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Modelling the process of $Al(OH)_3$ crystallization from industrial sodium aluminate solutions using artificial neural networks

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Abstract: This paper presents an attempt to define the non-linear correlation dependence between the degree of decomposition of the aluminate solution, the average diameter of the crystallized gibbsite, the total Na_2O content in the obtained alumina and the specific utilization level of the process on the one hand and important input parameters of the process on the other. As input parameters having an influence on the process, the concentration of Na_2O (caustic), the caustic ratio and the crystallization ratio, the starting and final temperature of the process, the average diameter of the crystallization seed and the duration of the decomposition process were considered. As the result of measurements of these process parameters and the acquisition of the resulting output parameters of the process, a database with 500 data lines was obtained. To define the correlation dependence, with the aim of predicting the process parameters of the decomposition process of the sodium aluminate solution, the artificial neural network (ANN) methodology was applied.

Keywords: aluminate solution; crystallization; modelling; artificial neural networks.

INTRODUCTION

In 1888, Karl Josef Bayer developed and patented a process which has become the cornerstone of the aluminium production industry worldwide.[1] The Bayer process involves the digestion of crushed bauxite in a concentrated sodium hydroxide (caustic) solution at temperatures of up to 270 °C.[2] The temperature depends on the mineral composition of the bauxite.[3] Under these conditions, the majority of the aluminium bearing species from the ore are dissolved leaving an insoluble residue (red mud) composed primarily of quartz, iron oxides, sodium

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aluminosilicates, calcium carbonate and titanium dioxide, which is removed by settling/filtration.[3]

The dissolved aluminium is precipitated entirely in the form of gibbsite ($Al(OH)_3$) with the characteristics of the final grains depending on the initial (seeding) material used and the conditions of the process.[4,5] This is achieved by cooling the solution to 52–55 °C and seeding with gibbsite grains, essentially reversing the initial dissolution process. The crystallization of aluminium hydroxide ($Al(OH)_3$) from caustic aluminate solution is the rate determining step within the Bayer cycle, which is used for alumina production.[6,7] In addition, due to the complexity of Bayer liquor speciation, the mechanisms of $Al(OH)_3$ crystallization are still not completely understood and are the subject of considerable research effort.[8]

The kinetics of gibbsite crystallization from the caustic sodium aluminate solution, as well as the size and the shape of the obtained particles, depend on the following process parameters: temperature, alumina/caustic ratio, amount and size distribution of the crystallization seeds, stirring speed and the presence of activation ions added to the solution.[9–12]

Most of the results published recently concerning gibbsite crystallization from the sodium aluminate solutions were obtained from laboratory investigations using the synthetic solutions,[9,10,13] in this way only simulating the conditions in the industrial Bayer process.[14] The industrial conditions of gibbsite crystallization are much more complex than those in laboratory experiments. At the same time, the process of gibbsite crystallization is much slower compared with other processes in the Bayer technology of alumina production,[6] which is another reason why this process demands further analysis under industrial conditions.

The main motive for the investigations presented in this paper was to draw conclusions about the possibilities of predicting the results of gibbsite crystallization from caustic sodium aluminate solutions under industrial conditions. The outputs of the process, the possibilities of prediction of which were analysed, are degree of decomposition of the solution, average diameter of the obtained gibbsite grains; content of Na_2O in the produced alumina and the specific utilization level of the process. As input parameters the concentration of caustic soda in the starting solution and its caustic ratio, the crystallization ratio of the solution (ratio between the content of $Al(OH)_3$ introduced into the solution in pulp form as crystallization seeds and the $Al(OH)_3$ content in the caustic sodium aluminate solution); the starting and the final temperatures of the process; the average diameter of the crystallization seeds and the duration of the process were considered. Defining the correlation dependence between the outputs and the inputs of this industrial process, with significant values of the correlation coefficient (R^2), presents a possibility for improved management of gibbsite crystallization from sodium aluminate solutions, as a part of the Bayer technology for alumina production.

THEORETICAL BACKGROUND AND THE METHODOLOGY OF THE INVESTIGATIONS

Parameters influencing the decomposition process of the caustic sodium aluminate solutions

The precipitate from a dilute aluminate solution is Bayerite, α-$Al(OH)_3$, and from a saturated solution, Gibbsite, $Al(OH)_3$.[8] The content of Na_2O (caustic) in the starting industrial solutions ranges between 150 and 160 g dm^{-3} with a caustic ratio (Na_2O/Al_2O_3 molar ratio) in the range 1.45–1.60. The starting temperature is in the range 60–70 °C, while the temperature at the end of the decomposition process ranges between 50 and 55 °C, which were reported to be the optimal temperatures of the process.[9,15] The amount of added crystallization seeds (CS) at the beginning of the process is determined by the crystallization ratio (CR), which presents the relationship: $\eta = Al_2O_{3(CS)}/Al_2O_{3(AS)}$ (where AS is the content of Al_2O_3 in the aluminate solution). Increasing CR positively influences the rate of the process, as does increasing the CS average diameter of the CS, under constant CR.[9,10,12,14]

Under industrial conditions, CR is in the range 2–2.5, with an average particle diameter of 100–120 μm. With time, the rate of the process decreases and the time required for 80 % of the $Al(OH)_3$ to precipitate is about 70 h under laboratory conditions.[9,12,16] Under industrial conditions, the degree of decomposition is even lower, ranging between 45 and 55 %, during 70–80 h. In addition, if producing coarse alumina (Sandy type), the granulation of the final product should be above 100 μm with an as low as possible content of $Na_2O_{(total)}$ ($\leq$ 0.4 %).[7]

RESULTS AND DISCUSSION

Modelling the dependence between the outputs (results) and the inputs (parameters of the process)

Industrial practice of the alumina production suggests that the input parameters of the process should be controlled on a daily basis because all have an influence on the kinetics of the decomposition of the sodium aluminate solution, the granulation of the produced $Al(OH)_3$ and the Na_2O content in the resulting alumina. Their synergetic action results in the output of the process, which determines its efficiency and effectiveness.

For the modelling of technological processes with established mathematical dependences between its results (dependent variables) and the predictors (process parameters), multiple linear regression analysis (MLRA), nonlinear regression (NLR) and artificial neural networks (ANNs) are the most employed methods.[17–23] Comparative analysis of the results of these statistical methods, indicate that the best results are usually obtained using ANNs.[20,21] For this reason, this methodology was used in the investigations presented in this paper.

Data from the factory Birač, Zvornik (Bosnia and Herzegovina), were used for modelling the process of the decomposition of aluminate solutions. The data were collected during the years 2008–2009 by measuring the input and output process parameters under stable operation of the production line. A total number of 500 data sets were collected this way, comprising the following:

a) Input parameters of the process. the Na_2O (caustic) content in the solution (g dm^{-3}) – X_1; the caustic ratio (α_k) of the solution – X_2; the crystallization

ratio – X_3; the starting temperature of the solution (°C) – X_4; the final temperature of the solution (°C) – X_5; the average diameter of the crystallization seed (μm) – X_6 and the duration of the crystallization process (h) – X_7.

b) Output parameters of the process. degree of decomposition of the solution (%) – Y_1; average diameter of the crystallized gibbsite (μm) – Y_2; total Na_2O content in the calcined alumina (%) – Y_3; and the specific level of solution utilization (t m^{-3}) – Y_4.

The values of the measured input parameters of the technological process (X_1–X_7) and the process quality indicators – outputs of the process (Y_1–Y_4), are presented in Table I in the form of the results of descriptive statistics.

TABLE I. Values of the input (X_i) and the output (Y_i) variables of the process of industrial sodium aluminate solution decomposition – descriptive statistics of 500 data sets

Parameter	Range	Minimum	Maximum	Mean		Standard deviation	Variance
				Statistic	Standard error		
X_1	12.330	144.000	156.330	150.944	0.076	1.703	2.900
X_2	0.180	1.470	1.650	1.530	0.001	0.033	0.001
X_3	3.410	1.260	4.670	2.285	0.030	0.662	0.438
X_4	11.000	58.000	69.000	64.656	0.054	1.199	1.437
X_5	22.200	36.300	58.500	50.582	0.184	4.112	16.909
X_6	37.710	87.220	124.930	106.473	0.381	8.510	72.416
X_7	76.000	49.000	125.000	77.080	0.624	13.944	194.426
Y_1	24.330	32.300	56.630	46.658	0.124	2.775	7.699
Y_2	37.630	86.520	124.150	107.703	0.382	8.552	73.135
Y_3	0.270	0.220	0.490	0.309	0.002	0.055	0.003
Y_4	0.039	0.052	0.091	0.076	0.000	0.005	0.000

It should be noted that one of the input parameters (X_2) has a small variance (Table I). However, it presents the caustic ratio of the solution that is one of the most important parameters of the Bayer process; thus, it cannot be omitted from the analysis. A small change in X_2 leads to a considerable change in the value of the output parameters, especially the degree of decomposition of the solution (Y_1).

For defining the correlation dependence in the form: outputs of the process (Y_1–Y_4) as function of the inputs of the process (X_1–X_7), a bivariate correlation analysis was performed. As the result of this analysis, the Pearson correlation (*PC*) coefficients with the corresponding statistical significance were calculated (Table I-S, Supplementary material).

To finally define the dependence of the output parameters as functions of the input parameters, using linear regression analysis (LRA) with an acceptable level of fitting (strong correlation), the value of *PC* must be above 0.5 or less than –0.5 with statistical significance ($p \leq 0.05$).[24,25]

The data presented in Table I-S reveals that this constraint is attained only in following cases: Y_1 (degree of aluminate solution decomposition) and X_5 (final

temperature of the process) with PC = –0.720 and p = 0.000 and X_7 (duration of the process) with PC = 0.661 and p = 0.000; Y_2 (average diameter of the crystallized gibbsite) and X_6 (average diameter of the crystallization seed) with PC = = 0.921 and p = 0.000; Y_3 (Na_2O – total in the produced alumina) and X_2 (caustic ratio) with PC = –0.602 and p = 0.000; Y_4 (specific utilization level) and X_5 (final temperature of the process) with PC = –0.583 and p = 0.000, and X_7 (duration of the process) with PC = 0.555 and p = 0.000. This was also the case for the following interdependence between outputs of the process: Y_1 (degree of aluminate solution decomposition) and Y_4 (specific utilization level of the process) with PC = 0.900 and p = 0.000.

Considering that only a small number of variables had an acceptable level of correlation (PC) and statistical significance ($p \leq 0.05$), it was concluded that the MLRA approach should not be considered as an adequate tool for modelling the investigated process because it would result in inadequate data fitting. In such cases, ANNs usually offer much better results.[20,21]

ANN Modelling

An artificial neural network is a network with nodes or neurons analogous to biological neurons.[26,27] ANNs have become a powerful tool for many complex applications, such as function approximation, optimization, non-linear system identification and pattern recognition. Artificial neural networks have seen an explosive growth in the last decade and are still being developed at a breathtaking pace. These methods represent a class of tools that can facilitate the exploration of large systems in ways not previously possible. Although neural networks originated outside the field of statistics, and have even been seen as an alternative to statistical methods in some circles, there are sings that this viewpoint is making way for an appreciation of the ways in which neural networks complement classical statistics.[28]

Owing to several attractive characteristics, ANNs have been widely used in chemical engineering applications, such as steady state and dynamic process modelling, process identification, yield maximization, non-linear control, and fault detection and diagnosis.[29–32] The most widely utilized ANN paradigm is a multi--layered perception (MLP) that approximates non-linear relationships existing between an input set of data (causal process variables) and the corresponding output (dependent variables) data set. A three layer MLP with a single intermediate (hidden) layer housing a sufficiently large number of neurons (also termed nodes or processing elements) can approximate (map) any non-linear computable function to an arbitrary degree of accuracy. It learns the approximation through a numerical procedure called “network training” wherein network parameters (weights) are adjusted iteratively so that the network, in response to the input patterns in an example set, accurately produces the corresponding outputs. A number of algo-

rithms,[28] each possessing certain positive characteristics, are employed to train an MLP network, *e.g.*, the most popular error back-propagation (EBP), quickprop and resilient back-propagation (RPROP).[33]

Error back-propagation has been applied to a wide variety of practical problems and it has proven very successful in its ability to make non-linear relationships. A typical back-propagation net, which was used for modelling procedure described in this paper, is presented in Fig. 1.

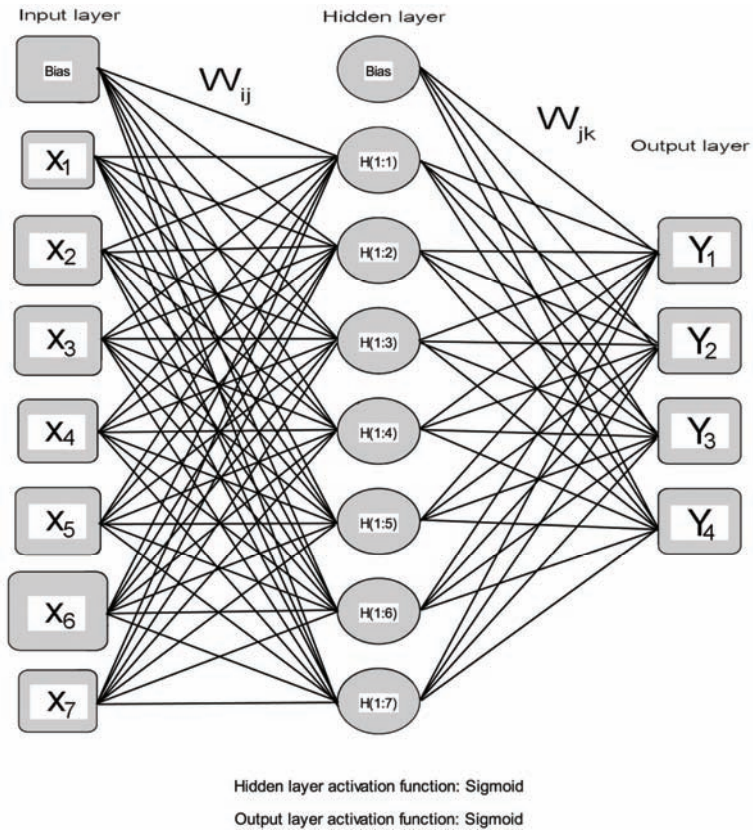

Fig. 1. The ANN architecture for the determination of Y_1, Y_2, Y_3 and Y_4 in the industrial process of decomposition of sodium aluminate solutions.

Generally, a MLP–EBP neural network contains one input layer, one or more hidden layers, and one output layer. Each layer comprises one or more neurons. The neurons are interconnected using weight factors. A neuron in a given layer receives information from all the neurons in the preceding layer (Fig. 1). It sums information, weighted by factors corresponding to the connection and the bias of the network, and transmits this sum to all neurons of the next layer using a mathematical function.[21,23]

As shown in the ANN architecture depicted in Fig. 1, the network used for modelling in this work consisted of three layers of neurons. The layers described as input, hidden and output layers comprise *i*, *j* and *k* numbers of processing nodes, respectively. Each node in the input (hidden) layer is linked to all the nodes in the hidden (output) layer using weighted connections. In addition to the *i* and *j* numbers of input and hidden nodes, the ANN architecture also houses a bias node (with a fixed output + 1) in its input and hidden layers and they provide additional adjustable parameters (weights) for model fitting. The number of the nodes *i* in the ANN network input layer is equal to the number of inputs in the process and the number of output nodes *k* equals the number of the process outputs. However, the number of hidden nodes *j* is an adjustable parameter the magnitude of which is determined by issues such as the desired approximation and generalization capabilities of the network model.[26,34]

The back-propagation algorithm modifies the network weights to minimize the mean squared error between the desired and the actual outputs of the network. Back-propagation uses supervised learning in which the inputs, as well as desired the outputs, are controlled and selected.[27]

The use of an ANN usually comprises three phases. First is the training phase, which is facilitated on 70 to 80 % randomly selected data from the starting data set. During this phase, the correction of the weighted parameters of the connections is achieved through the number of iterations to attain the minimal mean squared error between the calculated and measured outputs of the network. During the second phase, the remaining 20–30 % of the data is used for testing the "trained" network. In this phase, the network uses the weighted parameters determined during the first phase. These data lines, excluded during the teaching of the network, are now incorporated in it as new input values X_i which are then transformed to new outputs Y_k. The third phase is the validation of the network on a new data set. This data set consists of already measured data or data from new experimental measurements. The validation phase presents the final level of successful or unsuccessful prediction using the network developed in the previous two stages on a new database.[20,21]

Accordingly, ANN methodology was applied for modelling the process of sodium aluminate solution dissociation under industrial conditions using available data, the descriptive statistics of which is presented in Table I. The assembly of 500 input and output data sets was divided into two groups. The first group, which was used to train the network, consisted of 350 (70 %) randomly selected data lines, while the second group consisted of the remaining 150 (30 %) remaining from the starting database and was used to test the network.

For the development of the relational ANN configuration (Fig. 1), previously defined input parameters X_1–X_7 and output parameters Y_1–Y_4 were used as the elements of the network architecture. The ANN presented in Fig. 1 consists of

three layers: input, output and hidden layer. The neurons of the input layer are presenting the information on input process parameters, X_i (independent variables), while the neurons of the output layer generate the output information, *i.e.*, process quality indicators, Y_k (dependent variables). In the present case, $i = 7$ and $k = 4$. In addition, the best results of the model fitting were obtained with 7 neurons in hidden layer, *i.e.*, $j = 7$. The appropriate number of neurons in the hidden layer was determined by training and testing several networks. This process is necessary because too few neurons in the hidden layer produce high training and testing errors because of underfitting and statistical bias. On the contrary, too many hidden layer neurons leads to a low training error but high testing error as a result of overfitting and high variance. In this study, an iterative approach was employed to determine the optimal number of hidden layer neurons, yielding minimum model prediction error on the "test data set". In this way, 13 different network architectures were tried, ranging from 2 to 14 neurons in the hidden layer. The best results were obtained with the network architecture presented in Fig. 1.

The input to any neuron j, in the hidden layer, without its bias, is given by:

$$I_i = \sum W_{ij} X_j \tag{1}$$

where W_{ij} is the weight of the interconnection between neuron i and j and X_j represents the signal at the connection concerned.

An important component of an ANN is its activation function appearing after the input layer. Each hidden node and output node applies the activation function to its net input. For the case in question in this paper, a log sigmoid activation function was chosen. This function was most frequently used one for modelling similar systems:[20,21,34]

$$F(x) = 1/(1 - e^{-x}) \tag{2}$$

The overall transfer function of a neuron is thus structured as:

$$O_j = A_j = f(\Sigma W_{ij} X_j) \tag{3}$$

where O_j is the output of the neuron, A_j is its activation, X_j is the input to the neuron in the hidden layer, which is identical to the output of the preceding neuron with index j of the observed element.

The aim of the learning process is to minimize the overall network error:

$$E = 1/2\Sigma(y_j - O_j)^2 \tag{4}$$

where y_j is the target output value.

Adaptation of the weights is affected according to the equation:[35]

$$\Delta W_{ij} = W_{ij}(t + 1) - W_{ij}(t) = -\alpha \partial E/\partial W_{ij} \tag{5}$$

where α is defined as the learning rate. This results in:

$$\Delta W_{ij} = \alpha \beta_j X_j \tag{6}$$

where the local error of a hidden element is calculated *via*:

$$\beta_j = f(I_j)\Sigma\beta_k W_{jk} \quad (7)$$

The β_k components represent the errors of the elements in the subsequent layer, while W_{jk} represent the connection weights for these elements. The error of a neuron of the output layer is obtained *via*:

$$\beta_k = f(I_k)(y_k - O_k) \quad (8)$$

The error is first calculated and then back-propagated into the hidden layer located before the output layer.

The connection weights can then be modified according to the calculated ΔW_{ij} in the concluding stage of this process. To resolve a problem of a local minimum of the error space, a momentum term was introduced. The equation for the adoption of a weight is modified as follows:

$$\Delta W_{ij}(t) = \alpha\beta_j X_j + \mu\Delta W_{ij}(t-1) \quad (9)$$

where μ is defined as the momentum, t is the current learning step and $(t-1)$ the previous learning step.

The training used in this study is summarized in Fig. 2, for the benefit of the Y values in the investigated process.

In this way, in the training phase of the network, the necessary number of iteration was performed until the error between the measured outputs of the decomposition process of an industrial sodium aluminate solution (Y_1–Y_4) and the calculated values were not minimized and remained constant. The obtained results from the training stage can be evaluated by comparison of the calculated Y_1–Y_4 values with the measured ones. The obtained coefficients of determination, R^2: 0.729, 0.868, 0.785 and 0.732 for Y_1, Y_2, Y_3 and Y_4, respectively, present satisfactory fitting of the calculated and measured values obtained during the training phase and can be used in subsequent testing and validation.

After the development of this kind of "trained" network, the testing stage was performed using the second part of the database (a total of 150 vectors). In this phase, all 13 hidden layer structures were involved until a minimum model prediction error was obtained. The ANN structure presented in Fig. 1, with seven neurons in the hidden layer, resulted in the minimum model prediction error. During the ANN testing phase, the calculated coefficients of determination (R^2) were slightly increased in comparison with the training phase and equalled: 0.801, 0.91, 0.857 and 0.802, respectively, for Y_1, Y_2, Y_3 and Y_4. A comparative presentation of the measured and the values calculated using the ANN approach for the investigated process is presented in Figs. 1-S and 2-S, Supplementary material. The improvement in the fit obtained on the test set compared to that obtained on the training set suggests that most of the extreme points that are more difficult to model were in the training set. The selection of the variables for the

training and the testing stage was performed using a random number generator and hence, was not subjectively influenced.

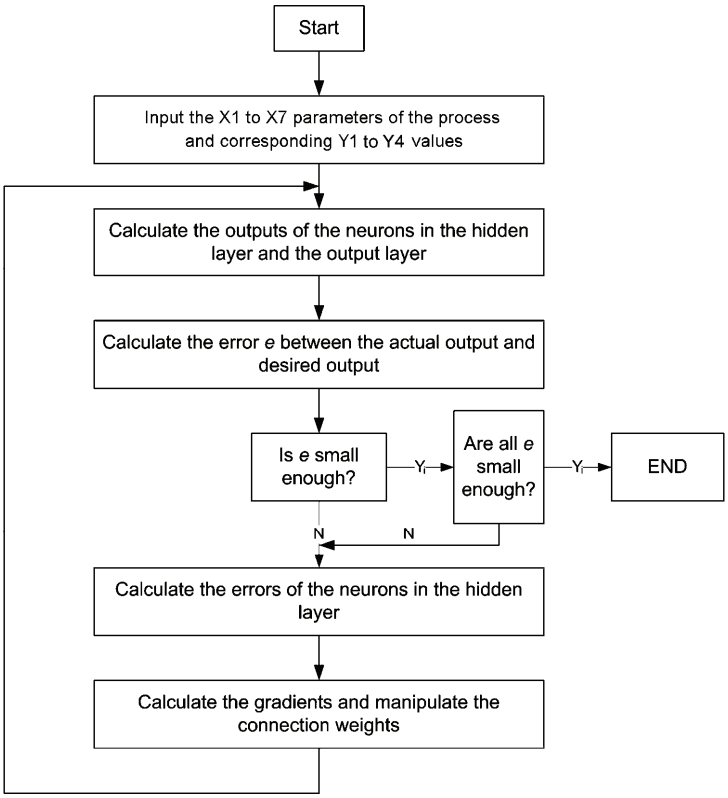

Fig. 2. Flow chart of the back-propagation learning algorithm.

According to the results presented in Figs. 1-S and 2-S, it could be concluded that the output variable Y_2 (average diameter of the crystallized gibbsite) has the best modelling potential, while other output variables, Y_1; Y_3 and Y_4, have to some extent smaller potentials for modelling. However, all the calculated coefficients of determination (R^2) were large enough to advocate that the investigated process can be adequately modelled using the approach presented in this paper.

Furthermore, the obtained results of the non-linear correlation defined using the ANN methodology enables a ranking of each individual input parameter (X_1–X_7) according to the degree of significance of their influence on the output results (Y_1–Y_4), Fig. 3.

The significances of the influence of the input parameters on the decomposition process of the industrial sodium aluminate solution are: X_6 = 0.26; X_2 = = 0.175; X_3 = 0.165; X_5 = 0.155; X_4 = 0.105; X_7 = 0095 and X_1 = 0045. The

obtained results could be quite important for managing the process of decomposition of industrial sodium aluminate solutions, concerning the minimization of Y_3, maximization of Y_1 and Y_4 and optimization of the Y_2 values.

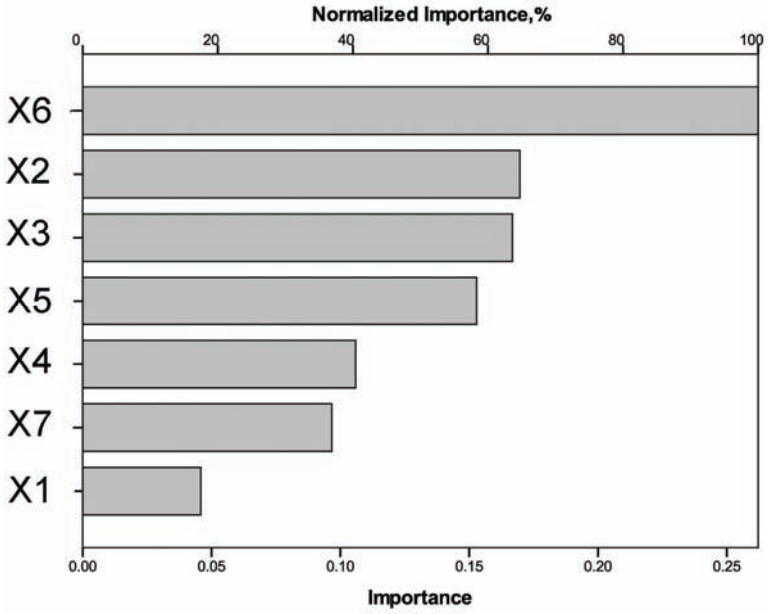

Fig. 3. Degree of significance of the individual input parameters (X_1–X_7) on the values of the output parameters (Y_1–Y_4).

CONCLUSIONS

Values of the degree of decomposition of an industrial sodium aluminate solution, the average diameter of crystallized gibbsite grains, the total Na_2O content in calcined alumina and the specific level of solution utilization (Y_1 to Y_4, respectively), under industrial conditions in the factory Birač, Zvornik (Bosnia and Herzegovina) were determined using MLRA methodology. The values of the coefficient of determination (R^2) were 0.801, 0.91, 0.857 and 0.802, respectively, for Y_1, Y_2, Y_3 and Y_4. These results indicated a highly acceptable degree of fitting of the dependence $Y_i = f(X_1–X_7)$, obtained using an ANN procedure as a part of the SPSS software application, version 18 (PASW Statistics). The selected ANN structure consisted of 350 (70 %) samples for training and 150 (30 %) for testing the network.

The significances of the influence of the input parameters of the process of the decomposition of the industrial sodium aluminate solution are: X_6 = 0.26, X_2 = 0.175, X_3 = 0.165, X_5 = 0.155, X_4 = 0.105, X_7 = 0095 and X_1 = 0045.

The variables with highest influence on the process output are the average diameter of the crystallization seeds and the caustic ratio of the solution. In addi-

tion to these parameters, the starting and final temperatures of the solution also have a large influence. The remaining inputs of the process demonstrated less importance on outcome of the process (Y_i), under industrial conditions of the Bayer technology for alumina production. The defined elements of the ANN structure can be applied generally to conditions in any factory that uses the Bayer technology for alumina production.

Since the training of the ANN structure could be facilitated on 70 to 80 % randomly selected data from the starting data set, further research will include the possibility of ANN training with different training set sizes. In this way, the influence of increasing the training set size from 70 to 75 or 80 % on the outcome of the model will be investigated.

SUPPLEMENTARY MATERIAL

Table I-S and Figs. 1-S and 2-S are available electronically at http://www.shd.org.rs/ /JSCS/, or from the corresponding author on request.

ИЗВОД

МОДЕЛОВАЊЕ ПРОЦЕСА КРИСТАЛИЗАЦИЈЕ $Al(OH)_3$ ИЗ ИНДУСТРИЈСКОГ НАТРИЈУМ-АЛУМИНАТНОГ РАСТВОРА ПОМОЋУ ВЕШТАЧКИХ НЕУРОНСКИХ МРЕЖА

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У овом раду је приказан покушај дефинисања нелинеарне корелационе зависности између степена разлагања алуминатног раствора, просечне величине пречника кристалисаног зрна гибсита, укупног садржаја Na_2O у добијеној глиници и специфичног степена искоришћења процеса на једној страни, и важних улазних параметара процеса, на другој. Као улазни параметри који утичу на процес у обзир су узети: концентрација каустичног Na_2O, каустични однос, кристализациони број, полазна и крајња температуре процеса разлагања, просечна величина пречника кристализационих центара и трајање процеса кристализације. Као резултат мерења и контроле наведених технолошких параметара, као и бележења резултујућих излазних величина, формирана је база података са 500 линија уноса. Како би се дефинисале корелације са циљем предвиђања технолошких параметара процеса разлагања алуминатних раствора, коришћена је метода вештачких неуронских мрежа.

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