RESEARCH NOTE

MODELING OF COMPRESSIVE STRENGTH OF METAKAOLIN BASED GEOPOLYMERS BY THE USE OF ARTIFICIAL NEURAL NETWORK

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Abstract In order to study the effect of R_2O/Al_2O_3 (where R=Na or K), SiO_2/Al_2O_3, Na_2O/K_2O and H_2O/R_2O molar ratios on the compressive strength (CS) of Metakaolin base geopolymers, more than forty data were gathered from literature. To increase the number of data, some experiments were also designed. The resulted data were utilized to train and test the three layer artificial neural network (ANN). Bayesian regularization method and Early Stopping methods with back propagation algorithm were applied as training algorithm. Good validation for CS was resulted due to the inhibition of overfitting problems with the applied training algorithm. The results showed that optimized condition of SiO_2/Al_2O_3, R_2O/Al_2O_3, Na_2O/K_2O and H_2O/R_2O ratios to achieve high CS should be 3.6-3.8, 1.0-1.2, 0.6-1 and 10-11, respectively. These results are in agreement with probable mechanism of geopolymerization.

Keywords Artificial Neural Network, Overfitting, Geopolymer, Compressive Strength, Metakaolin

چکیده با هدف مطالعه اثـر نسـبتهای مـولی R_2O/Al_2O_3 (کـه R = Na یـا K)، SiO_2/Al_2O_3 ، Na_2O/K_2O و H_2O/R_2O بر روی استحکام فشاری ژئوپلیمرهای پایه متاکائولن بیش از ۴۰ داده جمع آوری شد. همچنین برای افزایش داده‌ها تعدادی آزمایشات جدید طراحی گردید. داده‌های بدست آمده برای آموزش و آزمایش یک شبکه ۳ لایه‌ای استفاده شد. روش انتظام بیزی و توقف زودتر با تکنیک پس انتشار بـه عنـوان روش آمـوزش اسـتفاده گردید. توافق خوبی بین استحکام فشاری پیش بینی شده و تجربی بخاطر پـیش‌گیـری از بـیش بـرازش توسـط روش آموزش بکار برده شده حاصـل شـد. نتـایج نشـان داد کـه شـرایط بهینـه نسـبتهای مـولی SiO_2/Al_2O_3، R_2O/Al_2O_3، Na_2O/K_2O و H_2O/R_2O برای دستیابی به استحکام فشاری بهینه برابـر بـا ۳/۸–۳/۶، ۱/۲–۱/۰، ۱–۰/۶ و ۱۱–۱۰ به ترتیب می‌باشند. این نتایج با سازوکار محتمل ژئوپلیمریزاسیون همخوانی دارد.

1. INTRODUCTION

The reaction of reactive aluminasilicates such as metakaolin and fly ash with highly concentrated alkali hydroxide or silicate solution produces a kind of amorphous aluminasilicate which was first discovered by Chelokovski in 1950 and then called geopolymer after Davidovites [1]. This material exhibits good properties such as high compressive strength, low shrinkage, designable setting time, acid resistance, fire resistance and low thermal conductivity which are comparable with traditional cement and therefore makes it a good candidate to replace traditional cement in order to reduce greenhouse emissions. Another attractive application of geopolymers is encapsulation of hazardous and toxic heavy metals (such as Cu, Cr and W ion) and nuclear waste [1,2].

Geopolymer properties should be optimized in accordance with the application and the properties which are expected of them [3]. One of the fields in which geopolymer materials can have wide applications is the use of them as replacements for cement. For the purpose of fulfilling as a suitable replacement, geopolymers should possess high mechanical strength [4]. The work here aims to address the effect of compositional parameters on the mechanical properties of geopolymers. Some of these parameters are as follows; R_2O/Al_2O_3, SiO_2/Al_2O_3, Na_2O/K_2O and H_2O/R_2O. The effects of these parameters are investigated thoroughly and extensively throughout this paper. Although there are many researches regarding the effect of these parameters [5-8], the combined effect of the above parameters on mechanical properties has not been thoroughly investigated, yet.

Recently some researchers have used ANN for the prediction of mechanical properties of construction materials such as concrete and their properties [9-14]. At present, there is no literature regarding using ANN to predict geopolymers properties. However, it is expected that by implementing ANN, the number of experiments to investigate the effect of chief parameters on mechanical properties can be reduced. So, the input effective parameters can be changed parallel to each other and then the obtained data can be used to train and test the ANN.

Currently many researchers implement ANN to predict various nonlinear relations among experiment parameters, optimization, classification, control, etc [11, 14-20]. For optimization, the first step involves designing the network and then the selection of initial weights and biases and finally using the best algorithm to change weights and biases during the learning process to find the best weights and biases in order to produce desirable outputs from the input pattern [9, 14, 15, 17]. Most researchers prefer feed forward ANN and use back-propagation method for training method [11, 21]. In this method, in every interval, output is computed from the input pattern with current weights and biases and in the second step weights and biases are altered with a backward algorithm.

Conventional algorithms used in back propagation method are gradient descent, conjugate gradient descent, quasi-Newton method, Gradient Descent with Momentum, resilient back-propagation, variable learning rate back-propagation, Levenberg-Marquardt method [15].

In all the above algorithms, the performance functions (usually mean square error or sum square error) are selected and then, by changing the weights and biases step to step and numerically, performance functions are minimized.

Generalization of the network and selection of learning algorithm are based on the input data. Every selected algorithm has some advantages and disadvantages. So, choosing a suitable algorithm usually has no special discipline and comes with experience. One of the best training algorithm, having a fast rate of convergence (but more use of memory), is Levenberg-Marquardt method in which A Hessian matrix (second derivative) is approximated instead of calculating the whole Hessian matrix. Therefore, the rate of convergence in order to come to the desirable network is increased.

$$H=J^TJ,\ g=J^Te,\ X_{k+1}=X_k-[H+\mu I]^{-1}g \quad (1)$$

Here **J** is the Jacobian matrix containing the first derivatives of the network errors with respect to the weights and biases, **X** is weight or bias, **g** represents the gradient of performance function, **e** is error (true output-network output), **I** is Identity Matrix, J^T is the transposed matrix of **J**, **H** is the Hessian matrix and the **μ** is a scalar value between 0 to 1 [22-25].

One of the problems that occur during ANN training is called overfitting. The error on the training set is driven to a very small value, but when new data is presented to the network the error is large. The network has memorized the training examples, but it has not learned to generalize to new situations. The best way to avoid overfitting is to use lots of training data [11, 15, 26]. But this is impossible in some case due to increasing time and cost of experiments. Another most important method's for improving network generalization and avoid overfitting are, Model selection, Jittering, Early stopping, Weight decay, Bayesian learning [27] and Combining networks. In this work we used combined of Bayesian learning and Early-Stopping to avoid the overfitting. Bayesian learning resembles to the Levenberg-Marquardt method and differs only in the performance function [15].

2. EXPERIMENT

The Aim of this work is to investigate the effect of SiO_2/Al_2O_3, R_2O/Al_2O_3, Na_2O/K_2O and H_2O/R_2O molar ratios on CS of the geopolymers. For this purpose, more than forty data were gathered from literatures [28, 29]. In addition, for increasing the number of data, some other experiments were designed. WBB Kaolin was employed as starting material and was calcinated to produce metakaolin. Kaolin calcination was conducted at 750 °C and for 4 hours. Table 1 shows wet chemical analysis and composition of kaolin used for synthesis of the geopolymer. The Surface area of metakaolin was 15 m^2/g.

Table 1. Composition of WBB kaolin (wt%)

SiO_2	Al_2O_3	Na_2O+K_2O	Fe_2O_3	TiO_2	L.O.I
48.8	35.4	3	0.8	<0.1	11.6

Activated solution consisted of analytical grade sodium silicate solution, micro silica and analytical grade sodium hydroxide were utilized in order to control Na_2O/Al_2O_3, SiO_2/Al_2O_3 and H_2O/Na_2O ratios in the final product. Sodium silicate was first prepared by using initial sodium silicate, micro silica and sodium hydroxide to obtain the desired ratio of Na_2O/SiO_2 and H_2O, then metakaolin powder was added to the solution and mixed for 10 minutes. Then it was poured into Φ25mm×50mm polyethylene molds and sealed to protect the samples from excessive water loss. Curing regime consisted of heating at 70 °C for 4 hours in dry atmosphere and then keeping in ambient condition for 7 days. CS measurements were performed using ASTM C39-36 standard for CS. Finally, 54 data were gathered in order to get sufficient input data to train and test the ANN and thus investigating the effect of R_2O/Al_2O_3, SiO_2/Al_2O_3, Na_2O/K_2O and H_2O/R_2O molar ratios on CS. 45 data were used for training and 9 data were employed to investigate the generalization of ANN (Table 2 and 3). The data haphazardly separated to test and train in order to prevent from probable error. Three layered feed forward ANN was employed for the training. The network was trained utilizing the back propagation algorithm. The numbers of neurons in hidden layer and in the output layer were 3 and 1, respectively. The input pattern consisted of R_2O/Al_2O_3, SiO_2/Al_2O_3, Na_2O/K_2O and H_2O/R_2O ratios and the output data consisted of CS. (Figure 1) tan-sigmoid (tansig) function (Equation 2) was selected as the hidden layers transfer function and the linear function was selected for output layer transfer function due to their ability to learn complex nonlinear relation between input patterns and output data.

After gathering the data it should be preprocessed to increase the efficiency of the ANN training. The preprocessing involved converting all input data into values in the range of [0 1].To improve the generalization and reducing the overfitting problem Bayesian regularization training method (trainbr) was chosen.

$$\text{Tansig (N)} = \frac{2}{1 + e^{-2\times N}} - 1 \qquad (5)$$

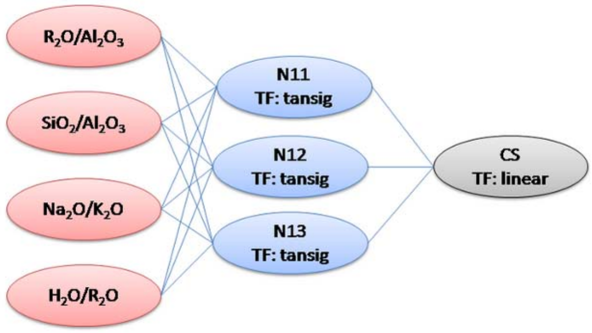

Figure 1. Neural Network topology.

3. RESULTS AND DISCUSSION

Figure 2 shows the reduction of sum of squared error (SSE) along the training interval. It can be seen that SSE at last converge to 13.79, in this point the ANN was stopped for inhibit the over fitting problems.

Figure 3a shows the generalization of the trained ANN in training and tested point and also shows the best linear fit for the data. The calculated R values by comparing data with y=x line were also presented in Figure 3b. With respect to the obtained results, it can be concluded that the predicted values of ANN for CS shows good correlation (R Value) with the real experimental values for both training data and test data. Also from Figure 3, Table 2 and 3, it can be seen that the error of data for both train and test data have Normal Distribution. Some uncertainty can be seen for the test data. This uncertainty can be related to wide distribution of comprehensive strength of ceramics.

Table 2. Train data besides predicted value of compressive strength (PCS) of geopolymer samples.

Sample N	Na/[Na +K]	$SiO_2:Al_2O_3$	$Na_2O:Al_2O_3$	$H_2O:Na_2O$	CS	PCS
10	1	4	0.8	10	2.30	2.08
15	1	4	1	10	2.74	3.29
2	1	2.5	0.6	10	6.60	6.73
11	1	2	1	10	8.23	10.17
15	0	2.3	1.00	11	8.94	9.20
6	1	2	0.8	10	12.09	10.23
30	0.75	2.3	1.00	11	12.62	14.55
25	0.5	2.3	1.00	11	13.81	12.94
35	1	2.3	1.00	11	15.79	14.29
54	1	3.8	1.14	17.5	25.03	25.02
43	1	4.4	1.2	11	26.62	26.83
53	1	3.55	1.04	20	27	27.07
40	1	4	1	15	30.02	29.99
7	1	2.5	0.8	10	30.56	31.72
45	1	3.9	0.95	12	32.57	33.38
44	1	3.05	0.7	11.42	33.43	33.42
52	1	4	1	11	33.86	34.49
12	1	2.5	1	10	34.19	33.10
41	1	4.2	1.1	11	36.57	36.38
46	1	3.5	0.75	14	37.93	36.91
50	1	3.5	1	11	38.44	41.83
36	1	2.8	1.00	11	38.93	40.54
48	1	3.63	1.04	12	39.05	37.59
42	1	3.5	1	9.3	40.55	40.73
16	0	2.8	1.00	11	46.07	45.02
21	0.25	2.8	1.00	11	47.16	48.86
31	0.75	2.8	1.00	11	48.71	46.70
26	0.5	2.8	1.00	11	49.71	49.21
8	1	3	0.81	10	52.36	52.27
37	1	3.3	1.00	11	57.91	56.54
5	1	4	0.6	10	59.11	59.04
14	1	3.5	0.98	10	60.22	60.15
29	0.5	4.3	1.00	11	60.73	60.68
17	0	3.3	1.00	11	65.90	65.96
39	1	4.3	1.00	11	66.83	66.20
34	0.75	4.3	1.00	11	71.02	70.60
9	1	3.5	0.805	10	74.09	73.98
22	0.25	3.3	1.00	11	74.41	74.14
24	0.25	4.3	1.00	11	75.66	75.49
32	0.75	3.3	1.00	11	77.68	78.69
23	0.25	3.8	1.00	11	78.78	79.05
38	1	3.8	1.00	11	81.60	79.66

Table 3. Test data besides predicted value of compressive strength (PCS) of geopolymer samples.

Sample N	Na/[Na +K]	$SiO_2:Al_2O_3$	$Na_2O:Al_2O_3$	$H_2O:Na_2O$	CS	PCS
1	1	2	0.6	10	4.30	0.10
20	0.25	2.3	1.00	11	9.11	10.97
49	1	4.5	1.57	10.3	24.95	16.37
47	1	4.01	1.16	13.1	31.98	40.23
51	1	3.275	0.85	10.58	38.83	37.43
3	1	3	0.6	10	46.28	47.25
4	1	3.5	0.595	10	57.26	48.20
19	0	4.3	1.00	11	65.31	73.00
27	0.5	3.3	1.00	11	74.33	78.55
18	0	3.8	1.00	11	83.22	77.69

Filled Contours which are shown in Figure 4, show the effects of R_2O/Al_2O_3, SiO_2/Al_2O_3, Na_2O/K_2O and H_2O/R_2O ratios on comprehensive strength of the geopolymers. Each on of the contours shows the effect of two molar ratios on CS. All of the contours were plotted in optimum value of other parameters (Table 4). By the employment of these contours, the effect of input parameters of ANN on CS can be predicted.

Table 4. Optimum value of R_2O/Al_2O_3, SiO_2/Al_2O_3, Na_2O/K_2O and H_2O/R_2O molar ratios for plotting Figure 4 contours.

R_2O/Al_2O_3	SiO_2/Al_2O_3	Na_2O/K_2O	H_2O/R_2O
1.15	3.7	1.0	10.5

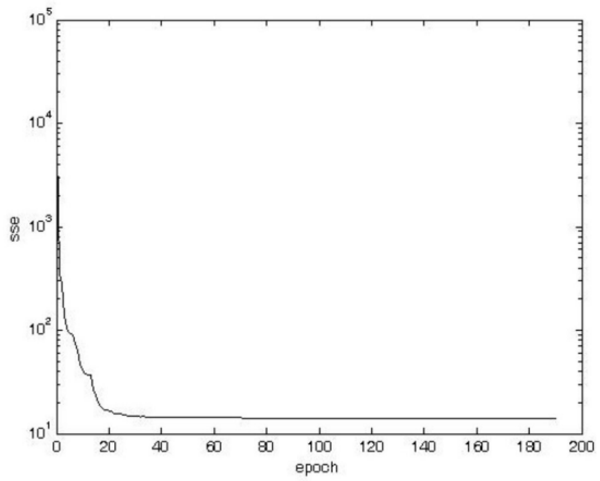

Figure 2. Reduction of SSE during the learning process.

3. 1. Effect of SiO_2/Al_2O_3 on Compressive Strength

Figures 4a and 4b show the effects of SiO_2/Al_2O_3 and H_2O/Na_2O and the effects of SiO_2/Al_2O_3 and H_2O/R_2O ratios on CS of samples, respectively. It can be seen that optimum value of SiO_2/Al_2O_3 is about 3.6-3.8 in optimum ratios of other parameter. This behavior is in consistent with the general behavior of CS of geopolymers, reported in literature [29, 30]. Most researchers believe that with increasing SiO_2/Al_2O_3 ratio, polysialatesiloxo and polysialatedisiloxo structures become dominant [29, 30]. Because of more strength and stiffness of these structures in compare with polysialate structures, which are present in low SiO_2/Al_2O_3 ratios, the CS is higher with increasing SiO_2/Al_2O_3 ratio. On the other hand solubility of metakaolin and gel formation decreases with increasing the SiO_2/Al_2O_3 ratio, therefore the CS of geopolymers tends to decrease in high SiO_2/Al_2O_3 ratios. In low SiO_2/Al_2O_3 ratios (<3.6), existence of coarse voids, compared with finely distributed voids in high SiO_2/Al_2O_3 ratios (>3.8), is another reason for low CS of the samples in that ratios. Superposition of theses parameters affects the CS of geopolymers and makes an optimum range for CS of geopolymers with respect to SiO_2/Al_2O_3 ratios.

3. 2. Effect of R_2O/Al_2O_3 on Compressive Strength

Figures 4a and 4d show the effect of R_2O/Al_2O_3 and SiO_2/Al_2O_3 molar ratios and the effects of H_2O/R_2O and R_2O/Al_2O_3 ratios on CS in optimum

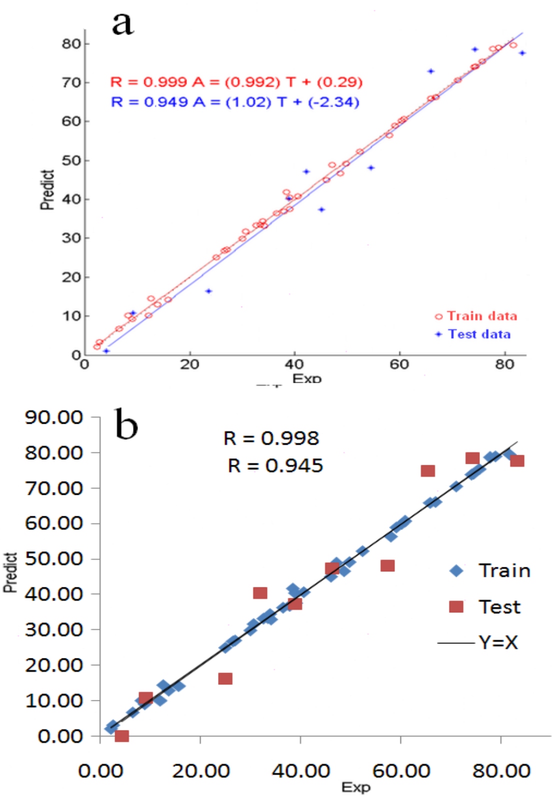

Figure 3. Correlation between the predicted value of CS (predict) with Experimental value (EXP) for test and train data a) best linear fit b) calculation of the R value by comparing data with y=x function..

amount of other parameters. It can be seen that the optimum values of R_2O/Al_2O_3 ratio are around 1-1.2. This result is in consistent with theoretical and experimental principles of geopolymer formation mechanisms. R^+ cations play the role of negative charge balancer and hence network stabilizer. This negative charge comes from four fold coordination of Al^{3+} in tetrahedral sites. Due to the fact that some R^+ cations are consumed to form R_2CO_3, in low R_2O/Al_2O_3 ratios, the amount of R^+ cations is not sufficient to balance the negative charges, so the geopolymer structure becomes distorted and unstabilized. Thus the optimum value of R_2O/Al_2O_3 ratio is 1-1.2.

3. 3. Effect of H_2O/R_2O on Compressive Strength

Figures 4b and 4d show the effect of H_2O/R_2O and R_2O/Al_2O_3 molar ratios and the effect of H_2O/R_2O

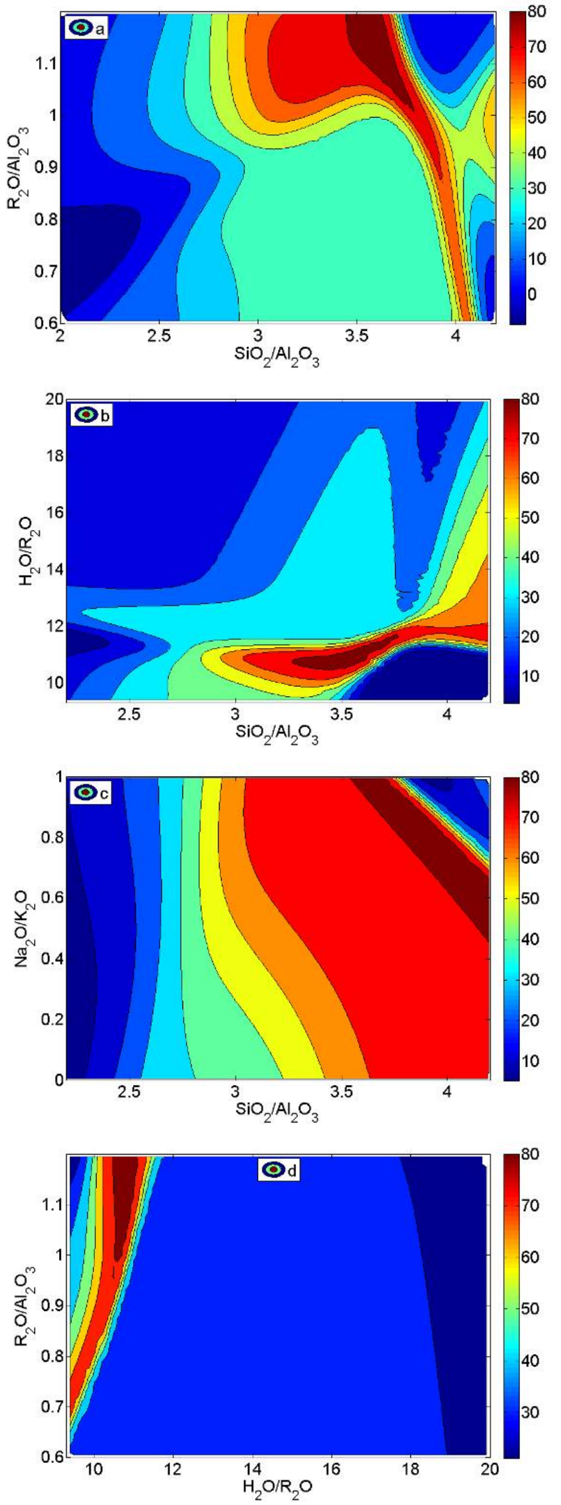

Figure 4. (a) Filled contours plot of CS that shows the effect of R_2O/Al_2O_3, SiO_2/Al_2O_3, Na_2O/K_2O and H_2O/R_2O ratios on CS. The contours are in units of MPa.

and SiO_2/Al_2O_3 molar ratios on CS. From these figures it could be obtained that optimum value of H_2O/R_2O ratio is about 10-11 to achieve high CS. In high H_2O/R_2O ratios the amount of OH^- groups are high. Thus the amount of porosity tends to increase after condensation and this phenomenon decreases the compressive strength of geopolymer. On the other hand, water provides the suitable media for geopolymerizasion reaction and in low level of H_2O/R_2O ratios the rate of geopolymerizasion reaction is low. Therefore, CS of geopolymer is decreased in low level of H_2O/R_2O ratios. Superposition of these mechanism make an optimum value of CS in H_2O/R_2O=10-11.

3. 4. Effect of K_2O/Na_2O on Compressive Strength

Figure 4c shows the effect of Na_2O/ K_2O and SiO_2/Al_2O_3 effects on the Comprehensive strength of geopolymer. It can be seen from this figure that in lower SiO_2/Al_2O_3 molar ratios, optimum value of Na_2O/ K_2O ratio is about 1, whereas in higher ratios of SiO_2/Al_2O_3 the optimum value was decreased up to 0.6. These results are in consistent with the results Duxson et al [29] work. They believed that K^+ ion is more active than Na^+ ion and potassium is preferentially incorporated into geopolymeric gels during formation. Therefore, in lower amount of SiO_2/Al_2O_3 molar ratios, sample containing potassium has lower levels of SiO_2/Al_2O_3 ordering and hence, has lower strength. This phenomenon was called Mixed Alkali Effect in geopolymer by Duxson [29].

4. CONCLUSION

For the purpose of studying the effect of the SiO_2/Al_2O_3, R_2O/Al_2O_3, Na_2O/K_2O and H_2O/R_2O molar ratios on the compressive strength of geopolymers more than forty data were gathered from literatures and for increasing the number of data some experiments were also designed. Then, the resulted data were applied for training and testing the ANN. In sum, following conclusions can be drawn from the proposed model:

1. ANN is a practical tool for modeling and optimization of the mechanical properties of geopolymers.
2. Optimized SiO_2/Al_2O_3, R_2O/ Al_2O_3, Na_2O/K_2O and H_2O/R_2O ratios were determined to be 3.6-3.8, 1.0-1.2, 0.6-1 and 10-11, respectively.
3. Bayesian regularization method in combination with early stopping of network during training can inhibit the overfitting problem better than the Bayesian regularization or early stopping alone.
4. The optimized value for the compressive strength (optimized for the defined synthesis method in this work) is approximately about 80 MPa.

5. ACKNOWLEDGMENTS

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