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ORIGINAL ARTICLE

Factorial design approach to investigate the significance of factors on the fire resistant, compression and adhesion properties of geopolymer binder

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ABSTRACT – Despite numerous studies on the factors which influenced the properties of the geopolymer binder have been conducted, the effect of different factors on the properties of geopolymer binder was not well determined. In this research, geopolymer materials were analyzed and profiled using a statistical approach called 2-level fractional factorial design (2-FrFD). The objective is to screen and identify important factors affecting the behaviour of geopolymer binder. MINITAB, a statistical software, was used to design the experiment, analyze data obtained and present the significance value of the factors via chart and plots. The result showed that the curing temperature (V_3) did not have a significant effect on the fire resistant properties of the geopolymer binder with the p-value of 0.526. Other factors and interaction were significant with RHA/AA ratio (V_2) was the most significant factor with the coefficient value of 135. For the compression strength properties, all factor and interactions were significant (p-value between 0.000 and 0.009) with the RHA/AA ratio (V_2) recorded the highly significant factor with the coefficient value of 8.838. For adhesion strength properties, NaOH concentration (V_4) and curing time (V_5) were found to be insignificant with the p-value of 0.223 and 0.133, respectively. Other factors and interaction were significant curing temperature (V_3) was the most significant factor with the coefficient value of 0.287. This result may hugely benefit future researchers, towards producing halal and sustainable polymer, in determining the suitable factors which have a significant effect of the properties (outcome) of the geopolymer binder.

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Geopolymer; factorial design; significance; statistical analysis; MINITAB

INTRODUCTION

In order to maximize the properties of geopolymer, many factors have to be considered including ratio of alkali-activated (AA) solution, sodium hydroxide (NaOH) concentration, curing temperature and time, rice husk ash (RHA)/AA ratio, sintering temperature and time, the water content of the mixture, and size of aggregate. To develop a process involving such a large number of factors to obtain the optimum result using conventional or classical statistical method are laborious and time-consuming [1].

In geopolymer research, although past researchers have studied numerous factors which influenced the properties of the geopolymer binder, the significant effect of the factors on the properties was not well studied. In addition, the comparison of the significance of the factors studied cannot be made due probably to the lack of knowledge on the statistical analysis. From the factors mentioned above, there are five factors identified as the most studied factors in the geopolymer world which are; (i) ratio of AA solution such as ratio of sodium silicate (Na_2SiO_3) to NaOH, (ii) ratio of solid to liquid (S/L) such as ratio of RHA to AA solution, (iii) NaOH concentration, (iv) curing temperature, and (v) curing time.

Although all factors have been reported to have a significant effect on the properties of the geopolymer, limited discussion on the level of significance of the factor, either the factor is the most significant or the least significance, based on specific properties of the geopolymer. It is, therefore, necessary to identify efficient experimental design based on the objectives of the experiment and the number of factors to be investigated. Therefore, geopolymer materials can be analyzed and profiled by adopting a relatively new statistical approach called 2-level fractional factorial design (2-FrFD). To date, statistical analysis has been used in various field of studies [2-5].

Fractional factorial design (FrFD) is mostly selected as a statistical design method in many industrial applications and scientific investigations to identify important factors since it is economical and flexible. The approach is widely reported in the literature. The determination of optimum parameters for multifactor experiments can be enabled by the use of appropriate statistical and mathematical techniques as well as the careful selection of suitable experimental designs [6]. According to Wu and Hamada [7], estimation of lower-order effects is particularly effective for screening experiments which involve higher number of factors and lower number of runs. In this study, only the main effects and two-factor

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interactions (lower-order effect) are considered while higher-order effects (interaction between 3 factors and above) are considered negligible.

The 2-FrFD consists of only two levels, namely, high (+) and low (-) for each factor tested [8]. However, it can be increased to three levels per factor when center points are added in each block [9]. This type of design selects an adequately chosen fraction of the treatment combinations required for the complete factorial experiment to be run. Compared to FFD which offers up to 100 levels per factor (maximum 15 factors), 2-FrFD which offers only 2 or 3 levels per factor (maximum 15 factors) is still able to provide sufficient information (such as number of significant factors, the interaction between factors and optimum point) for further experimentation.

The objective of this research is to screen and identify important factors affecting the behaviour of geopolymer binder. The response obtained was statistically evaluated, and the model was developed based on the variables with confidence levels of more than 95.00%. This research will be highly important and useful towards further research in producing halal and sustainable polymer.

METHODS AND MATERIALS

Fire Resistant Test

Five factors, which have a possible significant effect on the responses (time taken to reach 300°C (TT300)), were chosen based on previous literature. The factors were the ratio of AA solution (V_1), RHA/AA ratio (V_2), curing temperature (V_3), NaOH concentration (V_4) and curing time (V_5). Low and high levels were shown in Table 1.

Table 1. Coded and uncoded values of the factors for fire resistant test

Factor	Notation	Coded low level	Uncoded low values	Coded high level	Uncoded high values
Ratio of AA solution	V_1	-1	3.5	+1	5.5
RHA/AA ratio	V_2	-1	0.3	+1	0.7
Curing Temperature	V_3	-1	50°C	+1	70°C
NaOH concentration	V_4	-1	8 M	+1	12 M
Curing Time	V_5	-1	7 days	+1	21 days

Figure 1 shows a flowchart for fabrication process of geopolymer binder. Geopolymer was first prepared by mixing the sodium hydroxide and sodium silicate solutions at designated ratios. The AA solution was mixed with RHA, and the mixture stirred gently for 30 seconds before using mechanical stirrer for another 30 minutes or until the solution becomes homogenous. The mixture was strained through a small sieve directly onto a mild steel plate (100 mm x 100 mm x 1 mm) which was previously cleaned using sandpaper and degreased with acetone. The coated substrate was cured in an oven. The coating thickness obtained was 1.0 ± 0.3 mm.

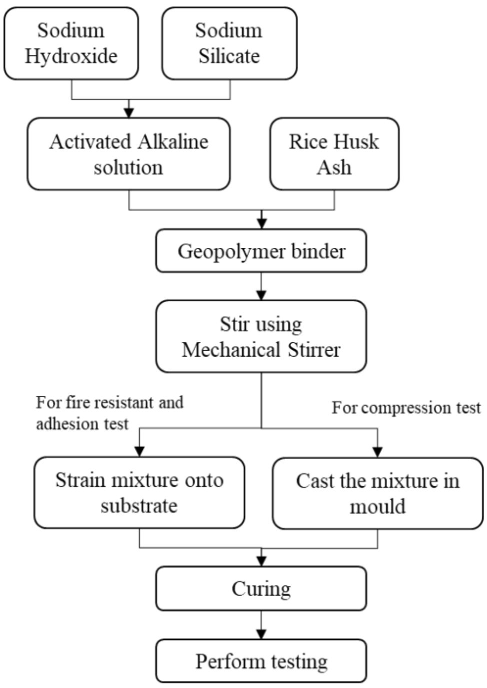

Figure 1. Flowchart for the fabrication process of geopolymer binder

The fire resistant tests as shown in Figure 2 were conducted by using the Thermacam S500 (infrared camera), and the recorded maximum temperature of the specimen was ensured to follow the ISO-834 standard fire curve. Distance between infrared camera and sample (X_1 = 60 cm) and between sample and blow (X_2 = 7 cm) were kept constant throughout the test. Bare and coated mild steel plates were exposed to fire for at least 20 minutes or until equilibrium temperature was reached. The temperature on the back of the mild steel plates was plotted as a function of time.

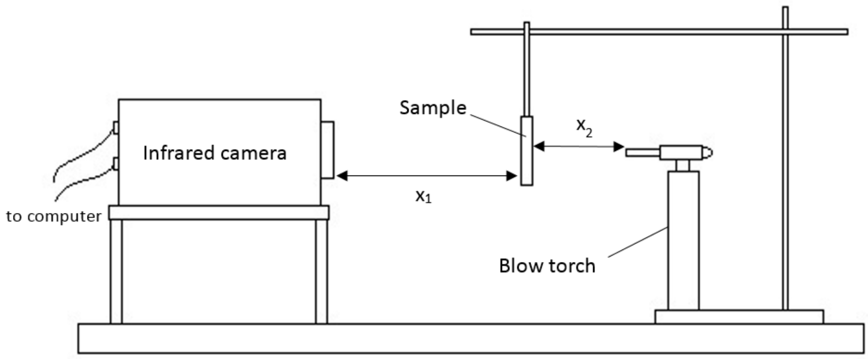

Figure 2. Schematic diagram of fire resistant test set-up

Compressive Test

Four factors were considered, which may potentially affect the response (compressive strength). The factors were the ratio of AA solution (V_1), RHA/AA ratio (V_2), curing temperature (V_3), and NaOH concentration (V_4). The coded and uncoded values of the factors are shown in Table 2. Based on the study conducted by many researchers, maximum compressive strength can be achieved within three days of curing [10]. A curing time of seven days was chosen since longer curing time would result in higher compressive strength and has been proved to affect the response significantly [11].

Table 2. Coded and uncoded values of the factors for compressive test

Factor	Notation	Coded low level	Uncoded low values	Coded high level	Uncoded high values
Ratio of AA solution	V_1	-1	3.5	+1	5.5
RHA/AA ratio	V_2	-1	0.3	+1	0.7
Curing Temperature	V_3	-1	50ºC	+1	70ºC
NaOH concentration	V_4	-1	8	+1	12

Preparation of the geopolymer binder was similar to that of the fire resistant test. For the compressive test, the mixture was strained and cast in a rectangle mould of dimension 12.7 mm x 12.7 mm x 25.4 mm. The specimen was then cured at a designated temperature. The test was conducted by using the Instron 3382 Floor Model Universal Testing System following ASTM D695 standard [12]. During testing, the load was applied at a constant speed of 1.30 ± 0.13 mm/min.

Adhesion Test

Five factors were considered in the experimental design with high potential to influence adhesion strength of RHA-based geopolymer coating which is the response. These were the ratio of AA solution (V_1), RHA/AA ratio (V_2), curing temperature (V_3), NaOH concentration (V_4), and curing time (V_5). The coded and uncoded values of the factors were shown in Table 3. The method in preparing the geopolymer binder is similar to that of the fire resistant test. The mixture was strained and coated onto a mild steel plate with a dimension of 50 mm x 50 mm x 1 mm. The sample was cured in an oven. The coating thickness obtained was 1.0 ± 0.3 mm. The test was conducted in accordance with ASTM D4541 [13] using an Elcometer 106 pull-off adhesion tester (0 MPa to 7 MPa).

Table 3. Coded and uncoded values of the factors for adhesion test

Factor	Notation	Coded low level	Uncoded low values	Coded high level	Uncoded high values
Ratio of AA solution	V_1	-1	3.5	+1	5.5
RHA/AA ratio	V_2	-1	0.3	+1	0.7
Curing Temperature	V_3	-1	50ºC	+1	70ºC
NaOH concentration	V_4	-1	8 M	+1	12 M
Curing Time	V_5	-1	7 days	+1	21 days

Experimental Analysis

The experiment was designed using two level-five factors fractional factorial (2-5 FFD). MINITAB, a statistical software, was used to generate a table of experiments to be conducted. Data obtained from experiments were inserted in the MINITAB software under response column and then analyzed. Figure 3 shows the flowchart for designing and analyzing using factorial design.

For analysis, a regression model was selected for up to 2-way interactions. The 2-way interactions refer to the interaction between two factors such as between the ratio of AA solution and curing temperature, curing temperature and time, etc. One of the criteria for determining the best-fitted model was by considering the p-value which provided a significant effect on the model and the value was given in an ANOVA table. The p of the factor or interaction was considered significant when it was lower than the confidence level of 95.00% (P = 0.050). Using coefficient and the terms from the ANOVA table, the regression equation was formulated. Plots were generated for further analysis, including coefficient plot, main effect, and interaction plots.

Coefficient plot greatly facilitates in resolving the magnitude and the importance of both main and interaction effects. The coefficient plot is developed using the coefficient values in the ANOVA table. The negative or positive sign of the coefficient values is ignored since it did not have any effect in determining the significance of the effects and interactions. Graph of the factor or interaction which is the longest is said to have the most significant effect on the response and vice versa. The main effect plot produces the mean response values between two levels of a factor, and the horizontal line represents the mean compressive strength for all runs. An interaction plot is a graphical tool which shows the impact of all possible combinations of factors on the response. This plot is crucial since it shows the relationship between two factors. If both lines are not parallel or intersect with each other, there is an interaction between the two factors.

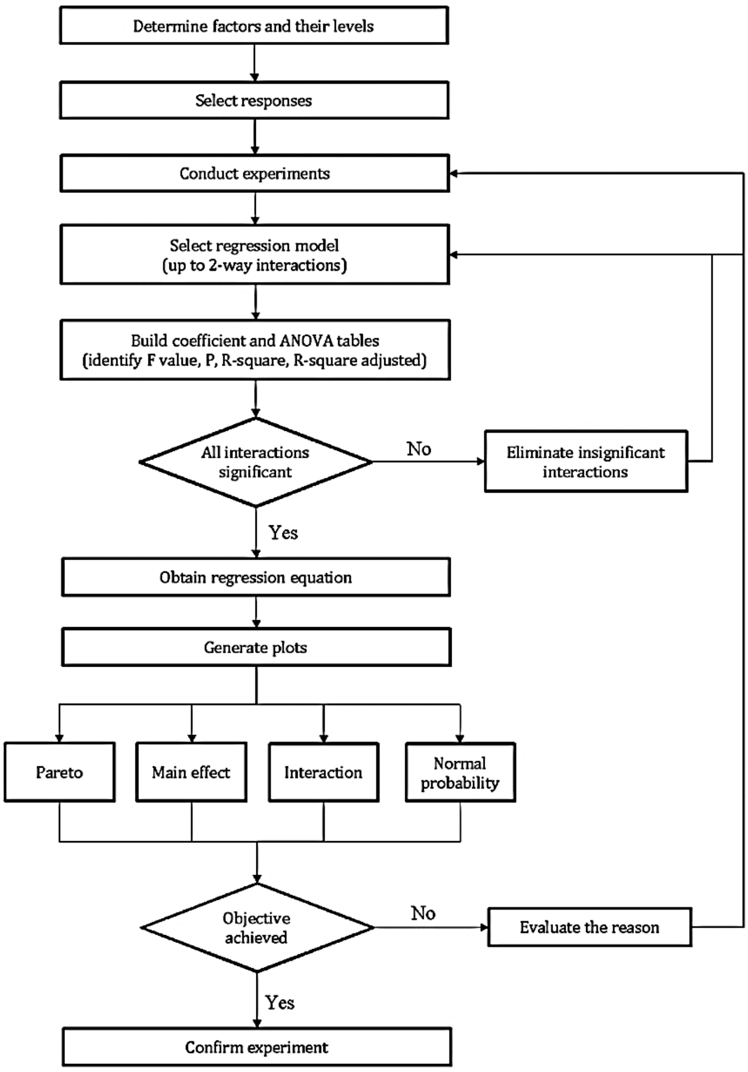

Figure 3. Flowchart of factorial design

RESULTS AND DISCUSSION

Fire Resistant Test

The uncoded (actual value) design matrix for the factors and all responses for 32 experimental runs including replication are shown in Table 4. Data shown in Table 4 was adopted from Mohd Basri, et al. [14] and further analysis on the data obtained was conducted to verify the coefficient plot, main effect, and interaction plots.

Table 4. Experimental results of fire resistant test [7]

Sample	V_1	V_2	V_3	V_4	V_5	TT300
S1	3.5	0.7	70	8	21	234
S2	3.5	0.7	70	12	7	260
S3	3.5	0.7	50	12	21	300
S4	3.5	0.3	50	12	7	218
S5	3.5	0.7	50	8	7	215
S6	5.5	0.3	70	12	7	405
S7	5.5	0.7	50	12	7	208
S8	5.5	0.3	50	12	21	432
S9	3.5	0.3	70	12	21	297
S10	3.5	0.3	70	8	7	767
S11	3.5	0.3	70	12	21	292
S12	3.5	0.7	50	8	7	263
S13	3.5	0.3	70	8	7	750
S14	5.5	0.3	50	8	7	991
S15	5.5	0.7	70	12	21	236
S16	5.5	0.7	50	12	7	152
S17	5.5	0.3	70	8	21	488
S18	3.5	0.3	50	8	21	438
S19	3.5	0.3	50	12	7	263
S20	3.5	0.7	70	8	21	227
S21	3.5	0.7	70	12	7	263
S22	5.5	0.7	70	8	7	214
S23	5.5	0.3	70	12	7	400
S24	3.5	0.7	50	12	21	305
S25	5.5	0.3	70	8	21	508
S26	5.5	0.7	70	8	7	399
S27	5.5	0.3	50	12	21	474
S28	5.5	0.7	50	8	21	180
S29	3.5	0.3	50	8	21	441
S30	5.5	0.7	50	8	21	200
S31	5.5	0.3	50	8	7	1025
S32	5.5	0.7	70	12	21	213

TT300 = Time taken to reach 300°C (in second)

In Table 5, except for curing temperature which showed insignificant values ($P > 0.050$) and had no effect on the responses, the other factors were highly significant ($P \leq 0.000$) at 95.00% confidence level. V_2 was the most significant factor, followed by V_4 and V_5. The value for R^2 (0.9785) and R^2 (adjusted) (0.9650) were very high as well. The value of the effects in the table determined either the factor had a higher or lower effect on the response. Interaction between V_1 and V_2 had the greatest influence on the response with 48.34, followed by "V_2*V_4" and "V_3*V_5" with 29.75 and 28.30, respectively. Other factors and interactions showed smaller effects between 20.31 and 6.42.

Table 5. Estimated effects and coefficient for TT300 in fire resistant test

Notation	Net Effect	Coefficient	Std. error of coefficient	Standardize Effect	P
Constant		376.8	7.360	51.20	0.000
V_1	62.0	31.0	7.360	4.21	0.000
V_2	-270.0	-135.0	7.360	-18.34	0.000
V_3	-9.5	-4.7	7.360	-0.65	0.526
V_4	-163.9	-81.9	7.360	-11.13	0.000
V_5	-95.5	-47.7	7.360	-6.49	0.000
V_1*V_2	-95.1	-47.6	7.360	-6.46	0.000
V_1*V_3	-90.4	-45.2	7.360	-6.14	0.000
V_1*V_5	-37.4	-18.7	7.360	-2.54	0.020
V_2*V_3	37.4	18.7	7.360	2.54	0.020
V_2*V_4	164.5	82.2	7.360	11.18	0.000
V_2*V_5	85.6	42.8	7.360	5.82	0.000
V_4*V_5	143.0	71.5	7.360	9.72	0.000

R^2 = 0.9785 R^2 (adj) = 0.9650

Using coefficient and the terms from Table 5, the regression models can be formulated as shown in Eq. (1) where Y_{TT300} is the response of TT300, V_1 is the ratio of AA solution, V_2 is RHA/AA ratio, V_3 is curing temperature, V_4 is NaOH concentration, and V_5 is curing time.

$$Y_{TT300} = 376.8 + 31.0\,(V_1) - 135.0\,(V_2) - 4.7\,(V_3) - 81.9\,(V_4) - 47.7\,(V_5) - 47.6\,(V_1V_2) - 45.2\,(V_1V_3) - 18.7\,(V_1V_5) + 18.7\,(V_2V_3) + 82.2\,(V_2V_4) + 42.8\,(V_2V_5) + 71.5\,(V_4V_5) \quad (1)$$

Figure 4 shows the coefficient plot on the effects of main factors and their interactions. RHA/AA ratio (V_2) was found to be the most influential since it extended the most. Except for curing temperature, which had no effect on the TT300, other factors were statistically significant at 95% confidence level.

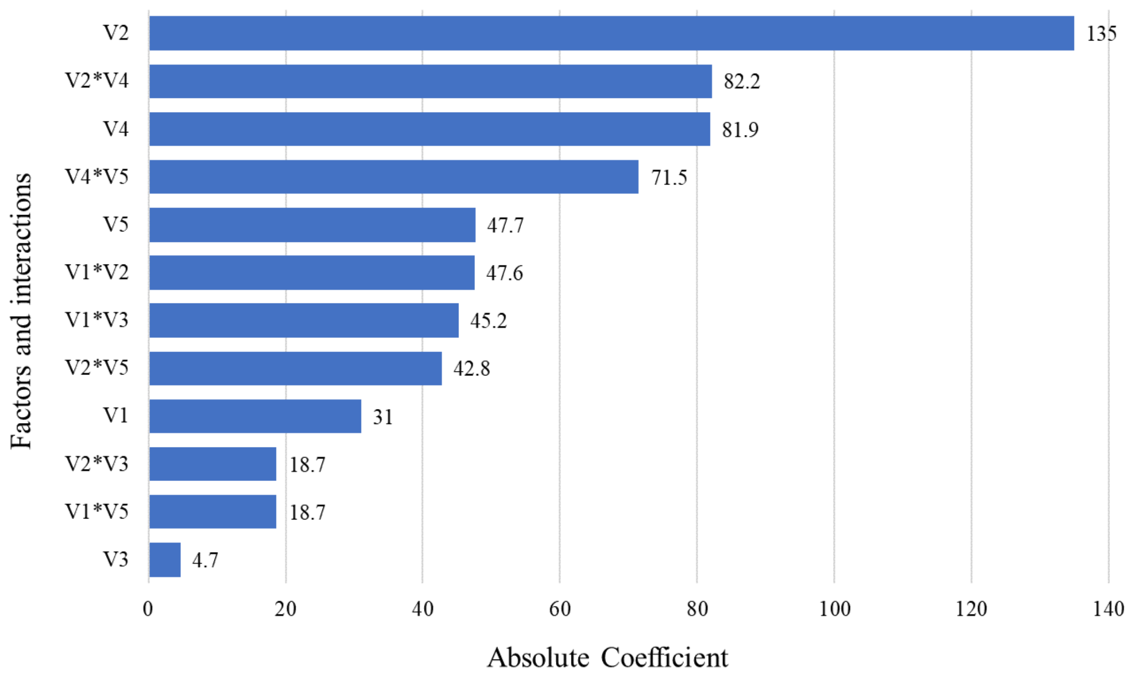

Figure 4. Coefficient plot of the factors on the TT300

As the curing temperature increased, the sample exhibited loss of water due to continuous evaporation of capillary water. Water completely evaporated during fire resistant as well. Although the amount of water evaporated at different curing temperature varies between samples, the geopolymer binder was mainly affected by the size and volume of pores when in contact with fire [15]. Compared to other interactions, those occurring between factors RHA/AA ratio and NaOH concentration (V_4) collectively had the strongest effect on both responses. On this basis, factors V_2 and V_4 were thus identified for the subsequent optimization process.

It can be seen from Figure 5 that the RHA/AA ratio has the greatest negative effect on the TT300 as indicated by the larger angle of inclination to the horizontal. All other factors showed smaller changes and most of the factors have negative effects on the response. This pattern proved to be statistically significant, as shown in Table 4.

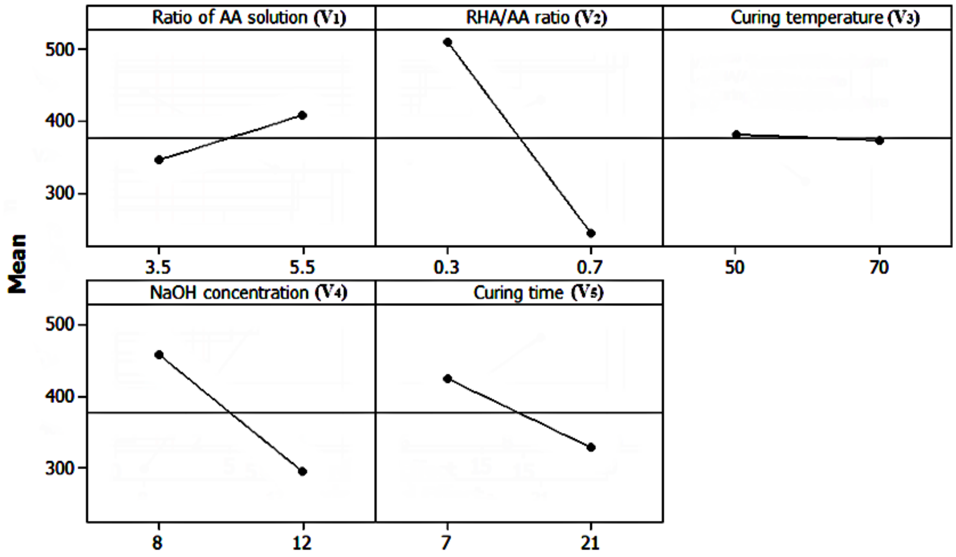

Figure 5. Main effect plot for TT300 in fire resistant test

As shown in Figure 6, the effect of V_4 was considered the strongest when it interacted with V_2 and V_5. Lines for V_1 and V_4 are almost parallel which indicate that the interaction between both factors is not significant. Regardless of the V_3, V_4, and V_5, lower V_2 of 0.3 produced a better result and vice versa. With lower V_4 of 8M, longer TT300 was achieved when the V_2 and V_5 are 0.3 and seven days, respectively. The V_1 and V_5 showed slight interaction with each other since their lines are approximately mutually parallel and somewhat connected at one end.

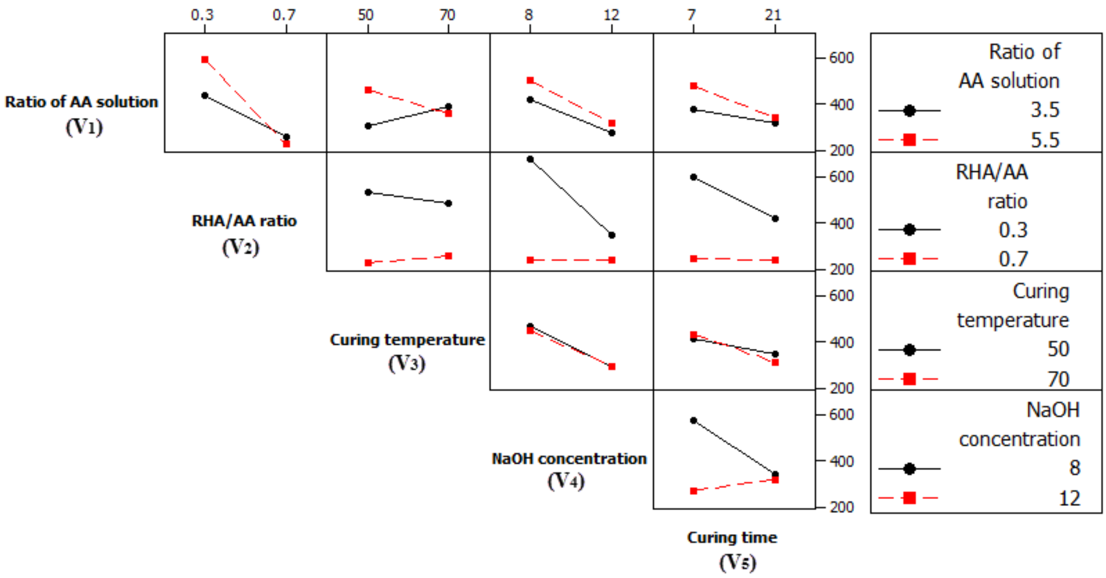

Figure 6. Interaction plot for TT300 in fire resistant test

Compression Test

The uncoded (actual value) design matrix for the factors and all responses for 16 experimental runs including replication are shown in Table 6.

In Table 7, all individual and interaction effects were shown to be significant at 95% confidence level. The effect of V_2 was highly significant with $P < 0.000$ while that of "V_2*V_4", very significant with $P < 0.000$. The value for R^2 of 0.9910 and R^2 (adjusted) of 0.9832 was high indicating good fit for the model with 99.10% of the results explainable. The model also showed high dependence and correlation between the observed and the predicted response values. The value of the net effects indicates the level of effect the factor has on the response. The RHA/AA ratio (V_2) had the greatest influence on the response with the value 17.675, followed by V_4 and "V_2*V_4" at 3.765 and 3.720, respectively. Other factors and interactions showed smaller effects ranging between 3.493 and 2.241.

Table 6. Experimental results of compressive test

Sample	V_1	V_2	V_3	V_4	Compressive Strength
S1	3.5	0.3	50	8	0.029
S2	5.5	0.3	70	8	0.203
S3	5.5	0.3	50	12	0.090
S4	5.5	0.7	70	12	13.75
S5	3.5	0.7	50	12	15.61
S6	3.5	0.7	70	8	14.80
S7	5.5	0.7	50	8	29.47
S8	3.5	0.3	70	12	0.050
S9	3.5	0.7	70	8	16.72
S10	3.5	0.7	50	12	14.77
S11	3.5	0.3	50	8	0.025
S12	5.5	0.7	70	12	11.97
S13	5.5	0.3	50	12	0.083
S14	5.5	0.3	70	8	0.200
S15	5.5	0.7	50	8	25.05
S16	3.5	0.3	70	12	0.057

Table 7. Estimated effects and coefficient for compressive test

Notation	Net Effect	Coefficient	Std. error of coefficient	Standardize Effect	P
Constant		8.930	0.3253	27.45	0.000
V_1	2.344	1.172	0.3253	3.60	0.007
V_2	17.675	8.838	0.3253	27.16	0.000
V_3	-3.422	-1.711	0.3253	-5.26	0.001
V_4	-3.765	-1.882	0.3253	-5.79	0.000
V_1*V_2	2.241	1.120	0.3253	3.44	0.009
V_2*V_3	-3.493	-1.746	0.3253	-5.37	0.001
V_2*V_4	-3.720	-1.860	0.3253	-5.72	0.000
$R^2 = 0.9910$	R^2 (adj) = 0.9832				

Using coefficient and terms from Table 7, the regression model developed as shown in Eq. (2) where Y_{CS} is the response of compressive strength, V_1 is the ratio of AA solution, V_2 is RHA/AA ratio, V_3 is curing temperature, and V_4 is NaOH concentration.

$$Y_{CS} = 8.930 + 1.172\,(V_1) + 8.838\,(V_2) - 1.711\,(V_3) - 1.882\,(V_4) + 1.120\,(V_1V_2) - 1.746\,(V_2V_3) - 1.860\,(V_2V_4) \tag{2}$$

Figure 7 shows the coefficient plot of the coefficient values. From the chart, RHA/AA ratio (V_2) was found to be the most influential factor for compressive strength as the factor extends well. Other factors are statistically significant at 95% confidence level and had smaller effects.

Figure 8 shows the main effect plot which produces the mean response values between two levels of a factor. The horizontal line represents the mean compressive strength for all runs. From the plot, V_2 has the greatest positive effect on compressive strength due to larger angle of inclination to the horizontal. All other factors showed smaller changes in mean compressive strength with curing temperature (V_3) and NaOH concentration (V_4) registering negative effect while the ratio of AA solution (V_1) showed positive effect on the response. This pattern established the statistical significance of the factors and their interactions as shown in Table 7.

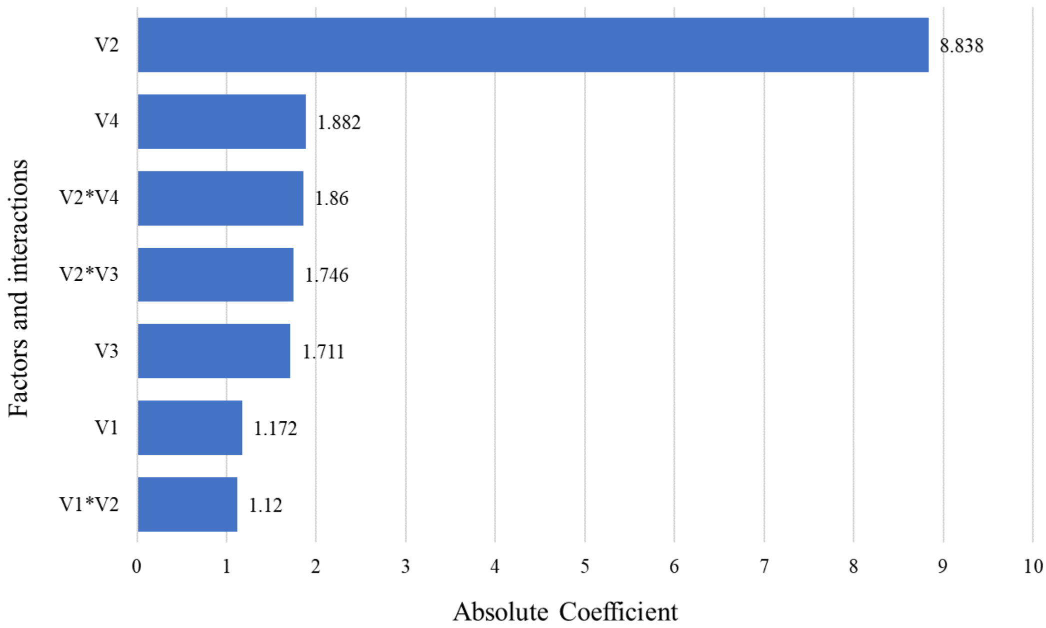

Figure 7. Coefficient plot of the factors on compressive strength

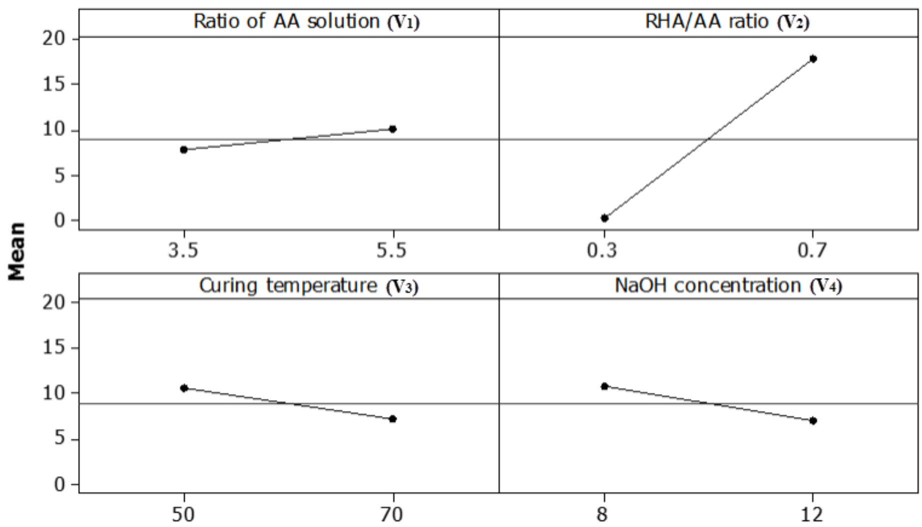

Figure 8. Main effect plot for compressive strength

It can be seen from Figure 9 that RHA/AA ratio (V_2) strongly interacted with two out of three factors such as curing temperature (V_3) and NaOH concentration (V_4). This indicates that V_2 is a predominant influencing factor in compressive strength. The high mean value for V_2 of about 0.7 indicates high compressive strength. Whether sample cured at low curing temperature of 50°C or high curing temperature of 70°C, the compressive strength achieved was low when V_2 is 0.3. Low value of V_4 of 8M and high value of V_1 at 5.5 resulted in high compressive strength. V_1 and V_2 showed slight interaction with each other since their lines are approximately mutually parallel. V_3 and V_4 also showed slight interaction with lower V_3 at 50°C producing high compressive strength at 8M and 12M, respectively.

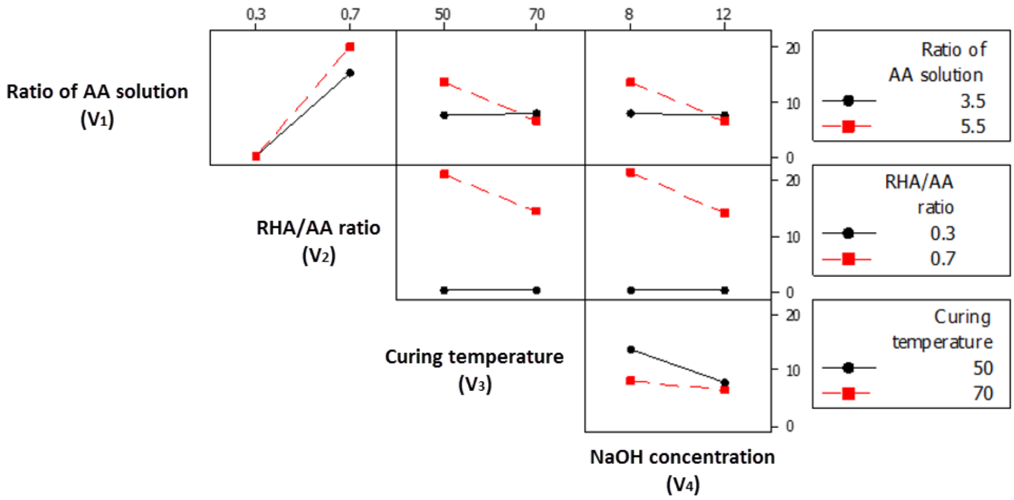

Figure 9. Interaction plot for compressive strength

Adhesion Test

The uncoded (actual value) design matrix for the factors and all responses for 32 experimental runs including replication are shown in Table 8.

Table 8. Experimental results adhesion test

Sample	V_1	V_2	V_3	V_4	V_5	Adhesion Strength
S1	5.5	0.3	50	12	21	1.4
S2	3.5	0.3	50	8	21	2.8
S3	5.5	0.3	70	12	7	4.3
S4	5.5	0.7	70	12	21	2.1
S5	3.5	0.7	70	8	21	2.7
S6	3.5	0.7	50	12	21	3.2
S7	5.5	0.7	70	8	7	1.8
S8	5.5	0.3	70	8	21	2.5
S9	3.5	0.3	70	8	7	2.5
S10	5.5	0.7	50	8	21	1.4
S11	3.5	0.7	70	8	21	2.9
S12	5.5	0.3	70	8	21	1.8
S13	3.5	0.3	70	8	7	2.5
S14	3.5	0.7	50	8	7	2.3
S15	5.5	0.3	50	12	21	1.4
S16	3.5	0.3	70	12	21	2.2
S17	3.5	0.7	70	12	7	2.3
S18	5.5	0.3	50	8	7	2.5
S19	5.5	0.7	50	12	7	1.5
S20	3.5	0.7	50	8	7	2.0
S21	3.5	0.3	70	12	21	2.8
S22	3.5	0.3	50	8	21	2.8
S23	3.5	0.7	70	12	7	2.6
S24	5.5	0.7	70	8	7	1.8
S25	5.5	0.3	70	12	7	4.6
S26	5.5	0.3	50	8	7	2.8
S27	3.5	0.7	50	12	21	2.8
S28	3.5	0.3	50	12	7	1.5
S29	5.5	0.7	50	8	21	1.0
S30	3.5	0.3	50	12	7	1.4
S31	5.5	0.7	70	12	21	2.1
S32	5.5	0.7	50	12	7	1.5

The P in Table 9 is important in determining the significance of the effects in the model. Since the confidence level is set to be 95.00% ($P \leq 0.05$) the effect is considered statistically significant. The results showed that all individual and two-way interaction effects were significant at 95.00% confidence level except for V_4 and V_5. The effects of V_2 and V_3 were highly significant ($P < 0.000$) while that of V_1, very significant ($P < 0.001$). The values for R^2 (0.9572) and R^2 (adjusted) (0.9220) were quite high indicating a very good fit for the model with 95.72% explained results. The value of the net effects determined whether the factor had higher or lower effect on the response. The interaction between V_1 and V_5 had the greatest influence on the response with 0.7625 followed by "V_1*V_2" and V_3 with 0.6500 and 0.5750 respectively. Other factors and interactions showed smaller effects between 0.4625 and 0.1000.

Table 9. Estimated effects and coefficient for adhesion strength

Notation	Net Effect	Coefficient	Std. error of coefficient	Standardize Effect	P
Constant		2.306	0.0396	58.30	0.000
V_1	-0.300	-0.150	0.0396	-3.79	0.001
V_2	-0.363	-0.181	0.0396	-4.58	0.000
V_3	0.575	0.287	0.0396	7.27	0.000
V_4	0.100	0.050	0.0396	1.26	0.223
V_5	-0.125	-0.062	0.0396	-1.58	0.133
V_1*V_2	-0.650	-0.325	0.0396	-8.22	0.000
V_1*V_3	0.363	0.181	0.0396	4.58	0.000
R^2 = 0.9572	R^2 (adj) = 0.9220				

Using the information on coefficient and terms in Table 9, the regression model can be formulated as shown in Eq. (3) where Y_{AS} = response (adhesion strength), V_1 = ratio of AA solution, V_2 = RHA/AA ratio, V_3 = curing temperature, V_4 = NaOH concentration, and V_5 = curing time.

$$Y_{AS} = 2.3062 - 0.1500 (V_1) - 0.1813 (V_2) + 0.2875 (V_3) + 0.0500 (V_4) - 0.0625 (V_5) - 0.3250 (V_1V_2) + 0.1812 (V_1V_3) + 0.1562 (V_1V_4) - 0.3812 (V_1V_5) - 0.1250 (V_2V_3) + 0.0875 (V_2V_4) + 0.2125 (V_2V_5) + 0.2313 (V_3V_4) - 0.1437 (V_3V_5) \quad (3)$$

Figure 10 shows the coefficient plot on the effects. The plot shows that V_3 was found to be the most influential factor for adhesion strength. Other factors, including V_2 and V_1 were statistically significant at 95% confidence level and had smaller effects. Curing time was found to be not significant due probably to the initial binding condition during curing process. As the sample cured, tension in the capillary pores increase resulting in a volume reduction (shrinkage) and simultaneously binding to the substrate [16]. Further curing does not change the inter-chemical bonding between geopolymer binder and substrate.

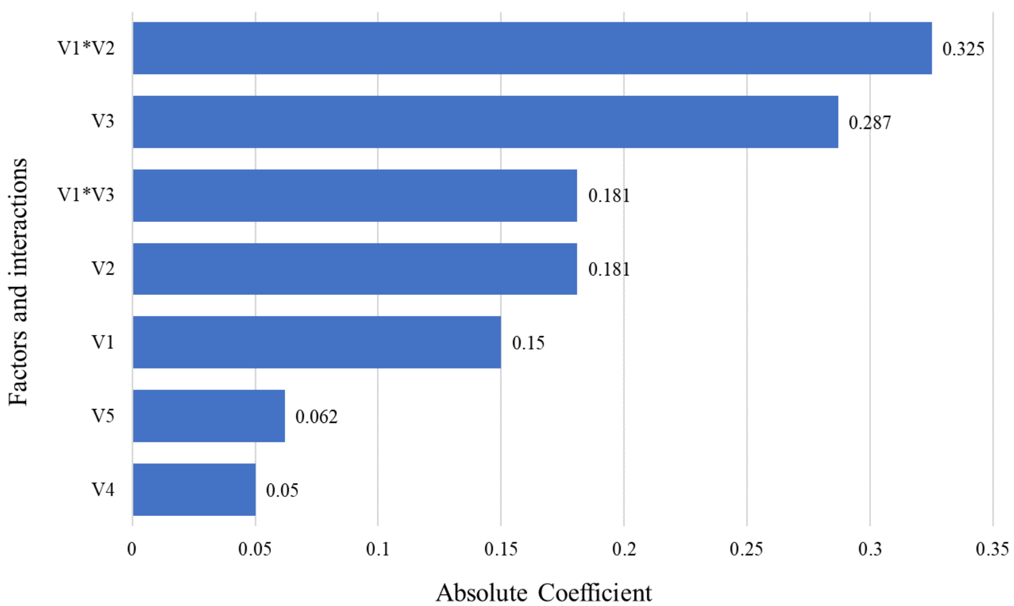

Figure 10. Coefficient plot of the factors for adhesion strength

Figure 11 shows the main effect plot which indicates that V_3 has the greatest positive effect on adhesion strength due to the larger angle of inclination to the horizontal. V_1 and V_2 show relatively strong negative effects on the response while the remaining two factors (V_4 and V_5) show a smaller variation in mean adhesion strength indicating the minimal effect on the response. This pattern concurs with statistical significance shown in Table 9.

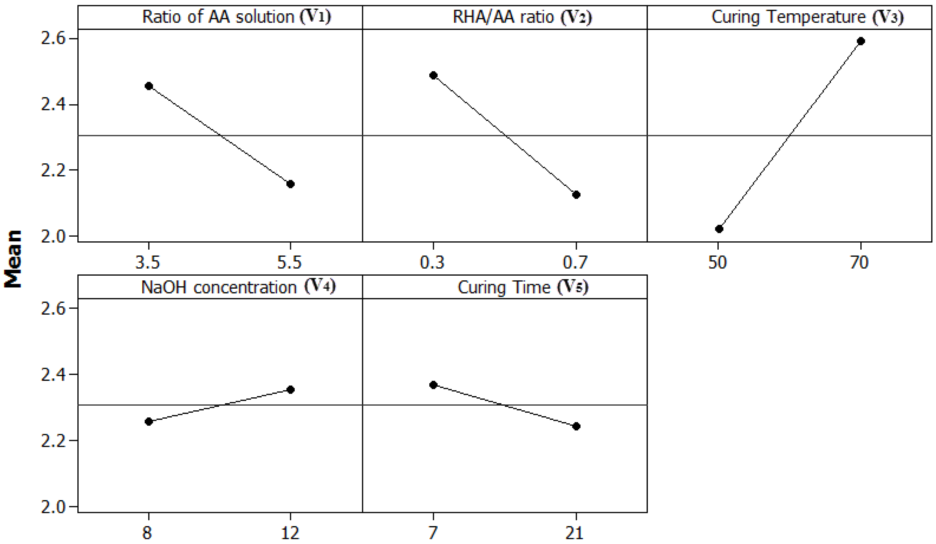

Figure 11. Main effect plot for adhesion strength

Figure 12 shows that the low V_3 (50°C) appeared to produce low adhesion strength regardless of V_4 and V_5. Higher V_1 (5.5) produced higher adhesion strength, recorded at V_2 and V_3 of 0.3 and 70°C, respectively. V_2 and V_4 showed slight interaction with each other since their lines did not intersect and were approximately mutually parallel.

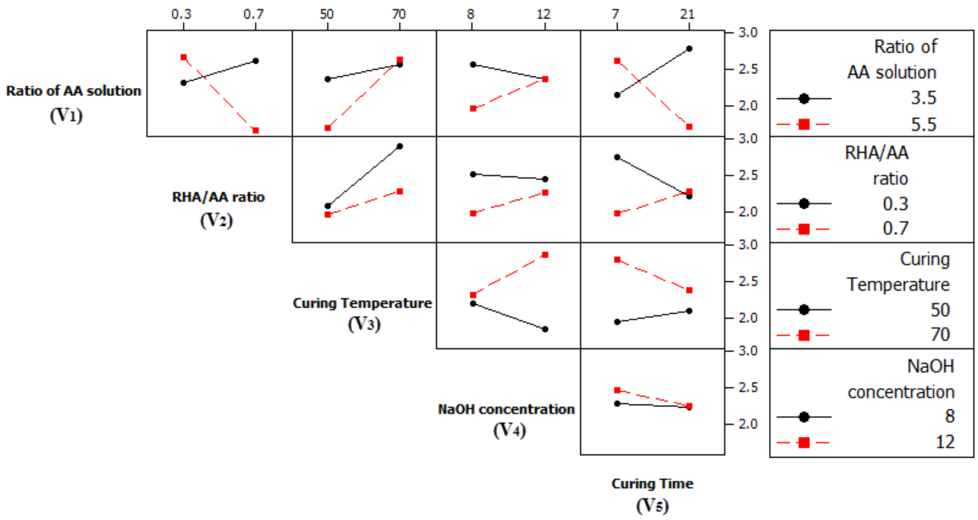

Figure 12. Interaction plot for adhesion strength

Significance of Factors

From Table 10, it can be concluded that the RHA/AA ratio is the most significant factor which highly affects the properties of the geopolymer binder. The curing temperature was found to not have any effect on the fire resistant properties while a study on the adhesion properties of the geopolymer binder should focusing on the factors other than the NaOH concentration and curing time.

Table 10. The significance of factor affecting the properties of geopolymer

Factor	Properties		
	Fire resistant	Compression	Adhesion
Ratio of AA solution	Significant	Significant	Significant
RHA/AA ratio	Highly significant	Highly significant	Significant
Curing temperature	Not significant	Significant	Highly significant
NaOH concentration	Significant	Significant	Not significant
Curing time	Significant	-	Not significant

CONCLUSIONS

For fire resistant test, results for TT300 showed that all factors and their interaction effects were significant except for curing temperature. The interaction between NaOH concentration and RHA/AA ratio were highly significant while that of RHA/AA ratio, the most significant factor. Similar results obtained for compression test with all factors were significant with RHA/AA ratio to have the most effect on the compressive strength of the geopolymer binder panel. For adhesion test, all factors and interactions were significant except for the curing time and NaOH concentration.

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REFERENCES

[1] K. Karthikeyan, K. Nanthakumar, K. Shanthi, and P. Lakshmanaperumalsamy, "Response surface methodology for optimization of culture conditions for dye decolorization by a fungus, Aspergillus niger HM11 isolated from dye affected soil," Iranian Journal of microbiology, vol. 2, no. 4, p. 213, 2010.

[2] M. Mazliah et al., "Optimization of physical and mechanical properties of glycerol-modified natural rubber/starch-filled carbon black composites using two level factorial design," Journal of Mechanical Engineering and Sciences, vol. 13, no. 2, pp. 4989-5005, 2019.

[3] N. Hussein, S. Laily, M. Salleh, and M. Ayof, "Statistical analysis of second repair welding on dissimilar material using Taguchi method," Journal of Mechanical Engineering and Sciences, vol. 13, no. 2, pp. 5021-5030, 2019.

[4] S. D. Sabdin, N. I. S. Hussein, M. K. Sued, M. Ayob, M. Rahim, and M. Fadzil, "Effects of ColdArc welding parameters on the tensile strengths of high strength steel plate investigated using the Taguchi approach," Journal of Mechanical Engineering and Sciences, vol. 13, no. 2, pp. 4846-4856, 2019.

[5] N. Ambhore, D. Kamble, and S. Chinchanikar, "Analysis of tool vibration and surface roughness with tool wear progression in hard turning: An experimental and statistical approach," Journal of Mechanical Engineering and Sciences, vol. 14, no. 1, pp. 6461-6472, 2020.

[6] M. Ibrahim, S. Sapuan, and A. Faieza, "Mechanical and thermal properties of composites from unsaturated polyester filled with oil palm ash," Journal of Mechanical Engineering and Sciences, vol. 2, pp. 133-147, 2012.

[7] C. J. Wu and M. S. Hamada, Experiments: planning, analysis, and optimization. John Wiley & Sons, 2011.

[8] K. Narayanan, V. Subrahmanyam, and J. Venkata Rao, "A fractional factorial design to study the effect of process variables on the preparation of hyaluronidase loaded PLGA nanoparticles," Enzyme research, vol. 2014, 2014.

[9] P.-W. Tsai, S. G. Gilmour, and R. Mead, "Statistical isomorphism of three-level fractional factorial designs," Utilitas Mathematica, vol. 70, pp. 3-10, 2006.

[10] M. I. Khan, K. Azizli, S. Sufian, and Z. Man, "Effect of Na/AI and Si/AI Ratios on Adhesion Strength of Geopolymers as Coating Material," Applied Mechanics & Materials, no. 625, 2014.

[11] S. Songpiriyakij, T. Kubprasit, C. Jaturapitakkul, and P. Chindaprasirt, "Compressive strength and degree of reaction of biomass-and fly ash-based geopolymer," Construction and Building Materials, vol. 24, no. 3, pp. 236-240, 2010.

[12] S. T. M. f. C. P. o. R. Plastics, "ASTM D695–15," ed: ASTM International West Conshohocken, PA.

[13] A. Standard, "Standard Test Method for Pull-Off Strength of Coatings using Portable Adhesion Testers (ASTM D4541)," ASTM International: West Conshohocken, PA, 2009.

[14] M. S. Mohd Basri, F. Mustapha, N. Mazlan, and M. R. Ishak, "Fire Retardant performance of rice husk ash-based geopolymer coated mild steel-A factorial design and microstructure analysis," in Materials Science Forum, 2016, vol. 841: Trans Tech Publ, pp. 48-54.

[15] S. Alehyen, M. Zerzouri, M. ELalouani, M. El Achouri, and M. Taibi, "Porosity and fire resistance of fly ash based geopolymer," Journal of Materials and Environmental Sciences, vol. 9, pp. 3676-3689, 2017.

[16] L. Biondi, M. Perry, C. Vlachakis, Z. Wu, A. Hamilton, and J. McAlorum, "Ambient cured fly ash geopolymer coatings for concrete," Materials, vol. 12, no. 6, p. 923, 2019.