Application of Response Surface Methodology for Mortar Compressive Strength Containing Glass Powder and Eggshell Powder

Adanu Olekwu Jessica[1], Adetoye Olubunmi[2], Olubajo Olumide Olu[1]

[1]Chemical Engineering Department, Abubakar Tafawa Balewa University, Bauchi, Nigeria
[2]Civil Engineering Department, Abubakar Tafawa Balewa University, Bauchi, Nigeria

*Corresponding author: ooolubajo@atbu.edu.ng

Abstract: This research investigates the impact of Glass Powder (GP) and Eggshell Powder (ESP) on the strength properties of various ternary cement blends, focusing particularly on mortar strength. The study explores the possibility of utilizing industrial waste as a viable replacement for Portland Limestone Cement in response to the growing environmental concerns and the increased energy requirements of non-renewable construction materials. In recent times, the application of waste materials such as GP and ESP in the concrete industry has gained considerable attention due to the associated environmental benefits and to manage the accumulation of these materials. Both GP and ESP contain substantial silica content, making them prospective materials for cement substitutes. The study specifically explores the combined effect of these materials on cement blends, which could potentially improve the cement's properties by mitigating some undesirable effects. In line with this, several tests, including X-ray fluorescence (XRF) were performed on the sample materials to understand their inherent properties and potential interaction when combined in cement blends. The mechanical properties of the resultant mortar were also examined. Using the response surface methodology via Central Composite Design (CCD), the study identified the optimal blending ratio, replacement level, and curing age for the cement blend. A blending ratio of 0.75, a replacement level of 8 wt.%, and a curing age of approximately 60 days resulted in a mortar compressive strength of 41.53N/mm^2. The study found that curing age significantly influenced the strength gain attributed to an increase in the hydration reaction time during curing from 3 to 60 days. These findings suggest that using GP and ESP as partial replacements for cement could not only alleviate environmental burdens but also yield significant strength gain in mortar, underlining the promise of these materials in sustainable construction practices. Future research is encouraged to validate these findings and explore large-scale, real-world applications of this approach.

Keywords: *Cement replacement, Eggshell powder, Glass powder, Optimization, Strength*

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1. Introduction

The performance of waste glass and other pozzolans in mortar is influenced by their chemical compositions, fineness, and pore sizes. It is also dependent on the raw materials used but differs slightly for each glass type (Islam *et al*., 2017). As an amorphous substance with large amounts of silicon oxide, iron oxide, and aluminum oxide above 70 wt. %, waste glass is considered an excellent pozzolanic material. The eggshell mostly contains calcium and magnesium carbonate (lime), and it is a common understanding that dried eggshells are a good source of calcium for animal feeds. The quality of lime in eggshell waste is influenced mainly by its exposure to sunlight, water, and harsh weather conditions (Gowsika *et al*., 2014). Eggshell waste powder is a commonly available byproduct, and its utilization as a cement additive presents an eco-friendly solution.

The recent introduction of wastes in concrete and mortar production has led to considerable improvements in their performance, saving costs and reducing the problems associated with the use of cement in building construction (Adetoye *et al*., 2023; Afolayan *et al*., 2022; Olubajo *et al*., 2017). This research seeks to assess the effect of glass powder and eggshell powder on the mortar strength of ternary cement blends. The findings of this research contribute to the knowledge base regarding sustainable construction materials and provide valuable information for construction industries, builders, architects, structural engineers, and policymakers.

2. Materials and methods

2.1. Experimental Design

The design summary for the dependent variables is the mortar strengths via Central Composite Design (CCD) and Box-Behnken Design (BBD). The independent variables were blending ratio, cement replacement, and curing ages. The blending ratio of 0.25, 0.50, and 0.75, cement replacement levels of 4 wt.%, 6 wt.%, and 8 wt.%, and the curing age were denoted as 3 days, 28 days, and 60 days were chosen, respectively. Face central composite factorial design comprising of 3 levels and 3 factors with Design Expert 13 where -1 denotes the low value of the independent variable (3 days, 0.25, 4 wt.%), 0 used for the medium value (28 days, 0.5, 6 wt.%) and for the high value (60 days, 0.75, 8 wt.%,) were employed to investigate the effect of the above factors on the responses. A model was fitted to the response surface generated by the experiment.

$$S_k = f(Curing\ age, Blending\ ratio, Replacement\ level) \quad (1)$$

Design Expert 13 software was employed to obtain the best-fit data and estimate the optimal conditions for mortar strength. RSM was employed in determining the optimal factors to obtain maximum strength via CCD and BBD as well and the interaction of variables was estimated.

$$S_i = \beta_o + \sum_{i=1}^{k} (\beta_i x_i) + \sum_{i=1}^{k} (\beta_{ii} A_i^2) + \sum_{i\ f=1(i \neq j)}^{k} (\beta_{ij} A_i A_j) \quad (2)$$

Where

S_i denotes the mortar strength of GP-ESP cement blends,

β_o is the coefficient constant, βi is the linear coefficient,

β_{ii} quadratic coefficient effect,

β_{ij} is the interaction coefficient effect and

A_i A_j is the coded values of variable *i j* respectively.

S_1 and S_2 denotes mortar strength from CCD and BBD respectively

A_1 as GP/GP-ESP ratio as blending ratio which is dimensionless,

A_2 as replacement level in wt.% and A_3 is curing age in days.

2.2. Mortar Compressive Strength of Cement blended with GP and ESP

The mortar compressive strength of cement blended with ESP and GP at various GP/GP-ESP ratios between 0.25 -0.75 at intervals of 0.25, cement replacements between 4-8 wt.% at various curing ages of 3, 28, and 60 days were tabulated in Table 1.

Table 1: Experimental matrix for compressive strength and various cement blends.

S. No	Blends	GP/GP-ESP	PLC %	GP %	ESP %
1	PLC		100	0	0
2	4ESP	0	96	0	4
3	3ESP1GP	0.25	96	1	3
4	2ESP2GP	0.5	96	2	2
5	1ESP3GP	0.75	96	3	1
6	4GP	1	96	4	0
7	6ESP	0	94	0	6
8	4.5ESP1.5GP	0.25	94	1.5	4.5
9	3ESP3GP	0.5	94	3	3
10	1.5ESP4.5GP	0.75	94	4.5	1.5
11	6GP	1	94	6	0
12	8ESP	0	92	0	8
13	6ESP2GP	0.25	92	2	6
14	4ESP4GP	0.5	92	4	4
15	2ESP6GP	0.75	92	6	2
16	8GP	1	92	8	0

3. Results and Discussion

3.1. Chemical Composition of the Starting Materials

The oxide composition for Portland limestone cement, eggshell and glass powders were tabulated below in Table 2

Table 2: Oxide Composition of Materials

Parameters	Portland limestone cement PLC wt.%	Eggshell Powder ESP wt.%	Glass Powder GP wt.%
SiO_2	17.5	0.52	51.9
Fe_2O_3	3.05	0.17	1.95
CaO	61.2	54.1	6.82
MgO	0.50	0.57	0.79
Al_2O_3	4.12	0.17	26.0
SO_3	1.96	0.55	11.9
K_2O	0.54	0.33	0.33
Na_2O	0.15	0.08	0.38
P_2O_5	0.20	0.34	0.00
Mn_2O_3	0.04	0.02	0.00
Cl	0.01	---	---
TiO_2	----	0.09	0.00
LOI	---	43.1	0.00
Total	89.3	100.00	100.00

Table 2 indicates the chemical composition of PLC, ESP and GP obtained via XRF spectrometer respectively. The percentage of reactive silicates content of CEM II A-L 42.5 R contains 17.54 wt.% which was lower than 51.87 wt.% for GP. Thus, GP can contribute silica required for pozzolanic reaction with the available lime to produce secondary CSH. Results indicate that GP can be considered as a pozzolan as it satisfies the requirements specified by ASTM C 618. SiO_2 + Al_2O_3 + Fe_2O_3 was greater than 50 wt.% with SO_3 content of 1.96 wt.% also satisfying the requirements which is less than the maximum of 5wt.%. GP contains 6.82 wt.% CaO which is within the range of 10-30 wt.% CaO thus considered a class C pozzolan.

The chemical composition of the eggshell (ESP) showed a high amount of CaO of 54.1 wt.% (containing $CaCO_3$ 96.55 wt.%) and other minute quantities of SiO_2, Al_2O_3, Fe_2O_3, MgO, SO_3, K_2O and Na_2O which were below 1 wt.%. ESP has a high LOI of 43.079 wt.% which can be attributed the decomposition of limestone to lime. Minor compounds such as PbO, Al_2O_3, TiO_2, ZrO_2, Cr_2O_3, Mn_2O_3, CuO, NiO are also found in glass samples under consideration, however, the amount of individual component was not more than 0.5%.

Table 3 presents the experimental results obtained from the determination of the mortar compressive strength of GP-ESP cement blend based on design of experiment via RSM for CC and BB designs. It helps to investigate its effect of blending ratio, replacement level and curing age on the mortar strength of GP-ESP cement blends respectively. The ANOVA technique was employed to undertake the statistical analysis of data.

Table 3: Experimental Design and Results for CCD (S_1) and BBD (S_2)

Curing-age days A	Blending ratio B	Replacement level wt.% C	Mortar strength N/mm^2 (S_1)	Mortar strength N/mm^2 (S_2)
28	0.75	6	32.04	
3	0.75	8	21.3	
28	0.50	6	32.9	32.9
28	0.25	6	26.05	
60	0.75	4	35.56	
28	0.50	6	32.9	32.9
60	0.25	4	29.08	
3	0.25	8	18.46	
3	0.50	6	17.17	
3	0.25	4	18.64	
60	0.75	8	40.24	
60	0.25	8	41.55	
28	0.50	4	29.56	
3	0.75	4	18.23	
28	0.50	8	29.52	
28	0.50	6	32.9	32.9
60	0.50	6	36.8	
28	0.75	4		30.92
28	0.25	8		33.22
60	0.50	8		35.74
3	0.75	6		22.04
3	0.25	6		17.17
28	0.75	8		33.52
28	0.25	4		26.8
3	0.50	8		22.62
60	0.75	6		38.20
3	0.5	4		20.12
60	0.25	6		31.16
60	0.5	4		34.27

The strength prediction equation for the determination of the mortar strength of GP-ESP- cement blends, some of the model terms were modified by selecting some model terms with probability values with 95% confidence level as well as F test of the experimental results employed to determine how statistically significant model terms are. The resultant equations were obtained from the ANOVA for strength prediction for GP-ESP cement blend using CCD and BBD models respectively:

$$S_1 = 11.07 + 0.306A + 5.44B + C \quad \ldots (3)$$

$$S_2 = -3.90 + 0.65A + 39.54B + 3.05C + 0.091AB - 0.0058AC - 1.91BC - 0.0065A^2 - 22.64B^2 - 0.0925C^2 \quad \ldots (4)$$

Equations (3) and (4) signify the quantitative effect of the independent variables such as curing age, blending ratio and replacement level (A, B, C) as well as their interactions with the response; mortar strength from CCD and BBD (S_1, S_2). The independent variables: A, B and C were inserted into the equations to determine the theoretical dependent variables; S_1 and S_2 respectively. The strength prediction employing both models significantly satisfied quadratic models based on the experimental design and factor interaction.

The analysis of variance ANOVA for strength prediction of GP-ESP-PLC from CCD and BBD are tabulated in tables 4-5. S_1 and S_2 for CC and BB designs were significant, thus there is 0.01% chance that the model has large F value of 22.33 and 28.86 respectively could occur due to noise.

Table 4: ANOVA for Mortar Strength Prediction of GP-ESP cement blends via CCD

Source	Sum of Squares	DF	Mean Square	F-Value	Prob > F	Remark
Model	825.37	3	275.12	22.33	< 0.0001	Significant
A-Curing age	766.90	1	766.90	62.25	< 0.0001	Significant
B-Blending ratio	18.47	1	18.47	1.50	0.2425	
C-Replacement	40.00	1	40.00	3.25	0.0948	
Residual	160.16	13	12.32			
Lack of Fit	160.16	11	14.56			

Table 5: ANOVA for Mortar Strength Prediction of GP-ESP cement blends via BBD

Source	Sum of Squares	DF	Mean Square	F-Value	Prob > F	Remark
Model	546.34	9	60.70	28.86	0.0009	Significant
A-Curing age	412.13	1	412.13	195.92	< 0.0001	Significant
B-Blending ratio	34.40	1	34.40	16.35	0.0099	Significant
C-Replacement	20.41	1	20.41	9.70	0.0264	Significant
AB	1.72	1	1.72	0.8158	0.4078	
AC	0.4456	1	0.4456	0.2118	0.6647	
BC	3.65	1	3.65	1.73	0.2450	
A^2	100.07	1	100.07	47.57	0.0010	Significant
B^2	7.39	1	7.39	3.51	0.1197	
C^2	0.5055	1	0.5055	0.2403	0.6447	
Residual	10.52	5	2.10			
Lack of Fit	10.52	3	3.51			

Table 6 illustrates the model fit statistics for models employed in the strength prediction of GP-ESP cement blends. The Predicted R^2 value (0.7174) and Adjusted R^2 value (0.8000) for CCD model was very close compared with BBD with Predicted R^2 value (0.6977) and Adjusted R^2 value (0.9471). CCD model was found to be the most suitable model to describe the mortar strength of the cement blends between 4 wt. – 8 wt. % replacement level. The models produced adequate precision ratios indicating a desirable signal which was greater than 4 similar to Koocheki *et al*., (2009) and Olubajo *et al.*, (2022).

It is evident in Table 6 that strength prediction via CCD and BBD produced R^2 values greater than 80%, as several researchers suggested that a fitted model with R^2 more than 80% was considered acceptable but not lower than 75% while the predicted values for the developed models should have a good correlation with the experimental data (Sarkin-Shanu *et al*., 2024; Olubajo *et al*., 2022; Sani & Adetoye 2024). The results indicated that CCD is the most suitable to predict the strength for Portland limestone cement blended with GP and ESP. The regression values and adjusted regression values for CCD and BBD were 0.7174 and 0.8000, 0.6977 and 0.9471, respectively, demonstrating the developed model's appropriateness to predict the mortar compressive strength for GP-ESP cement blends by their R^2 and R^2_{adj} value near 1.

Table 6: Fit Statistics for CCD and BBD

Source	CCD	BBD
Sum of Squares	825.37	546.34
DF	3	9
Mean square	275.12	60.70
F value	22.33	28.86
Prob> F	<0.0001	0.0009
Std. Dev.	3.51	1.45
R^2	0.8375	0.9811
Adj. R^2	0.8000	0.9471
Pred. R^2	0.7174	0.6977
PRESS	278.48	168.33

3.2. Contour and Three-Dimensional Surface Graphs

The 3D surface graphs represent the relationship between the response, i.e., mortar strength of GP-ESP cement blend, and factors such as blending ratio, replacement level, and curing age. The response surface curves demonstrate the interactions between the various factors and determine the optimum for each factor to obtain maximum response. According to Dockery (2017), the parabolic nature of contours indicates that the interaction between both factors is significant. From the regression equation, the most significant parameters influencing the prediction of the mortar strength of GP-ESP cement blends in the order are curing age A, replacement level C, and blending ratio B, respectively. Experiments were conducted by varying the parameters using experimental design to investigate the interaction between the factors. The experimental results of the central composite design and Box-Behnken design were fitted into equations 3 and 4, respectively. According to ANOVA, the curing age and cement replacement are the most significant variable, with the effect of curing age (62.25) and the effect of cement replacement (3.36) with blending ratio (1.51), which had the least significant effect on the mortar compressive strength of GP-ESP cement blend.

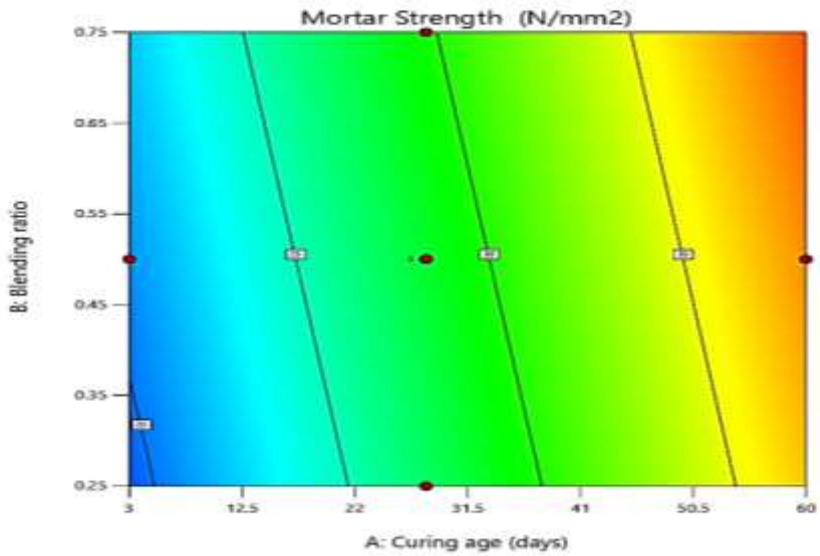

Figure 2: Contour plot for interaction on mortar strength of GP-ESP-cement blends between blending ratio and curing age via CCD.

Figure 3 depicts the response surface curves illustrating the interactive effects of the variables for CCD, while Figures 4 to 6 indicate response plots for the interactive factors and the mortar strength for BBD. Mortar strength increased as the cement replacement, blending ratio, and curing age were increased simultaneously. It could be observed that as the blending ratio and cement replacement were increased simultaneously at constant curing ages of 3, 28, and 60 days, the mortar strength increased, respectively. The most significant factor is that the curing age of the cement blends has a positive effect, which could be related to the hydration reactions coupled with pozzolanic reactions stemming from silica present in GP and excess lime present. Thus, the production of more CSH as the curing days progressed. The trend agrees with the fact that the time of hydration determines the rate of hydration of cement (Jarumi *et al.*, 2022; Olubajo *et al*., 2019 and Olubajo *et al*., 2020).

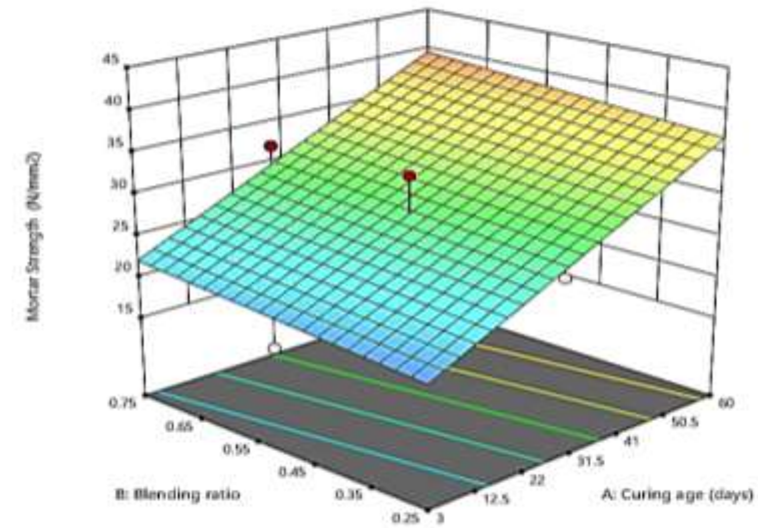

Figure 3: 3-D of GP-ESP-cement blends between blending ratio and curing age

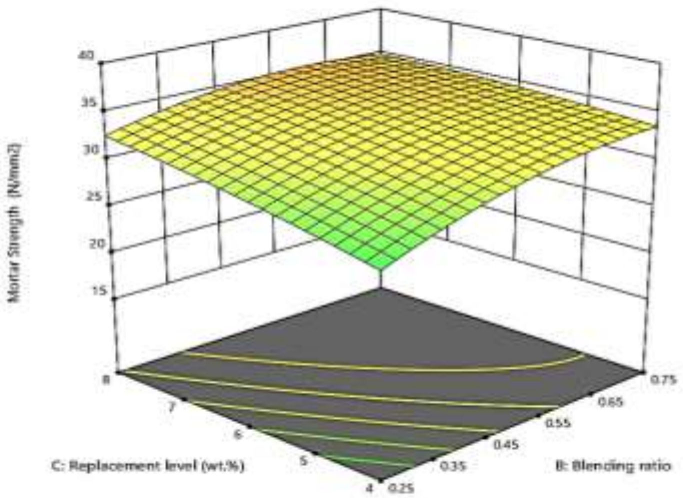

Figure 4: Response surface for interaction on mortar strength of GPESP-cement blends between curing age and replacement via BBD

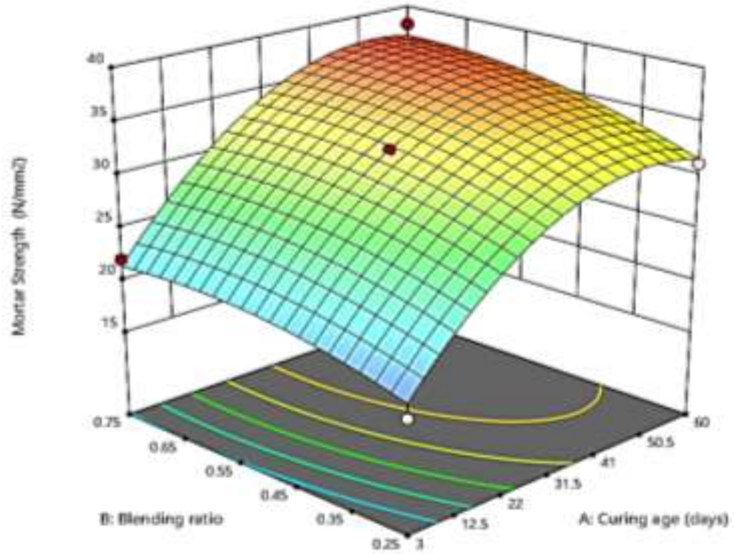

Figure 5: Response surface for interaction on mortar strength of GPESP-cement blends between blending ratio and curing age via BBD

Figure 4 indicated an increase in the mortar compressive strength as the curing age was constant for 3, 28, and 60 days when blending ratio and cement replacement were increased simultaneously. Figure 5 shows that the mortar strength increased when the blending ratio and curing ages are increased simultaneously at constant replacement levels from 4 -8 wt.%, respectively. Figure 6 shows that the mortar strength increased when the cement replacement and curing ages are increased simultaneously at the constant blending ratio between 0.25 -0.75, respectively.

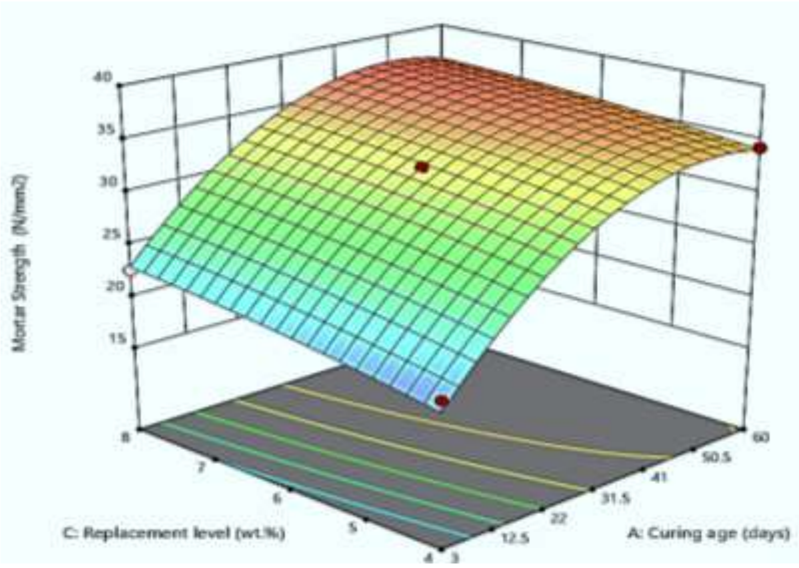

Figure 6: Response surface for interaction on mortar strength of GPESP-cement blends between curing age and cement replacement via BBD

3.3. Optimization of Mortar compressive strength of cement blends

Optimization of the mortar strength of GP-ESP-cement blends was conducted and the optimal conditions were a blending ratio of 0.75, replacement level of 8.00 wt.%, and curing age of approximately 60 days with a mortar compressive strength of 41.53 N/mm^2 with desirability of 0.999 for CCD whereas BBD optimal conditions with blending ratio of 0.64 replacement level of 8.00 wt.%, curing age of 50.84 days and mortar compressive strength of 37.73 N/mm^2 and desirability of 1.00. Figure 7 depicts graphs of the effect of the interactions of the various factors as a function of mortar strength.

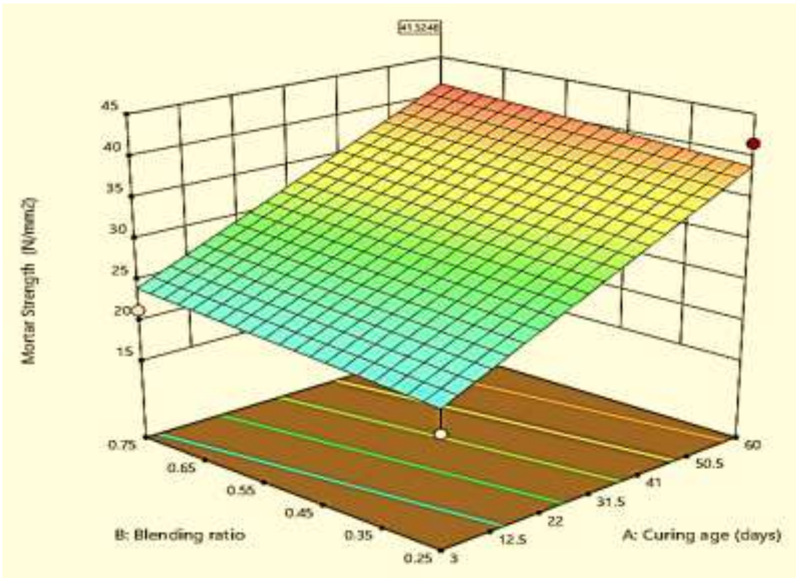

Figure 7: Response surface indicating the optimal conditions for interaction on mortar strength of GP-ESP-cement blends between curing age and blending ratio via. CCD

4. Conclusion

This work investigated the factors influencing the mortar strength of GP-ESP-PLC conducted under laboratory conditions. The use of response surface methodology via CCD was found to be most effective in the determination or prediction of the mortar strength of ternary cement blends within blending ratio 0.25 -0.75, replacement level 4 – 8 wt. %, and curing age between 3 and 60 days. The process optimization was carried out using response surface methodology, and the model equations for the strength prediction were obtained from 17 and 15 runs for both CCD & BBDs, respectively, with CCD being the most reliable. The optimum blending ratio is 0.75, the replacement level is 8.000 wt.%, and the curing age is approximately 60 days with a mortar compressive strength of 41.53N/mm^2. Amongst these factors, curing age significantly influences strength gain due to an increase in the hydration reaction time during curing, which is from 3 to 60 days. The model equation developed via CCD can adequately predict the mortar strength of ternary cement blended with GP and ESP between replacement levels of 4 -8 wt. %.

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