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Taguchi-grey Relational Analysis for Optimizing the Compressive Strength and Porosity of Metakaolin-based Geopolymer

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ABSTRACT

Geopolymer is a promising eco-friendly material that can be produced with a variety of physical and mechanical properties through alerting the processing parameters. Obtaining Geopolymer with high compressive strength and high porosity may make this material a preferred candidate for many thermal and physicochemical applications. This research aims to identify the set of the processing parameters that yield such as these Geopolymer materials. Taguchi method combined with Grey relational analysis has been used to solve this multi-response trouble. The analysis and the experimental results showed that it is possible to achieve this aim by using a low amount of hydrogen peroxide as a foaming agent, a low amount of yeast as a catalyst, and a low amount of vegetable oil as a stabilizer. Furthermore, the polymerization time elapses before adding the foaming agent is found to be an important processing parameter. Also, the experimental results showed that high porosity and adequate compressive strength can be obtained at the same geopolymer body by choosing the suitable values of the processing parameters. Moreover, it has been found that the use of yeast as the catalyst and the polymerization time is important processing parameters. Also, it has been noticed that the amount of the vegetable oil, which is used as a stabilizer, should be kept in low values to obtain the optimal compressive strength and porosity.

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1. INTRODUCTION

Joseph Davidovits is considered the first to formulate the geopolymer term in1970s [1]. Geopolymers refer to the three-dimensional aluminosilicates cementitious material that can be produced by the precipitation, polycondensation and dissolution of the aluminosilicates origin [2]. Geopolymer has a typically amorphous or semi-crystalline structure with aluminum and silicon sites tetrahedrally coordinated. The geopolymer structure consists of a polymeric Si–O–Al framework [3]. geopolymers can be consolidated at room temperature and, for selected applications, can be used at high temperatures up to 1200°C [4].

Geopolymer materials have an advantage over the Portland cement-based binders which cause the emission of very large amounts of carbon dioxide. Fly ash, waste glass and slag were used as raw materials for the geopolymer which can decrease the carbon emission from these binder materials [3-6]. Metakaolin-based geopolymers are supposed as ‘‘model-system” without the drawbacks inserted by using fly ash, slag, and other alternative starting materials which include several difficult-to-characterize amorphous phases [7].

Geopolymer is an intrinsically porous material with a small pore size and variable pore shape. The synthesis of highly porous geopolymer generally involves the addition of blowing agents to the geopolymer paste, such as hydrogen peroxide (H_2O_2), metallic powders (aluminum and zinc). The addition of these agents in the geopolymer synthesis process affects the polymerization kinetics as well as the rheology of the produced paste [8-17].

In the production of lightweight geopolymers, Hydrogen peroxide has been widely used as a chemical foaming agent. In a highly alkaline environment of

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geopolymer, hydrogen peroxide decomposition can be an unlimited reaction resulting in a porous material with undesirable coarse voids [18]. The bubbles of the gas inclosed within the material extend and generate voids. The volume expansion of this material is rely on the amount of oxygen result in the following reaction [2].

$H_2O_2 \rightarrow H_2O + ½ O_2$

The high alkalinity catalyzed the hydrogen peroxide decomposition [19], and the solution of sodium silicate is known to stabilize the decomposition reaction through decreasing and slowing down the reaction [18].

Many processing factors can affect the structure and the properties of the geopolymer porous materials, such as the content and type of foam agents/stabilizer, the design of mix proportion, the quality of raw materials. Of special importance is the stabilizer which could limit the surface free energy of the bubble and increase the toughness of the bubble. This is reducing the burst and coalesce of bubble offering a great effect on the stability of pore in geopolymer porous materials [20- 22].

The stablizers (surfactants) like Tween 80, protein, and vegetable oils are classified in the hydrophilic group and have been used in the production of porous geopolymers. The vegetable oils is one of the interesting stablizers because of their low cost and availability. The reaction between highly alkaline solution of geopolymer and vegetable oils, by a saponification reaction, result in the interconnect porous structure [23, 24].

It is well known that the pores inside the material reduces the mechanical strength of the geopolymer [25, 26]. On the other hand, in the many applications were desired to have a material that combines high porosity and high mechanical strength. Such material can be used, for example, as load bearing thermal insulator, load bearing light weight material and highly porous catalyst or adsorbent that can be used under high pressure conditions. As per our review, a study that combines the optimization of both porosity and mechanical strength is not reported in the literatures.

The main target of this research is to improve both porosity and the compressive strength of metakaolin-based geopolymer. Taguchi method was adopted to design the experiments depend on the orthogonal analysis method. Based on the Taguchi-Grey relational multi-responses analysis method, the optimal mix proportion of the geopolymer was obtained.

The constraction industry through using of less polluting technology to the environment is already imminent and more had to do. Usually it can only consider single quality characteristics. While, Grey Relational Analysis (GRA) is usually employed to deal with the multi-quality characteristics [26]. This is because, GRA is a normalization evaluation technique to solve a complicated multi-performance characteristics optimization effectively [27]. It can be used with Taguchi method to solve the multi response problems. According to our review, Grey relational method is not well reported in the litearetuer to be used in the multi-responses analysis in the field of geopolymers except in the work of Prusty and Pradhan [28]. They were used Taguchi-Grey relational analysis to investigate and optimize the effect of ground granulated blast furnace slag replacement, water to geopolymer solids ratio, molarity of NaOH solution, binder content and Na_2SiO_3 to NaOH solution ratio on setting time, workability and compressive strength of fly ash-based geopolymer concrete [28]. However, it is well documented that it has been utilized to optimize many other products and processes, these includes literature [29-33].

In the current work, three common factors were selected to design the experiments including, the concentration of hydrogen peroxide, the quantity of hydrogen peroxide and the quantity of vegetable oil as stabilizer. These factors were commonly studied in the field of geopolymers and their influnce on the properties of the geopolymer are well documented in many studies [21, 24]. Furthermore, for the first time, two additional factors were studied in the current study; these are (i) the time of polymerization elapse before adding the hydrogen peroxide to the produced geopolymer paste prior to casting, (ii) the amount of yeast added to the mix which has been used to catalyze the decomposition of the hydrogen peroxide to produce pores. The first factor i.e the time of polymerization, is expected to affect the pore size, pore shape and the distribution of the pores along with geopolymer body; this is due to its effect on the viscosity of the geopolymer paste. The later factor, i.e. the amount of yeast, is chosen to have a controllable factor that affect the decomposition of hydrogen peroxide rather than the uncontrollable factors of the alkalinity and the amount of sodium silicates, which have fixed values based on the preselected composition of the geopolymer, that have been optimized in our previous work [34].

Five levels, having strongest impact on the performance of the specimens, for each of the five factors were selected in the design of the experiments based on primary rough experiments. The lower and the upper values of a given level were chosen through rejecting the values that produce a geopolymer body with: (i) very low mechanical strength, due to high porosity or large pore size or (ii) very low porosity due to low expansion upon adding the foaming agent. This information was obtained from this test is very useful in understanding how the strength of materials involved in natural application [35].

2. EXPERIMENTAL WORK

2. 1. Design of Experiments

An efficient approach for the optimizing a single quality response issues is Taguchi method [36]. However, through these days, many operations were performed, manufactured products have more than one quality response of main interest [37]. Thus; traditional Taguchi method does not able to solve such multi-objective optimization problems. Taguchi method shared with Grey relational analysis to solve the more complicated multi response problems [38].

In this technique, orthogonal experimental design, adopting Taguchi method, it was used to design the experiments. GRA was used to combine the multi responses into a single effective response. Then, Taguchi method was used to analyze that the single effective response and suggest the values of the experimental parameters that are expected to achieve optimal responses.

Relay on the orthogonality, orthogonal experimental design chooses several representative points from the comprehensive experiments; these points have constant waste and similarity. The level in the orthogonal analysis refers to the specific conditions for each factor to be compared. The factors are parameters that affect the properties of product.

In the current study, the five factors and their corresponding levels are shown in Table 1. According to Taguchi method, the L_{25} ($5^{\wedge 5}$) orthogonal test scheme should be used in the experiments with the details given in Table 2.

TABLE 1. Five factors and five levels of orthogonal test design

Level \ Factor	A	B	C	D	E
1	10	0.1	0.2	0	0.1
2	20	0.2	0.4	30	0.2
3	30	0.3	0.6	60	0.3
4	40	0.4	0.8	90	0.4
5	50	0.5	1.0	120	0.5

where A: Concentration of H_2O_2%, B: Quantity of the yeast (g), C: Quantity of H_2O_2 (ml), D: Polymerization time (min) and E: Quantity of vegetable oil (ml).

TABLE 2. Orthogonal test scheme L25 ($5^{\wedge 5}$)

Experiment No.	A	B	C	D	E
1	10	0.1	0.2	0	0.1
2	10	0.2	0.4	30	0.2
3	10	0.3	0.6	60	0.3
4	10	0.4	0.8	90	0.4
5	10	0.5	1.0	120	0.5
6	20	0.1	0.4	60	0.4
7	20	0.2	0.6	90	0.5
8	20	0.3	0.8	120	0.1
9	20	0.4	1.0	0	0.2
10	20	0.5	0.2	30	0.3
11	30	0.1	0.6	120	0.2
12	30	0.2	0.8	0	0.3
13	30	0.3	1.0	30	0.4
14	30	0.4	0.2	60	0.5
15	30	0.5	0.4	90	0.1
16	40	0.1	0.8	30	0.5
17	40	0.2	1.0	60	0.1
18	40	0.3	0.2	90	0.2
19	40	0.4	0.4	120	0.3
20	40	0.5	0.6	0	0.4
21	50	0.1	1.0	90	0.3
22	50	0.2	0.2	120	0.4
23	50	0.3	0.4	0	0.5
24	50	0.4	0.6	30	0.1
25	50	0.5	0.8	60	0.2

2. 2. Materials and Methods Metakaolin can be obtained by the calcination of kaolin clay at 750°C for 3 hours in the air atmosphere via using heating rate of 5ºC/ min. The Kaolin was supplied from Dwaikhla, a local area in the western desert of Iraq. Sodium silicate ($Na_2SiO_3.5H_2O$, Thomas Barker), sodium hydroxide (NaOH, Thomas Barker), silica gel ($SiO_2.nH_2O$, Thomas Barker), hydrogen peroxide (50%-H_2O_2, Thomas Barker) were used as received without further treatment or purification. Instant yeast and sun flower vegetable oil were supplied from local market, they were used as a catalyst and stabilizing agent, respectively to synthesize the porous geopolymer.

Na_2O. Al_2O_3. $3.8SiO_2$. xH_2O formula describes the composition of the geopolymer synthesized in the current study. This formula and the processing parameters, including the amount of water of 11ml per 10.73g of metakaolin, the mixing time of 5 min and the sodium silicates to sodium hydroxide ratio of 3.02, were obtained from our previous study on the optimization of the composition and the processing parameters of geopolymer, as illustrated elsewhere with more details [34].

In this study the alkaline liquid is a solution of sodium hydroxide, sodium silicate and silica gel. Firstly, water is introduced in the beaker , then sodium hydroxide have been added to the required amount of water. Then, the silicate salt was added, while the solution was heated to 80°C and agitated at 600 rpm. After all silicates salt is disolved, silica gel was added to the solution , then stirred for one hour. Later, a desired quantity of water was added to compensate the water lost because of the vapoization, the solution was left to be cooled naturally to room temperature. The metakaolin was added to the cold solution, and mixed using a mechanical mixer at a fixed agitation rate (3550 rpm) for sufficient mixing time. After that, the alkaline solution is cooled to room temperature. Finally, the hydrogen peroxide, yeast and vegetable oil were added to the solution, after the desired polymerization time to form the geopolymer paste.

For molding the pastes of geopolymer, the molds made of PVC plastic were used. The molds were kept in the lab conditions at a temperature of 23°C± 2 for one day and then de-molded. These specimens have been cured at the room temperature for 28 days before testing. Figure 1 display the fractured surface of the samples obtained according to the pre-mentioned preparation method.

The compressive strength of the sample, having a height to diameter ratio of 2 was obtained via the compressive test, by using universal test machine. The water absorption, porosity and density of the produced samples were measured via using Archimedes method. Each measurement in this study is the average of three measurements.

2. 3. Optimization The experimentally obtained compressive strength and porosity values were transformed into a signal-to-noise (S/N) ratio. The value of S/N ratio mention the dissipation around the required results and includes, three types of performance characteristics: higher-the-better, lower-the-better and nominal-the-better. In this study, the S/N ratio of the higher-the-better was used, as it is desired to obtain a higher compressive strength and higher porosity, and calculated using Equation (1):

$$(S/N)_{ij} = -10\log\left(\frac{1}{n}\sum_{i=1}^{n}\frac{1}{y_{ij}{}^2}\right) \quad (1)$$

where; n is the number replications and yij is the experimental value of ith experiment for the jth response.

In order to spread out the data evenly, and measure it into an acceptable range for further analysis the (S/N) ratio were normalized [39] using Equation (2), to obtain

Figure 1. Porous geopolymer samples with different porosity

Zij which represents the normalized value of S/N ratio for the larger is better.

$$Z_{ij} = \frac{y_{ij} - \min(y_{ij})}{\max(y_{ij}) - \min(y_{ij})}; i = 1,2,\ldots n; j = 1,2,\ldots n \quad (2)$$

According to GRA, Equation (3) was used to calculate of the deviation sequences (Δ):

$$\Delta = (Z_{max} - Z_{ij}); i = 1,2,\ldots, m; j = 1,2,\ldots, n \quad (3)$$

where; Zmax is the maximum value of response Z_{ij}:the current value of the response

Equation (4) was used to calculate the grey relational coefficient (GRC) and Equation (5) was used to calculate the grey relational grade (GRG).

$$GRC_{ij} = \frac{\min(\Delta) + \lambda\max(\Delta)}{\Delta_{ij} + \lambda\max(\Delta)}; i = 1,2,,m; j = 1,2,,n \quad (4)$$

$$GRG_i = \sum_{j=1}^{n}\varphi_j GRC_{ij} \;\; ; i = 1,2,\ldots,m \quad (5)$$

where: φj is the normalized non-negative coefficient assigned to the jth response with the sum of all φj is equal to 1, and Δij is the difference between the optimum value of the normalized S/N ratio, λ is the identification coefficient that ranges from 0 to 1 , the ith normalized S/N ratio value for the jth response. While, in the equation numerator, max(Δ) is means the largest optimum value of the normalized and max(Δ) is mean the smallest optimum value of the normalized. In this study all the responses (characteristics) are equally weighted.

3. RESULTS AND DISCUSSION

3. 1. Grey-based Taguchi Optimization Results For compressive strength and porosity, the average value of the experimentally obtained results are given in Table 3.

TABLE 3. Experimental results for the compressive strength and the porosity

Experiment No.	Compressive strength (MPa)	Porosity (%)
1	66.95	30.67
2	50.34	31.91
3	33.60	30.83
4	25.56	21.57
5	21.39	9.52
6	30.20	26.29
7	19.94	16.84
8	6.12	24.35
9	9.99	34.51
10	6.32	19.58
11	7.37	28.26
12	3.09	59.43

13	3.51	65.76
14	24.41	35.00
15	12.40	21.64
16	2.76	74.66
17	6.28	65.25
18	2.65	84.10
19	5.95	31.06
20	4.39	41.09
21	8.85	80.12
22	10.66	60.98
23	4.57	42.02
24	7.69	65.00
25	2.76	76.18

This raw data can be transformed into S/N ratio by using Equation (1). According to L_{25} orthogonal array the corresponding S/N ratio values for experimental parametric setting are summarized in Table 4.

The normalized values of the S/N ratio, calculated according to equation 2, are given in Table 5.

TABLE 4. Signal-to-noise (S/N) ratio values for the compressive strength and the porosity

Experiment No.	(S/N) ratio (compressive strength)	(S/N) ratio (Porosity)
1	36.52	29.73
2	34.04	30.08
3	30.53	29.78
4	28.15	26.68
5	26.60	19.58
6	29.60	28.39
7	25.99	24.53
8	15.74	27.73
9	19.99	30.76
10	16.01	25.84
11	17.35	29.02
12	9.79	35.48
13	10.91	36.36
14	27.75	30.88
15	21.87	26.71
16	8.82	37.46
17	15.96	36.29
18	8.46	38.49
19	15.49	29.84
20	12.85	32.27
21	18.94	38.07
22	20.56	35.70
23	13.19	32.47
24	17.72	36.26
25	8.82	37.64

TABLE 5. Normalized S/N ratio values for the compressive strength and the porosity

Experiment No.	Normalized S/N (Compressive strength)	Normalized S/N (Porosity)
1	1.00	0.54
2	0.91	0.56
3	0.79	0.54
4	0.70	0.38
5	0.65	0.00
6	0.75	0.47
7	0.62	0.26
8	0.26	0.43
9	0.41	0.59
10	0.27	0.33
11	0.32	0.49
12	0.05	0.84
13	0.09	0.89
14	0.69	0.59
15	0.48	0.38
16	0.01	0.94
17	0.27	0.88
18	0.00	1.00
19	0.25	0.54
20	0.16	0.67
21	0.37	0.98
22	0.43	0.85
23	0.17	0.68
24	0.33	0.88
25	0.01	0.95

The grey relation coefficients for the normalized S/N ratios, which were calculated according to Equation 4, are given in Table 6. These values are corresponding to a value of λ equal to 0.5 for the compressive strength, as well as the porosity. Next, by Equation 5, the grey relational grade could be computed. Finally, these grades were examined for optimizing the multi response parameter, design problem via Taguchi method.

The S/N ratio plot of grey relational coefficient, which combines the compressive strength and the porosity with respect to concentration of H_2O_2, quantity of the yeast, quantity of H_2O_2, polymerization time, and quantity of oil is shown in Figure 2, for a λ value of 0.5. It can be easily seen that the optimal parameter conditions are (A_5, B_1, C_1, D_3 and E_2). The subscript number indicates the level of the factor at which the optimal response could be obtained.

Similarly, the optimal multi response parameter can be obtained for the different values of λ as given in Table 7. Therefore, it can be seen that the suggested optimal conditions were similar for the some different values of λ; this indicates that Taguchi method is not sensitive for the minor changes in the values of λ. Moreover, according to the analysis of variance was found that the factor E, corresponding to the quantity of vegetable oil has a lowest rank among the studied factors. Keeping these in mind, one can reduce the number of the suggested optimal conditions to four experiments only, with the S/N ratio plot of grey relational coefficient given in Figure 3, as optimal parameter is given in Table 8.

These conditions were verified experimentally in the confirmation experiments. It is important to note that all the suggested optimal conditions have common low values of the factors B and C corresponding to the quantity of yeast and hydrogen peroxide, respectively. This indicates that these low values are necessary to achieve the optimal results regardless the values of the other parameters.

TABLE 6. Grey relational coefficients and grey grade values for *λ* values of 0.5

Experiment No.	GRC_{1j}	GRC_{i2}
1	1	0.52
2	0.85	0.53
3	0.70	0.52
4	0.63	0.44
5	0.59	0.33
6	0.67	0.48
7	0.57	0.40
8	0.40	0.47
9	0.46	0.55
10	0.41	0.43
11	0.42	0.49
12	0.34	0.76
13	0.35	0.82
14	0.62	0.55
15	0.49	0.45
16	0.34	0.90
17	0.41	0.81
18	0.33	1.00
19	0.40	0.52
20	0.37	0.60
21	0.44	0.96
22	0.47	0.77
23	0.38	0.61
24	0.43	0.81
25	0.34	0.92

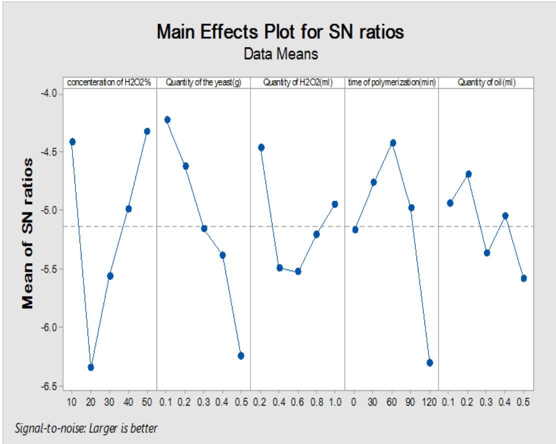

Figure 2. Effect of process parameters on the grey relational coefficient of geopolymer for λ value of 0.5

TABLE 7. Optimal parameter levels for different values of λ for compressive strength and porosity

λ Compressive strength	λ Porosity	A	B	C	D	E
0.1	0.9	10	0.1	0.2	0	0.1
0.2	0.8	10	0.1	0.2	60	0.1
0.3	0.7	10	0.1	0.2	60	0.1
0.4	0.6	10	0.1	0.2	60	0.2
0.5	0.5	50	0.1	0.2	60	0.2
0.6	0.4	50	0.1	0.2	60	0.2
0.7	0.3	50	0.1	0.2	60	0.2
0.8	0.2	50	0.1	0.2	90	0.2
0.9	0.1	50	0.1	0.2	90	0.2

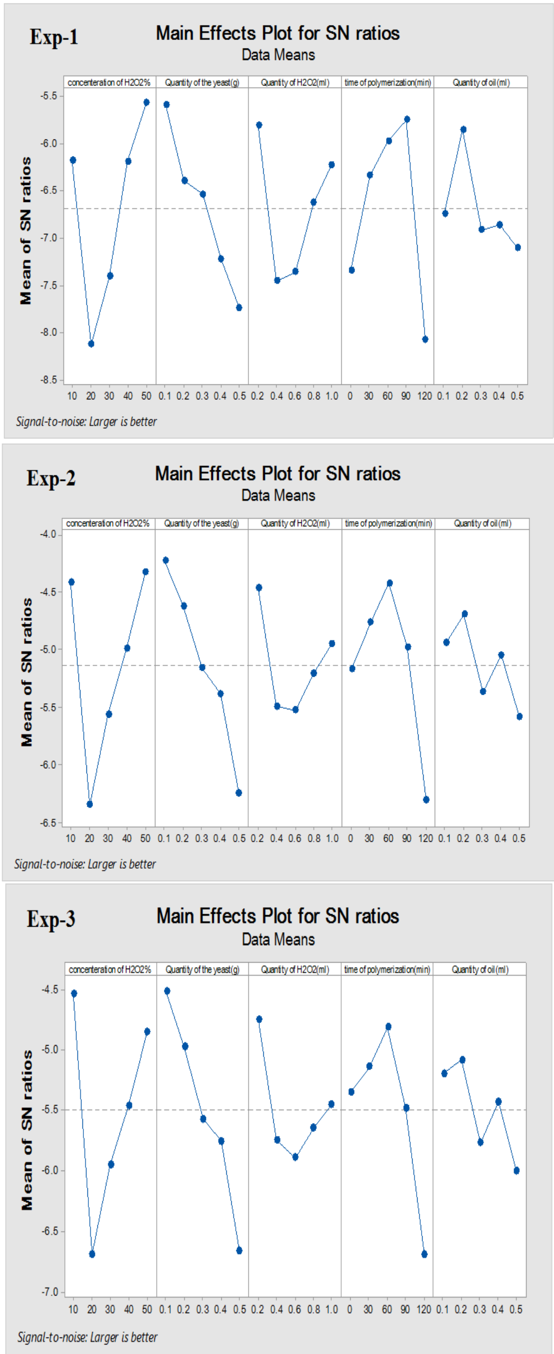

Figure 3. influence of process parameters on compressive strength and porosity of geopolymer for different values of λ

TABLE 8. Suggested optimal conditions for combined optimal compressive strength and porosity

Experiment Code	A	B	C	D	E
Exp-1	50	0.1	0.2	90	0.2
Exp-2	50	0.1	0.2	60	0.2
Exp-3	10	0.1	0.2	60	0.2
Exp-4	10	0.1	0.2	0	0.1

3. 2. Confirmation Experiments Emphasization experiments were carried out to the validate that the suggested optimal circumstances, can enhance of the compressive strength and porosity for the geopolymer. The experimental results to the four emphasization experiments performed at the optimal settings of process parameters. They have exposured that the responses can be enhanced effectually via suggested optimal circumstances as given in Table 9. It could be observed that the obtained values for the compressive strength and porosity from the confirmation tests, especially for Exp-3, combined high values of compressive strength, as well as porosity. These values are superior as compared with that obtained in Table 3 of the current study, also, they are superior to that reported for the metakaolin-based geopolymer in the literatures [2, 21, 23, 40, 41] as summarized in Table 10.

TABLE 9. Compressive strength and porosity obtained for the confirmation experiments

Experiment	Compressive strength (MPa)	Porosity (%)
Exp-1	15.66	26.30
Exp-2	11.42	38.90
Exp-3	88.30	22.00
Exp-4	66.95	30.67

TABLE 10. Compressive strength and porosity reported in many literature for the metakaolin-based geopolymer

Reference	Compressive Strength (Mpa)	Porosity (%)
[2]	0.26 - 5.9	28 - 83
[21]	0.3 - 11.6	66 - 83
[23]	3.64 - 7.60	62.5
[20]	1.45	82
[24]	0.35 - 56.5	50 - 86

4. CONCLUSIONS

The goal of the present work was to optimize both compressive strength and the porosity of geopolymer using Taguchi method, with the help of Grey relational analysis. It was found that this route of the optimization is suitable to fulfill the aim. However, the analysis showed that Taguchi method is not sensitive toward the minor changes in the identification coefficient λ of the grey relation alanalysis. Nevertheless, the experimental results showed that high porosity and adequate compressive strength can be obtained at the same geopolymer body by choosing the suitable values of the processing parameters. Moreover, it was found that the use of yeast as a catalyst and the polymerization time are important processing parameters. Also, it was noticed that the amount of the vegetable oil, which is used as a stabilizer, should be kept in low values to obtain the optimal compressive strength and porosity.

5. FUTUR WORK

According to the obtained results from the current study, the study proposes the following future works: 1) Studying the influence of utilizing higher potassium communicate on the characterisations of the geopolymer. 2) Studying the influence of dissimilar parameters on the improvement of compressive strength on the characterisations of fly ash based geopolymer higher potassium contact.

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Persian Abstract

چکیده

ژئوپلیمر یک ماده سازگار با محیط زیست است که می تواند با پارامترهای پردازش با انواع خواص فیزیکی و مکانیکی تولید شود. به دست آوردن ژئوپلیمر با مقاومت فشاری بالا و تخلخل زیاد ممکن است این ماده را برای بسیاری از کاربردهای حرارتی و فیزیکوشیمیایی به عنوان کاندیدای مطلوب تبدیل کند. هدف این تحقیق شناسایی مجموعه ای از پارامترهای پردازشی است که مانند این مواد ژئوپلیمر تولید می کند. روش تاگوچی همراه با تحلیل رابطه خاکستری برای حل این مشکل چند پاسخ استفاده شده است. تجزیه و تحلیل و نتایج تجربی نشان داد که می توان با استفاده از مقدار کم پراکسید هیدروژن به عنوان عامل کف کننده ، مقدار کم مخمر به عنوان کاتالیزور و مقدار کم روغن نباتی به عنوان تثبیت کننده به این هدف دست یافت. علاوه بر این ، زمان پلیمریزاسیون قبل از افزودن عامل کف کننده به عنوان یک پارامتر مهم پردازش سپری می شود. همچنین ، نتایج تجربی نشان داد که با انتخاب مقادیر مناسب پارامترهای پردازش ، می توان تخلخل بالا و مقاومت فشاری کافی را در یک جسم ژئوپلیمری یکسان بدست آورد. علاوه بر این ، مشخص شده است که استفاده از مخمر به عنوان کاتالیزور و زمان پلیمریزاسیون پارامترهای مهم پردازش است. همچنین ، توجه شده است که مقدار روغن نباتی ، که به عنوان تثبیت کننده استفاده می شود ، باید در مقادیر کم نگه داشته شود تا مقاومت فشاری و تخلخل مطلوب به دست آید.