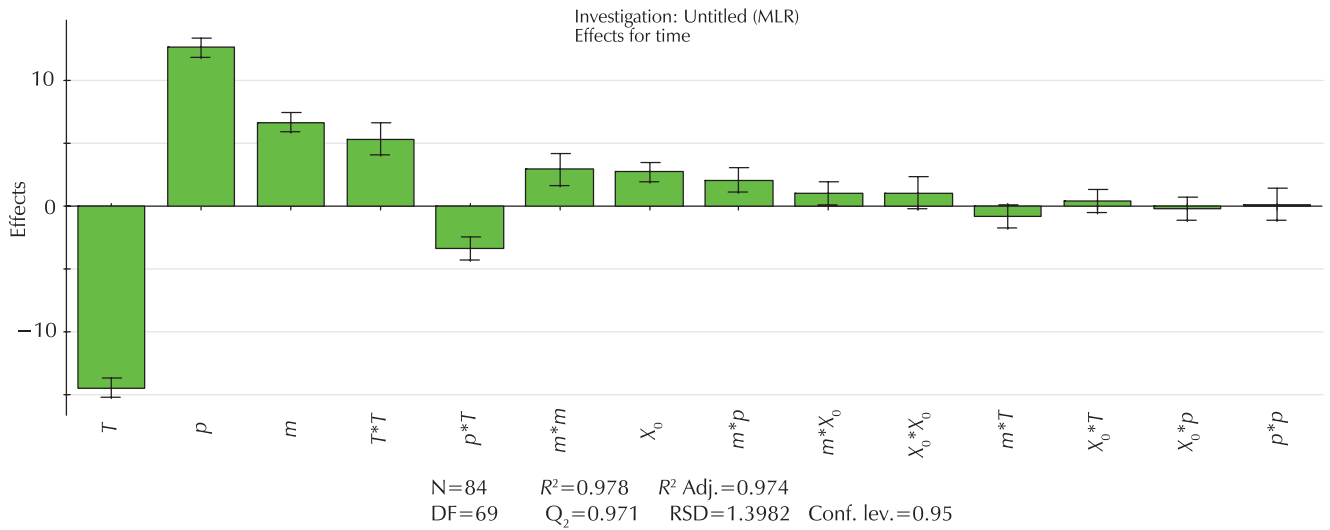


Fig. 1 – Effects of different input combinations on drying time

From this equation and based on the results found by response surface methodology (see Fig. 1), temperature is shown as the most influencing parameter on drying time. The accuracy of the three models was tested using R^2 and RMSE as expressed in the following equations:

$$R^2 = 1 - \frac{\sum_{i=1}^N (t_{cal} - t_{exp})^2}{\sum_{i=1}^N (t_{exp} - \bar{t}_{exp})^2} \quad (2)$$

$$RMSE = \sqrt{\frac{1}{N} \sum_{i=1}^N (t_{exp} - t_{cal})^2} \quad (3)$$

Fig. 2 depicts the observed response vs. the predicted response curve using the RSM model. Results showed that the RSM was found with an acceptable R^2 and RMSE of 0.9756 and 1.3293 respectively, which demonstrated the acceptable performance of the model to fit the drying time.

2.2 Artificial neural network (ANN) modelling

Artificial neural network methods might be employed to implement a non-linear modelling and provide a substitute to logistic regression.¹⁵ The ANN model was designed with the multi-layer feed-forward network (MLP) type and trained with experimental data using back-propagation. The input parameters are initially selected based on system knowledge and availability of reliable data. Several stages are needed for the implementation of ANN in MATLAB software, details of these steps are shown in Fig. 3. The best ANN architecture was determined by optimisation of many parameters, such as the number of hidden layers and neurons, transfer function {tangent sigmoid Eq. (4), Log-sigmoid Eq. (5) and Linear}, number of iterations and network training algorithm {Levenberg–Marquardt, Bayesian regularisation}, as well as the convenient set of weights and biases. To avoid ANN divergence caused by the random initialisation of weights and biases, each ANN architecture was repeated twenty times. The number of

neurons in the hidden layer was changed from 5 to 15 neurons. Moreover, three transfer functions were tested, and the best performance was obtained with {sigmoid, linear} transfer function for the hidden and the output layer, respectively. In addition, the ANN with more than one hidden layer was tested, and results showed no significant performance.

Two different types of transfer functions were employed in this work for the hidden layer, hyperbolic tangent sigmoid (named in MATLAB as *tansig*), log-sigmoid (named in MATLAB as *logsig*). These functions are defined in Eqs. 4 and 5, respectively.^{16,17}

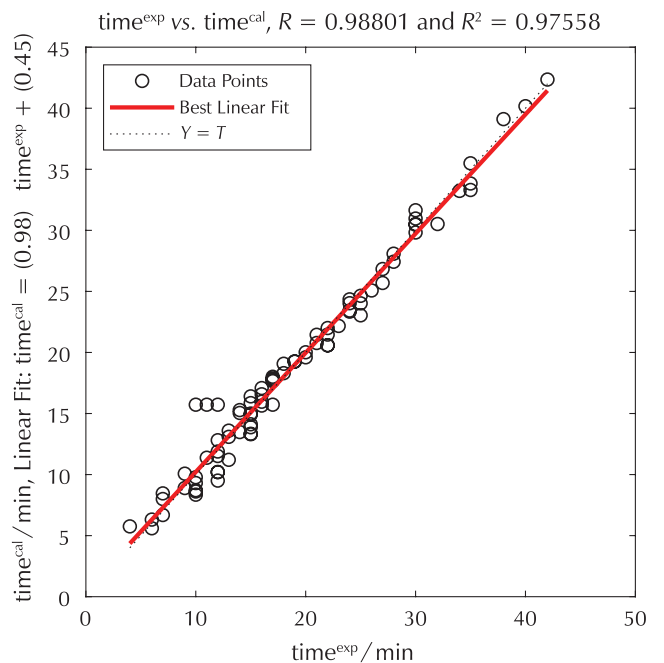


Fig. 2 – Correlation between predicted values by RSM model and experience

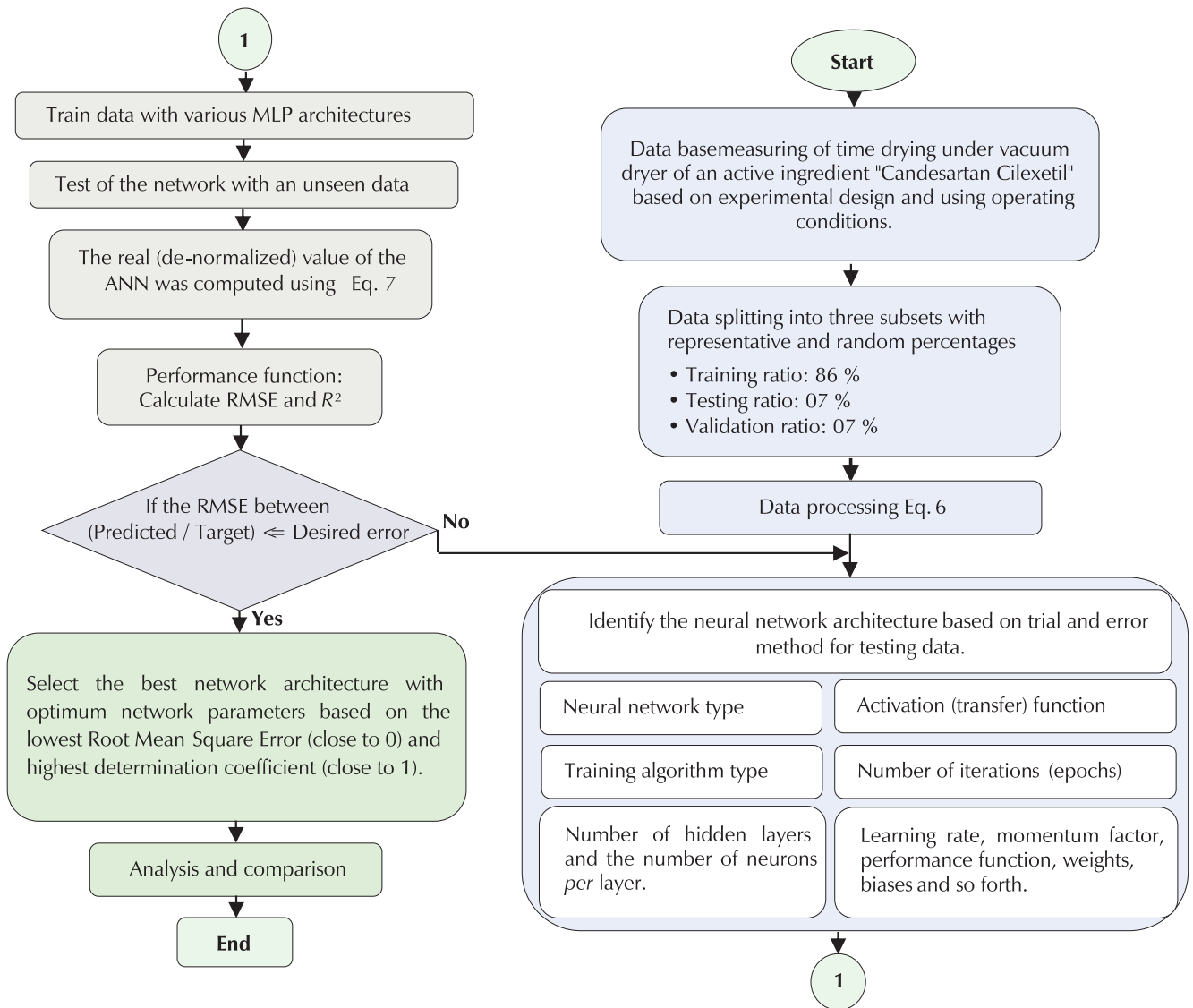


Fig. 3 – Flow chart of ANN development¹⁶

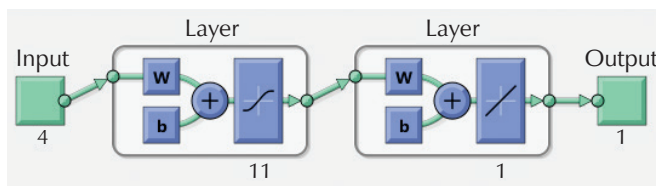


Fig. 4 – Best architecture of FFNN-BP

$$Y_j = \text{tansig}(X) = \frac{\exp(X_j) - \exp(-X_j)}{\exp(X_j) + \exp(-X_j)} \quad (4)$$

$$Y_j = \text{logsig}(X) = \frac{1}{1 + \exp(-X_j)} \quad (5)$$

where $X_j = \sum_{i=1}^m W_{ij} Y_i + b_j$, m is the number of dependent neurons, W_{ij} is the weight of connections linking layers i and j , Y_i is the output on the neurons in layer i , b_j is the bias of the neurons in layer j .

This data set will be normalised between $[-\beta\gamma, (1-\beta)\gamma]$ which leads to the stable convergence of network weights and biases by having all inputs with the same range of values and using *premnmx/postmnmx* function already programmed in MATLAB software expressed by the following equation:¹⁸⁻²¹

$$x_{in} = \frac{x_i - \min(x_i)}{\max(x_i) - \min(x_i)} \quad (6)$$

In this work, $\beta = 0.5$ and $\gamma = 2$ were selected and the scaled values of each input were computed. Output descaling can be performed using the following expression:

$$x_i = \left(\frac{1}{Y}\right) \cdot (x_{in} +) \cdot (\max(x_i) - \min(x_i)) + \min(x_i) \quad (7)$$

Table 1 shows the performance of the ANN model versus the use of two different transfer functions in its hidden layer. The best ANN model was found with the architecture of {4-11-1} (see Fig. 4) neurons in the input, hidden, and output layer, respectively. In this work, *tansig* was found to be the best function in the hidden layer.

Table 1 – RMSE of the tested ANN models

Number of hidden neurons	Levenberg-Marquardt		Bayesian regularisation	
	<i>tansig</i>	<i>logsig</i>	<i>tansig</i>	<i>logsig</i>
5	0.8325	0.7858	0.8471	0.8205
6	0.7999	1.6345	1.6588	0.9919
7	0.9393	0.7725	0.8269	0.7318
8	0.7769	1.7850	0.8138	0.7895
9	1.0698	0.7626	0.8263	0.6789
10	0.8020	0.6886	1.0866	0.8114
11	0.3397	0.6394	1.8936	0.7141
12	0.6316	1.9061	0.7740	0.6555
13	0.7313	0.8397	1.2866	0.8072
14	1.5541	0.5817	0.7475	0.6784
15	0.8810	0.8846	0.6400	0.5757

The performance of ANN models is depicted in Fig. 5 as a scatter plot of the experimental against the estimated time for the global data set using MATLAB function “postreg”. This plot shows the dispersion of the cloud of points of the entire data set around the first bisector. The degree of dispersion can be evaluated using equation ($t^{cal} = a \cdot t^{exp} + b$), where *a* is the slope, and *b* is the intercept. Those constants could be validated using a determination coefficient. The ideal performance is achieved when {*a* = 1, *b* = 0, and $R^2 = 1$ }. Values of *a*, *b*, and R^2 are 1, 0.016, and 0.9984, respectively. These results can be supported by a very low value of RMSE = 0.33970 min, which explains the performance of the obtained ANN when estimating drying time. The expression of the developed model is given by Eq. (8):

$$\text{drying time} = \sum_{s=1}^S W_{o(l,s)} \left[\frac{2}{1 + \exp(-2(\sum_{k=1}^K (W_{i(s,k)} \cdot I_{n(k)} + b_{1(s)}))} - 1 \right] + b_{2(l)} \quad (8)$$

where *k*, *S*, *l*, are the number of neurons in the input, output, and hidden layer, respectively. $W_{i(s,k)}$, $W_{o(l,s)}$ are the weights, and $b_{1(s)}$, $b_{2(l)}$ are the biases.

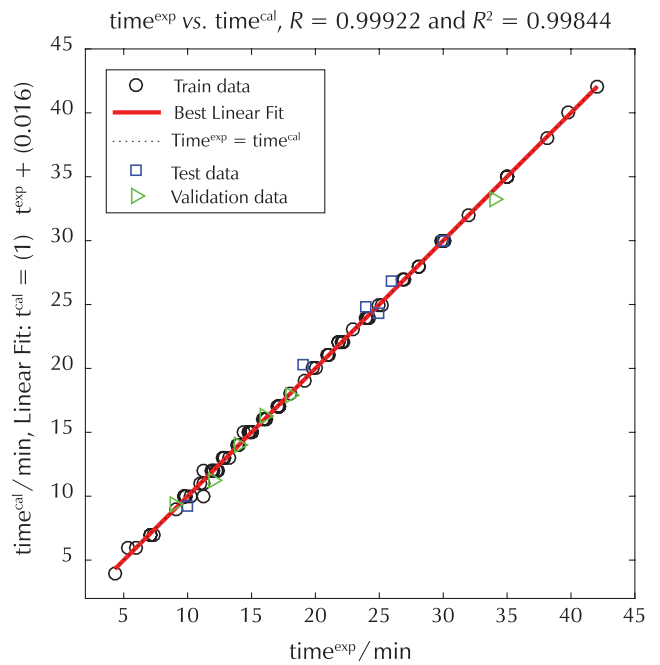


Fig. 5 – Scatter plot of the drying time estimated by ANN vs experimental drying time

2.3 Support vector regression (SVR) modelling

Compared with traditional regression and neural networks methods, recently, SVRs have been considered as a powerful technique in solving the nonlinear regression problem,^{22,23} the details of the theory and evolution of SVM developed by Vapnik’s can be found in ref.²⁴ The advantages of SVRs are that they do not require a step similar to the selection of ANN topology, do not suffer from a high risk of local minima or overfitting,²⁵ are not sensitive to starting points, and require less data in comparison to the ANN.²⁶

The determination of the model depends on the optimization of several parameters, including capacity parameter *C*, ϵ -insensitive loss function ϵ , the kernel function type and its corresponding parameters.²⁷ The flowchart of modelling using SVM technique is presented in Fig. 6.^{28,29} Many kernel functions have been tested and the Gaussian function shows its high capability of representing the complex and non-linear relation between the required drying time and its four operating conditions (Table 2). The data set was scaled (X_{in}) based on the proposed expression in this work, which is given by equation Eq. (9):

$$X_{in} = 1/0.009 \sqrt{X_i} \quad (9)$$

A scatter-plot between the observed against experimental data based on the SVM results is given in Fig. 7. Results show a satisfactory performance with high determination coefficient of 0.9991, and very low RMSE of 0.2616 min in comparison to the above models.

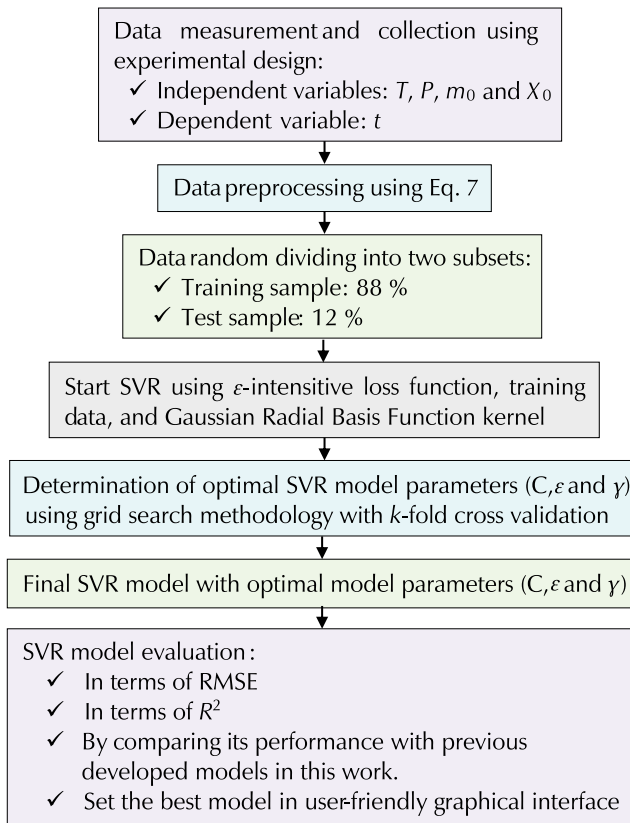


Fig. 6 – SVR modelling steps of vacuum drying time

Table 2 – Performance of SVR using different kernel functions

Kernel function	RBF	Gaussian	Polynomial	Linear
RMSE	0.3046	0.2616	0.8635	1.4852
cross-validation	$7.27 \cdot 10^{-4}$	$6.45 \cdot 10^{-4}$	0.004	0.012
quantity of support vectors	74	74	74	74
R^2	0.9988	0.9991	0.9899	0.9687

The optimisation of SVR parameters, namely, C , γ , and ϵ , was performed by varying them in the range of $[10^{-3}, 10^3]$, $[10^{-3}, 10^3]$, and $[10^{-9}, 10^{-1}]$, respectively.³⁰ The obtained best SVR parameters are given in Table 3.

Table 3 – SVR model properties

C	γ	ϵ	Kernel type	Loss function	Quantity of support vectors	Number of training data	Cross-validation error
3.0	1.45	0.0046	Gaussian	ϵ -insensitive	74	74	$6.4458 \cdot 10^{-4}$

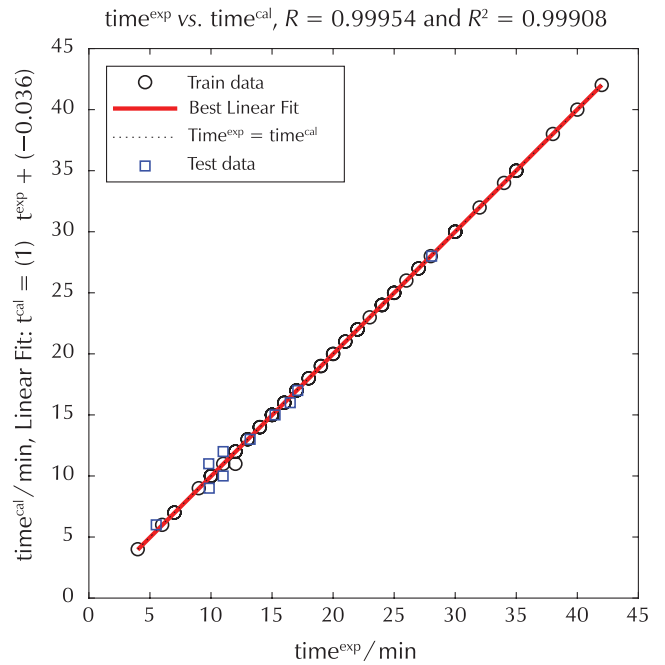


Fig. 7 – Regression plot between experimental and predicted time by SVR model

3 SVR-based model versus ANN and RSM methodology

A comparison was performed using a bar plot of R^2 and RMSE of RSM, ANN, and SVR when estimating vacuum drying time. Figs. 8 and 9 clearly show the high accuracy of support vector regression model. Results confirm that the SVR model highly outsmarts the ANN and RSM with low RMSE of 0.2616 min, and high determination coefficient close to one.

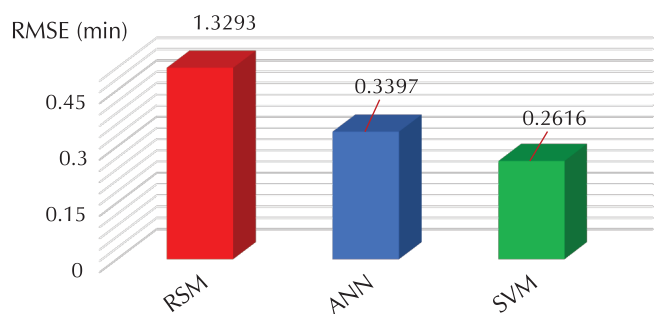


Fig. 8 – Comparison between RSM, ANN, and SVM in terms of RMSE

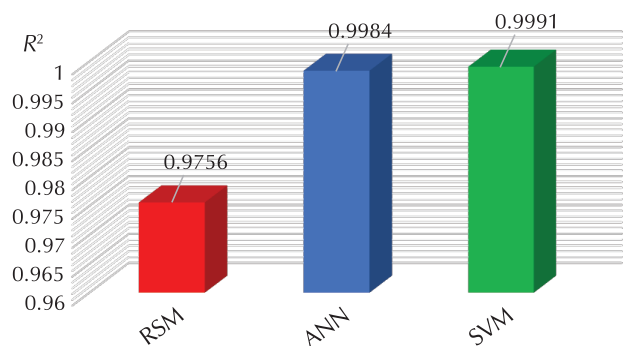


Fig. 9 – Comparison between RSM, ANN, and SVM in terms of determination coefficient

Fig. 10 shows that the data predicted by SVR model follow accurately the tendency of the experimental data, which again confirms the superiority of the SVR approach against ANN and RSM methodology.

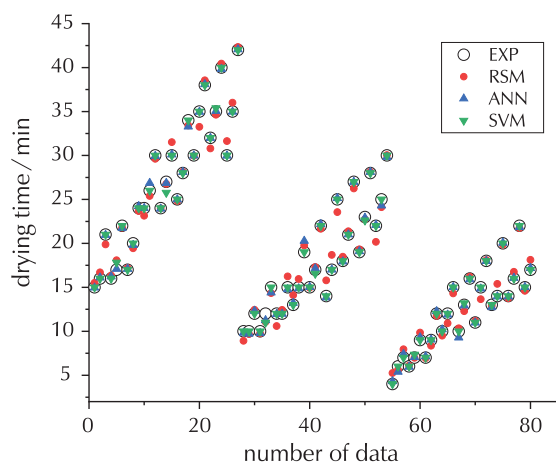


Fig. 10 – Comparison between estimated and experimental data for the generalisation phase

4 Graphical user interface for drying time calculation

An instantaneous drying time estimation of *Candesartan Cilexetil*, as an active ingredient, based on the obtained SVR model can be done through a graphical user interface within the domain of training data and solely based on four operating conditions. The created offline graphical user interface (GUI) is a user-friendly environment for those without formal knowledge of support vector regression method or any other special skills. The screen image of the user interface is shown in Fig. 11.

Temperature (°C)	<input type="text" value="40"/>	GUI for vacuum drying process time estimation of <i>Candesartan Cilexetil</i>
Pressure (bar)	<input type="text" value="0.4"/>	
Initial water content (%)	<input type="text" value="10"/>	
Initial mass (g)	<input type="text" value="1"/>	
<input type="button" value="Run"/>		Time (min) <input type="text" value="15.002"/>

Fig. 11 – Screen image of the graphical user interface

5 Conclusion

A comparative study between RSM, ANN, and SVR approaches was conducted to model the final drying time of an active pharmaceutical ingredient. Experiments were conducted under vacuum drier based on an experimental design implemented in Modde software. Compared with the RSM model {RMSE = 1.3293 and $R^2 = 0.9756$ } and ANN model {RMSE = 0.3397 and $R^2 = 0.9984$ }, the performance of the SVM model presently developed was improved for testing phase by 25 % in terms of RMSE, and with very acceptable determination coefficient of 0.9991. The SVM model proved to be much more powerful and accurate in estimating the time drying within the range of trained data compared to ANN and RSM models. A friendly user interface was designed for the SVM model.

NOTES

Authors declare that they are in no conflict of interest.

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SAŽETAK

Modeliranje vremena sušenja praha *Candesartan Cilexetil* primjenom tehnike računalne inteligencije

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Cilj ovog rada bio je primjena dvije tehnike računalne inteligencije (umjetne neuronske mreže (ANN) i regresije potpornih vektora (SVR)) za modeliranje vremena sušenja farmaceutskog praha *Candesartan Cilexetil*, koji se primjenjuje za liječenje arterijske hipertenzije i zatajenje srca. Eksperimentalni skup podataka korišten u ovom radu prikupljen je iz prethodno objavljenog rada o kinetici sušenja *Candesartan Cilexetila* pomoću vakuumske sušionike i pod različitim radnim uvjetima. Usporedba između dva modela provedena je pomoću različitih statističkih parametara, odnosno korijenom srednje kvadratne pogreške (RMSE) i koeficijenta određivanja (R^2). Rezultati su pokazali da u usporedbi s modelom ANN model SVR pokazuje visoku točnost za predviđanje nelinearnog ponašanja vremena sušenja koristeći odgovarajuće varijable ($R^2 = 0,9991$, RMSE = 0,262} u odnosu na { $R^2 = 0,998$, RMSE = 0,339} za SVR i ANN.

Ključne riječi

Candesartan Cilexetil, metodologija odgovora površine, vakuumsko sušenje, umjetna neuronska mreža, regresija potpornih vektora

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