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Optimization of Mortar Compressive Strength of Orange Peel Ash - Eggshell Powder Cement Blends

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Abstract

This study explores the effect of replacing eggshell powder (ESP) in a cement blend with orange peel ash (OPA) on mortar compressive strength. OPA was produced by calcining orange peels at 650°C for 2 hours, followed by sieving through a 90µm mesh. Portland limestone cement (CEM II 42.5R A-L) was blended with ESP and partially replaced with OPA at 0-6 wt.% in 2 wt.% increments. The mortar was prepared using a 1:3:5 ratio (water, binder, sand) and tested using 50 mm cubes. Chemical analysis revealed that OPA primarily contained lime, silica, and potassium oxide, with a combined $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ content of 19.77 wt.%, classifying it as non-pozzolanic per ASTM C618-19. ESP, on the other hand, acted as a filler with high lime content (54.09 wt.%) with a CaCO_3 composition of 96.55 wt.%. The effect of OPA and ESP on mortar strength was assessed based on blending ratio, replacement level, and curing age, with optimization performed using Response Surface Methodology (RSM). Central Composite Design (CCD) and Box-Behnken Design (BBD) were employed for predictive modeling and compared for accuracy. Results indicated that increasing curing age (7-60 days) and either blending ratio or replacement level (0.25-0.75, 2-6 wt.%) led to improved mortar strength. Analysis of variance (ANOVA) confirmed that curing age had a significant effect, with high F-values in both CCD and BBD models. CCD provided the best predictive accuracy, with a predicted R^2 (R^2_{pred}) of 0.8464, making it the most suitable model for describing the mortar compressive strength of these ternary cement blends. Optimization revealed that the ideal conditions for CCD were 0.75 blending ratio, 2.0 wt.% replacement, and 60 days curing period, achieving a mortar compressive strength of 42.17 N/mm² with a desirability of 1.0. For BBD, the optimal conditions were a 0.25 blending ratio, 2.0 wt.% replacement, and a 60-day curing period, yielding 43.86 N/mm² with a desirability of 0.885. The interactive effects of blending ratio, replacement level, and curing age were also analyzed, confirming their influence on mortar strength.

Keywords

Mortar Compressive Strength, Optimization, Orange Peel ash, Eggshell powder, Cement blends

INTRODUCTION

As economy grows, societies develop and get wealthier, resulting in an increase in the demand for construction materials which include concrete and mortar. Mortar, a composite material consisting of cement, water, fine aggregate, is the most widely produced and consumed material for construction.

The main ingredient required in the production of mortar is Portland cement which is a material made by heating a mixture of limestone and clay or shale in a kiln at about 1400-1450°C to form clinker, then grinded to a fine powder with a small addition of gypsum. However, the method of producing Portland cement is costly, resource and energy intensive. Moreover, the production of cement is associated with 8 % of carbon dioxide (CO_2) emission which in turn contribute to the global climate changes (Rodgers, 2018).

According to World Bank (2018) waste generation will increase from 2.01 billion tons in 2016 to 3.04 billion tons globally in 2050 in which at least 33% of this waste is mismanaged through open dumping or burning. In order to free the environment and the society of the dangers associated with this accumulated waste, there is need for proper utilization of this waste which have being issues of serious concern to most countries, especially developing countries like Nigeria.

Agricultural wastes generated from households and companies/industries which pollute the environment, if not properly taken care of. The increasing demand for cement can address the problem of dumping waste and drive sustainability via the proper utilization or recycling of these biogenic wastes such as eggshell powder. Eggshell powder is byproducts of agricultural wastes obtained from egg. Eggshell is composed of mostly 93.7 % of calcium carbonates and other constituents like phosphates and magnesium which is similar to limestone, thus can be employed as a partial cement replacement material (Okonkwo et al., 2012). Recently, several research attempts are focused on incorporating agricultural and industrial waste materials such as orange peels into concrete mixes to decrease the use of cement materials (Asman et al., 2017).

Orange is the most widely cultivated fruit worldwide. The global orange production for 2021-22 is estimated at 49 million metric tons (Source: US Department of Agricultural service) which accounts for about 50-60% of total citrus production. About 50-60% of these were consumed as fresh fruit, and the remaining 40-50% was destined for industrial processing. An enormous amount of waste results from this production; considering that the 50% is composed of peels (FAO, 2017).

In a bid to overcome the challenge of cumbersome and time-wasting technique of strength determination of cement blends. Several avenues are being exploited in prediction of the mortar compressive strength of ternary blended cement (Olubajo et al. 2022, Jarumi et al. 2023, Olubajo and Stephen, 2024, Tumba et al. (2025), Jessica et al. (2024) and Jibrin et al. (2025). It can be summarized that there is limited work done using RSM for blended cement mortars particularly replacement level with orange peel ash and eggshell powder respectively. Compared to the conventional experimental works, the Response Surface Methodology (RSM) is a numerical computation modeling application that is also a subset of Design of Experiment (DOE). It is a popular tool and is one of the most widely used DOE methodologies compared to other methods like Taguchi, mixture, factorial and screening. Furthermore, RSM based model has various advantages such as a superior approach in anticipating the responses, the ability to build an accurate model with a small quantity of experimental data, reducing the number of trials required, evaluating the correlations between factors and comprehending the interaction effect of independent and dependent variables.

Several research works have been conducted on the use of agricultural wastes as cement replacement material

with various successes. Some of the notable works are as follows; Olubajo et al. (2019) investigated the effect of replacing cement with orange peel ash between 2.5-10 % at 2.5 % interval. The OPA was prepared by calcining orange peel at various temperatures between (600°C, 700°C and 800°C) and time (1 hour and 2 hours) from which the optimum condition was obtained at 600°C for 2 hours. They observed that as the OPA content was increased from 2.5-10% resulted in a reduction in the compressive strength with a significant increase in the water required for consistency from 29.8% to 42.4%.

The initial and final setting time increased by 20.64% and 47.37% respectively. It was suggested that cement replacement with OPA should not exceed 5 wt.% due to the fact that the physico-mechanical properties were adversely affected. Ofuyatan et al. (2018) conduct an experiment to investigate the effect of partial replacement of ordinary Portland cement with cassava peel ash between 5-25% at 5% interval which was prepared by calcining cassava peel at 700°C and observed that the density of the blended cement with CPA did not depend on the percentage replacement level while the compressive and tensile strength decrease with increase in CPA replacement. Similarly, an increase in strength was experienced as the curing progressed with

the optimal cement replacement with CPA was at 10%. Parthasarathi et al. (2017) investigated the use of eggshell powder and silica fume as cement replacement materials. Eggshell powder was replaced by 5%, 10%, and 15% while silica fume was replaced by 2.5%, 5%, and 7.5% of the weight of cement. An increase in the split tensile, compressive and flexural strengths by inclusion of ESP content and silica fume.

Jhatial et al. (2019) experimentally replaced cement between ESP 5-15% ESP at 5% interval by weight of cement in M40 grade concrete. Ground ESP with fineness of 50 and 100 μ m were considered as supplementary cementitious materials. Cement samples partially replaced with ESP produced enhanced strength compared with control sample. At 10% ESP replacement, the highest compressive strength was achieved. It was also observed, that the fineness of ESP also influenced the compressive strength with ESP of 50 μ m, resulted in improved strength with the optimum ESP determined at 10% for both fineness.

Genera (2015) work indicated that the optimum replacement ratio for eggshell powder was 10% with strength of cement blend equivalent with control and concluded that when replacement was increased up to 20%, problems of bleeding, micro cracks and segregation occur.

Yu et al. (2018) investigated the use of ESP content from 5 - 20% replacement at 5% interval by volume of cement in concrete at various curing conditions. Eggshell concrete greatly increased the compressive and flexural strength of concrete by up to 51.1 and 57.8 %, respectively. However, because the eggshell includes a large amount of calcium, which reacts rapidly with acid and alkali liquids, the compressive strength of the eggshell concrete decrease gradually as the amount of eggshell powder increases during immersion in alkali and acid liquids. The reduction of compressive strength during immersion in sulphur and sodium sulfate solutions was 27.5% and 31.2%, respectively, when 20% of eggshell powder was used to replace cement. It was concluded that the optimum percentage of oven-dried eggshell powder as a partial cement replacement is 15%.

Amarnath, (2014) developed different ESP concrete were by replacing 5-15% ESP for cement with results covering strength development and transport properties indicating ESP as partial replacement for cement in concrete. Concrete compressive strengths were higher than control for 5% replacement at 7 and 28 days. However, replacement beyond 10% had lower strength than control concrete. Sharma and Kaushik, (2013) studied strength prediction of self-compacting mortar using RSM to create regression models

and optimize the compressive strength and considered factors like the water-cement ratio, sand-cement ratio, and super plasticizer dosage. The results showcased the usefulness of RSM in predicting and optimizing the strength of self-compacting mortar.

Agarwal *et al.* (2014) employed RSM to develop prediction models for optimizing the compressive strength of cement mortar. Various factors like cement content, sand content, water-cement ratio, and curing time were considered. The models derived from RSM were validated, and the optimized values were determined for achieving the desired strength. Aslani *et al.* (2016) investigated the use of RSM in predicting and optimizing the compressive strength of cement mortar containing ground granulated blast-furnace slag (GGBS).

The researchers considered variables such as GGBS content, water-binder ratio, and curing time. RSM was utilized to develop mathematical models that accurately predicted the strength based on the chosen variables. The optimization process led to the identification of the optimal values for each factor to achieve the desired strength performance. Sisomphon *et al.* (2012) focused on optimizing the compressive strength of cement mortar with the incorporation of nano-alumina and calcium carbonate. RSM

was employed to study factors such as nano-alumina content, calcium carbonate content, and curing time. The results demonstrated that RSM is an effective tool for determining the optimal composition of cement mortar to enhance its strength properties. Olubajo *et al.* (2017), Olubajo *et al.* 2022, Jarumi *et al.* 2023, Olubajo and Stephen, 2024, Tumba *et al.* (2025), Jessica *et al.* (2024) and Jibrin *et al.* (2025) investigated the use of RSM in predicting and optimizing the mortar strength of Portland limestone cement blended with coal bottom ash and limestone; saw dust ash and eggshell powder and date palm seed ash and eggshell powder; animal bone ash and metakaolin; velvet tamarind pod ash and eggshell powder respectively. These studies highlight the successful application of response surface methodology (RSM) in predicting and optimizing the compressive strength of cement mortars by considering various factors and utilizing RSM techniques, researchers were able to develop mathematical models that accurately predicted the strength and identify optimal combinations of variables to achieve the desired results. RSM proves to be a valuable approach for optimizing cement mortar composition and improving its performance in terms of strength.

Materials and Method

Materials

The orange peels employed in this research were sourced from Kaduna metropolis and sun dried for several days to obtain uniform weight. The orange peels were sorted to remove unwanted materials and then calcined at 600°C for 2 hours to obtain orange peel ash. The resultant ash was first cooled and then ground and sieved with 90µm sieve. Portland limestone

cement CEM II 42.5R was obtained from Dangote supplier and employed for this research while distilled water was used for the physico-mechanical properties of the cement blended with orange peel ash and eggshell powder. The standard sand was obtained in Bauchi by sieving sand into various proportions according to Indian standards which was used as aggregates in the production of mortars.



Plate 1: Ground Orange peels Plate 2: Orange peel ash

Plate 3: Eggshell powder

Experimental Design

The mortar compressive strength of cement blended with ESP and OPA at various OPA/OPA-ESP ratios between 0.25 - 0.75 at interval of 0.25, replacement levels between 2 - 6 wt.% at various curing ages of 7, 28 and 60 days were tabulated in Table 1. The design summary for the dependent variables are the mortar strengths via CCD and BBD models with blending ratio, replacement level and curing age as independent variables. The independent variables were chosen as blending ratio's lower, middle and upper limit denoted as 0.25, 0.50,

0.75; replacement level lower, middle and upper limits denoted by 2 wt.%, 4 wt.%, 6 wt.%; curing age lower, middle and upper limits are denoted as 7 days, 28 days, 60 days respectively. Face central composite factorial design comprising of 3 levels and 3 factors with Design Expert 13 where -1 denotes low value of the independent variable (0.25, 2 wt. %, 7 days), 0 used for the middle value (0.5, 4 wt. %, 28 days) and for the high value (0.75, 6 wt. %, 60 days) were employed to investigate the effect of the above independent variables on the dependent variables. A model was fitted to

the response surface generated by the experiment.

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

the interaction of variables was estimated. 14 runs each were carried out to fit the general model equation (1) for CCD and BBD respectively and to obtain optimum condition for blending ratio, curing age, replacement level for the optimum mortar strength of the cement blend.

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

$$S_i = \beta_0 + \sum_{i=1}^k (\beta_i x_i) + \sum_{i=1}^k (\beta_{ii} A_i^2) + \sum_{i \neq j=1}^k (\beta_{ij} A_i A_j) \quad (2)$$

Where S_i denotes the mortar strength of OPA-ESP cement blends, β_0 is the coefficient constant, β_i 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 ij respectively in equation (2).

S_{CCD} and S_{BBD} denote mortar strength from CCD and BBD respectively while A as curing age

in days, B as OPA/OPA-ESP ratio as blending ratio which is dimensionless and C is replacement level in wt.%. The blended cement mortars were prepared by mixing OPA, ESP, PLC, standard sand along with distilled water. The experimental mix design comprising of PLC, OPA, ESP, standard sand with the proportions of sand, cement, calcined samples and water were tabulated in Table 1.

Table 1: Experimental Design of various cement blended at various proportions of OPA and ESP.

S/No	Blends	OPA/OPA-ESP	PLC wt. %	OPA wt. %	ESP wt. %
1	PLC	-	100	0	0
2	1.5ESP0.5OPA	0.25	98	0.5	1.5
3	1ESP1OPA	0.5	98	1	1
4	0.5ESP1.5OPA	0.75	98	1.5	0.5
5	3ESP1OPA	0.25	96	1	3
6	2ESP2OPA	0.5	96	2	2
7	1ESP3OPA	0.75	96	3	1
8	6ESP	0	94	0	6
9	4.5ESP1.5OPA	0.25	94	1.5	4.5
10	3ESP3OPA	0.5	94	3	3
11	4.5ESP1.5OPA	0.75	94	4.5	1.5

Characterization of OPA and ESP

The chemical compositions of OPA and ESP were determined using the X-ray fluorescence spectrometer.

RESULT AND DISCUSSION

Figure 1 indicates the spectra generated for TGA/DTA by heating from 30°C - 900°C at 10°C/min. It can be identified on the DTA curve by U-shaped depression in the curve which symbolizes rapid loss of weight followed by rapid gain in the

weight. The thermogravimetric analysis of orange peel represented by the red line reveals a several stages of decomposition process, where between a temperature range of 140 -380°C, a bulk of the weight loss occurs indicating the active pyrolysis stages of orange peel with distinct phases related to the evaporation of moisture, degradation of volatile components and lastly the breakdown of the cellulose and lignin structures with the peel material.

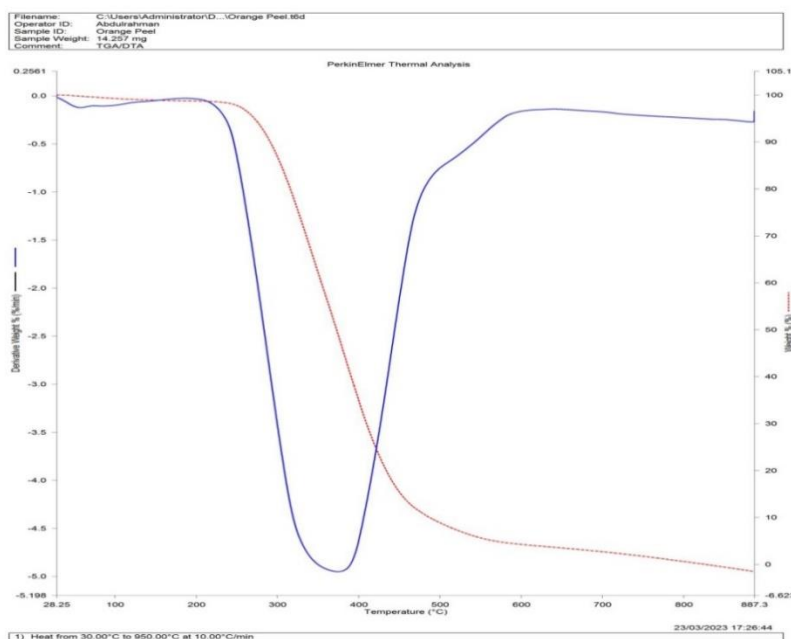


Figure 1: Thermogravimetric / Differential Thermal Analysis for Orange peel

The chemical compositions of OPA, ESP and PLC were presented in Table 2. The PLC is composed of Lime (CaO) content (61.17 wt.%), Silica (SiO₂) (17.55 wt.%) with small Iron Oxide (Fe₂O₃) content (3.05 wt.%), Alumina (Al₂O₃) content (4.12 wt. %), Magnesium oxide (MgO) content (0.50 wt.%), Sulphur trioxide (SO₃)

content (1.96 wt.%) Sodium oxide (Na₂O) content (0.15 wt.%), Potassium oxide (K₂O) content (0.54 wt.%). According to Ahmed et al (2016) and Ahmed (2019) reported that orange peel waste mainly composed of sugars, pectin, cellulose and hemicellulose along with a low content of lignin.

Chemical analysis revealed that the cement employed was classified as CEM II A-L 42.5R composed of four major oxides greater than 85 wt. % with Fe_2O_3 , MgO , Al_2O_3 , SO_3 , K_2O except CaO and SiO_2 which fell within the approximate amount for Portland limestone cement. The cement employed is a type II- A-L cement indicating that ordinary Portland cement is blended with 5-10% limestone with OPA's chemical analysis revealed that orange

peel calcined at 650 °C for 2 hours contained less than 70 % of $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ (19.77 wt.%) while CaO content was 13.74 wt.% which met class C with CaO content > 10 wt.%. Hence the ash does not satisfy as a pozzolan according to (ASTM C618-19). The OPA had a loss on ignition of 15.81 % which indicates a high degree of unburnt carbon of ash (Külaots et al., 2004; Olubajo et al., 2019).

Table 2: Chemical Analyses of PLC, OPA & ESP

Parameters	PLC wt. %	ESP wt. %	OPA wt. %
SiO_2	17.55	0.52	15.64
Fe_2O_3	3.05	0.17	1.34
CaO	61.17	54.10	13.74
MgO	0.50	0.57	4.57
Al_2O_3	4.12	0.17	2.79
SO_3	1.96	0.55	1.23
K_2O	0.54	0.33	42.02
Na_2O	0.15	0.08	0.19
P_2O_5	0.2	0.34	2.49
Mn_2O_3	0.036	0.02	0.03
Cl	0.01		0.00
TiO_2		0.09	0.15
Cr_2O_3			0.02
LOI		43.08	15.81
Total	89.28	100.00	100.00
CaCO_3		96.55	24.52

Table 3 presents the experimental results obtained from the determination of the mortar compressive strength of OPA-ESP cement blends based on design of experiment via RSM for CC and BB designs

to investigate its effect of blending ratio, replacement level and curing age on the mortar strength of OPA-ESP cement blends respectively.

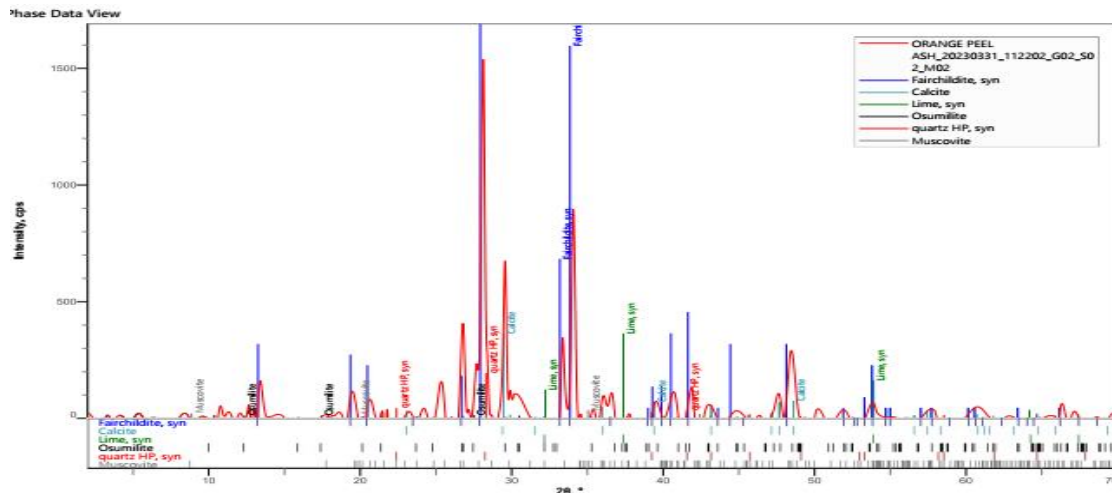


Figure 2: X-ray pattern for orange peel ash

Figure 2 depicts the X-ray pattern of OPA primarily reveals the presence of crystalline minerals like CaCO_3 represented by Calcite and Lime as the dominant peak at (CaCO_3 $2\theta = 36.2^\circ, 29.8^\circ$) which was close to works by Kaknics et al. (2015) and Ahmed (2019) at (CaCO_3 $2\theta = 33.8^\circ$) along with other smaller peaks potentially indicating inorganic components such as potassium, magnesium and silica/quartz (SiO_2 at $2\theta = 28.5^\circ, 29.9^\circ, 33^\circ, 33.6^\circ, 48^\circ$) while Mishra and Das (2010) indicated (SiO_2 at $2\theta = 26.2^\circ$) owing to the growing condition and the processing of the orange peel. The dominant peaks in the XRD pattern corresponds to the crystalline calcium carbonate due to the high calcium content in citrus fruits. Minor peaks correspond to potassium, magnesium and silica which varies with the soil composition in which the oranges were grown. Other components include Fairchildite $\text{K}_2\text{Ca}(\text{CO}_3)_2$, Osumilite (a rare potassium,

sodium iron aluminum silicate mineral and it is part of milarite groups of cyclosilicates $(\text{K.Na}) (\text{Fe.Mg}) (\text{Al.Fe})_3 \text{Si}_2\text{Al}_2\text{O}_{10}$ and muscovite. Results indicated that the XRF analysis of OPA seemed to tally with the components revealed from XRD pattern.

Mortar Compressive Strength of OPA-ESP cement blends

The mortar compressive strength for 7, 28 and 60 days for PLC blended with OPA and ESP between 2-6 wt.% at interval of 2 wt.% and blending ratio between 0.25 - 0.75 at interval of 0.25 were determined and tabulated in Table 3. An increase in the mortar strengths of PLC blended with 2 wt.% replacement level with OPA and ESP for various blending ratios was experienced as the curing age progressed which is agreement with principle of cement hydration and works by Olubajo and Osha (2013), Olubajo et al. (2020), Olubajo et al. (2020b), Kaya (2011), De

Weerdt et al. (2011). All cement blends produced lower strengths compared to control except for PLC-ESP with blending ratio of 0 (PLC blended with 2 wt.% ESP. it could be seen from Figure 3 that the strength of the cement blended with 2 wt.% ESP was enhanced by 9.39%, 3.68% and 2.20% compared with control for 7, 28 and 60 days respectively. It was also observed that for other blending ratios,

the strength produced by the various blends were lower than the control. This lower strength produced by various cement blends of blending ratio beyond 0 could be attributed to clinker diminution. Whereas the enhancement in strength could be linked with pozzolanic reaction despite reduction in C/S ratio which is agrees with Olubajo and Osha (2013).

Table 3: Mortar Compressive Strength of various OPA-ESP-cement blends

S/No	Cement blends	Blending ratio	Replacement level wt. %	7 days	28 days	60 days
1	PLC	-	0	36.05	45.75	53.29
2	2ESP	0	2	39.79	47.50	54.49
3	1.5ESP0.5OPA	0.25	2	26.3	35.27	41.12
4	1ESP1OPA	0.5	2	26.45	32.76	36.96
5	0.5ESP1.5OPA	0.75	2	30.01	33.77	41.42
6	2OPA	1	2	22.73	30.37	36.88
7	4ESP	0	4	32.4	38.52	48.58
8	3ESP1OPA	0.25	4	22.72	28.46	46.92
9	2ESP2OPA	0.5	4	20.44	27.01	35.62
10	1ESP3OPA	0.75	4	21.61	29.28	43.48
11	4OPA	1	4	18.23	21.90	36.64
12	6ESP	0	6	30.97	37.45	49.72
13	4.5ESP1.5OPA	0.25	6	27.02	34.12	41.28
14	3ESP3OPA	0.5	6	20.2	24.75	27.20
15	1.5ESP4.5OPA	0.75	6	22.47	26.14	33.52
16	6OPA	1	6	22.76	27.26	39.80

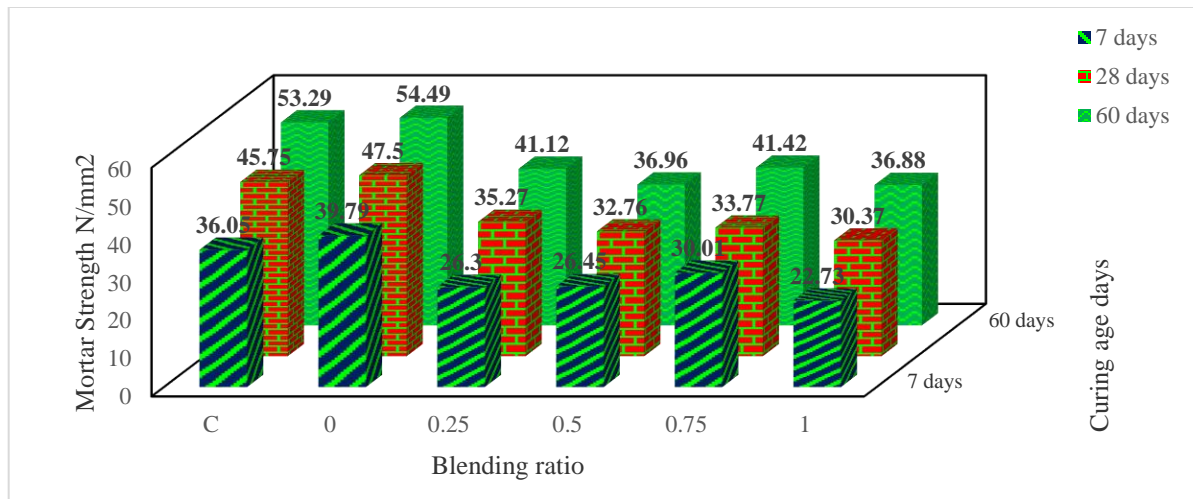


Figure 3: Effect of factors on OPA-ESP cement blends on the mortar compressive strength for 2 wt.%

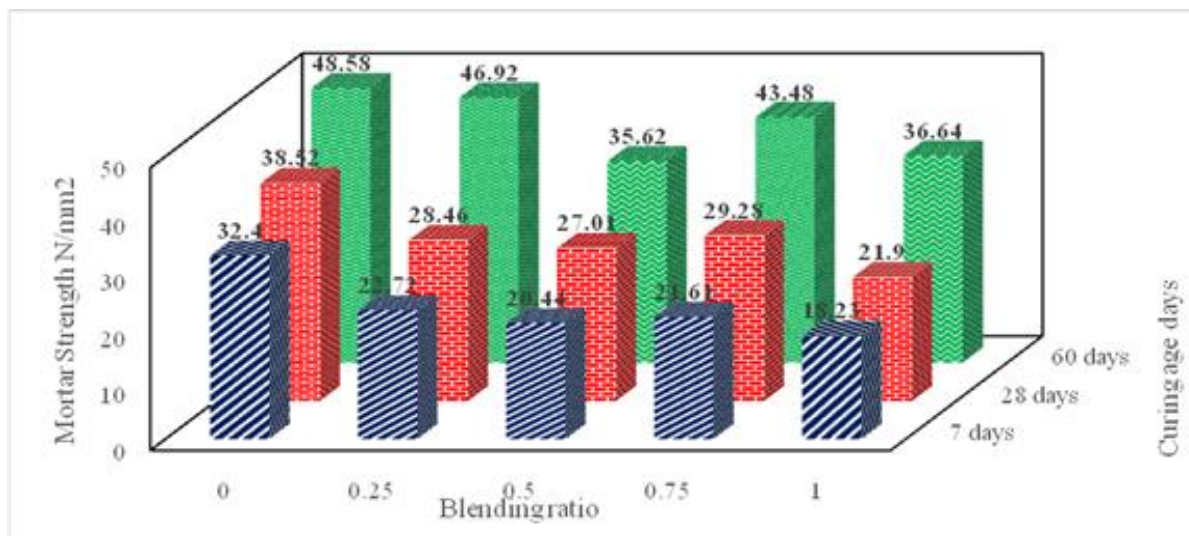


Figure 4: Effect of factors on OPA-ESP cement blends on the mortar compressive strength for 4 wt.%

Figures 3 and 4 indicate the effect of the factors on OPA-ESP cement blends on the mortar compressive strength. (0.25, 4wt.%, 60 days) produced strength/strength index of 46.92 N/mm² (88.05%); (0.75, 4 wt.%, 60 days) produced strength of 43.48 N/mm² (81.59%); (0, 28 days) produced strength of 38.52 N/mm² (84.20%); (0.25, 4 wt.%, 7 days) produced

strength of 41.29 N/mm² (77.48%) for 4 wt.% replacement level respectively. It could be observed from both Figures 4 and 5 that as the blending ratio were increased at a given curing age produced lower strength compared to control. For 4 wt.% the following produced enhanced strength despite clinker diminution which include (0, 4 wt.%, 60 days) produced

strength/strength index of 48.58 N/mm² (91.16%); (0.25, 4 wt.%, 60 days) produced strength of 46.92 N/mm². Whereas for 6 wt.% include (0,7 days) produced strength of 30.97 N/mm² (85.91%); (0, 28 days) produced strength of 37.45 N/mm² (81.86%);(0, 60 days) produced strength

of 49.72 N/mm² (93.30%);(0.25, 28 days) produced strength of 34.12 N/mm² (74.58%); (0.25, 60 days) produced strength of 41.28 N/mm² (77.46%) and (1, 60 days) produced strength of 39.0 N/mm² (73.18%).

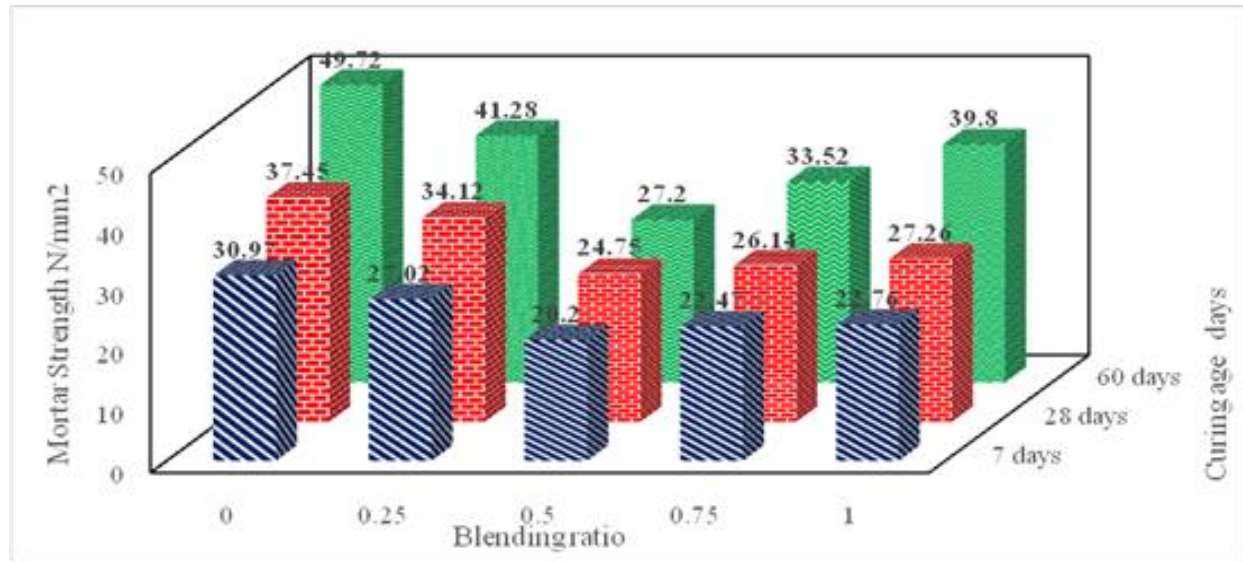


Figure 5: Effect of factors on OPA-ESP cement blends on the mortar compressive strength for 6 wt.%

It could also be observed that at replacement level above 2 wt.% at various blending ratios, a number of cement blends produced enhanced strength despite clinker diminution which could be attributed to pozzolanic activity suggested by Olubajo and Osha (2013). The optimal OPA content to obtain strength slightly above the control was 2 wt.% beyond which produced lower strengths. This trend of enhanced strength could be attributed to the provision of nucleation sites due to the

inclusion of eggshell powder into the cement matrix compared to control which agreed with Iliya et al. (2023).

Optimization of mortar strength of OPA-ESP cement blends

The significance of the prediction model was to determine the mortar strength of OPA-ESP- cement blends. Several model terms were modified by selecting several model terms with probability values of 95% confidence level to determine that the model terms were statistically significant. The individual and interactive

effects of the factors were described by graphs for 3D response surfaces and contours. Application of Design Expert 13 in which the results obtained from experimental run were inserted and employed to conduct investigation of

several factors on the response via equation. ANOVA was employed as well to produce the resultant equations for predicting the strength of OPA-ESP cement blends using CCD and BBD models respectively:

$$S_{CCD} = +33.91664 + 0.306930A - 20.06293B - 3.55560C - 0.132831AB - 0.000455AC - 4.08000BC + 0.000195A^2 + 37.69465B^2 + 0.56022C^2 \quad (3)$$

$$S_{BBD} = +30.93889 + 0.296318A - 7.02500BC \quad (4)$$

Table 4: ANOVA for Response Surface Quadratic Model Analysis of Variance for Mortar Strength Prediction of OPA-ESP cement blend via CC and BB designs

Source	Sum of Squares	DF	Mean Square	F Value	Prob > F	
Model	646.10	9	71.79	38.75	< 0.0001	Significant
A-Curing age	445.16	1	445.16	240.29	< 0.0001	Significant
B-Blending ratio	6.14	1	6.14	3.32	0.1114	
C-Replacement level	50.88	1	50.88	27.46	0.0012	Significant
AB	6.25	1	6.25	3.37	0.1089	
AC	0.0047	1	0.0047	0.0025	0.9613	
BC	33.29	1	33.29	17.97	0.0038	Significant
A ²	0.0456	1	0.0456	0.0246	0.8798	
B ²	14.87	1	14.87	8.03	0.0253	Significant
C ²	13.45	1	13.45	7.26	0.0309	Significant
Residual	12.97	7	1.85			
Lack of Fit	12.97	5	2.59			
Model	604.82	3	201.61	8.87	0.0028	Significant
A-Curing age	503.20	1	503.20	22.14	0.0006	Significant
B-Blending ratio	24.68	1	24.68	1.09	0.3198	
C-Replacement level	76.94	1	76.94	3.38	0.0929	
Residual	250.05	11	22.73			
Lack of Fit	250.05	9	27.78			

The quantitative effects of the independent variables such as curing age, blending ratio, replacement level and (A, B, C) as well as their interactions with the response: mortar strength from CCD and

BBD (S_{CCD} , S_{BBD}) are represented by Equations (3) and (4). The independent variables: A, B, and C were placed into the equations to evaluate the theoretical responses; S_{CCD} and S_{BBD} respectively.

Based on the experimental design and factor interaction, the prediction of strength via CCD and BBD models significantly satisfied quadratic and linear models for CCD and BBD respectively. Table 4 illustrates the analysis of variance (ANOVA) for predicting the strength of OPA-ESP-PLC via CCD and BBD represented by S_{CCD} and S_{BBD} for CCD and BBD designs were significant, thus there is 0.01% chance that the model has large F value of 38.75 and 8.87 respectively could occur due to noise.

The model fit statistics for CCD and BBD models for the prediction of strength of OPA-ESP cement blends are tabulated in Table 5. CCD model produced Predicted R^2 value of 0.8464 and Adjusted R^2 value of

0.9550 which were closer compared to BBD model which produced Predicted R^2 value of 0.3650 and Adjusted R^2 value of 0.6277 respectively. CCD model was seen to be the most suitable model to determine the mortar strength for the cement blends between 2 - 6 wt. % replacement levels. Whereas, both models produced adequate precision ratios which indicated desirable signal greater than 4 which was in agreement with Olubajo et al. (2022) Koocheki, (2004) and Olubajo (2017). The evaluation of the model fit for CCD model was confirmed by the coefficient of determination R^2 of 0.9803. The result indicated CCD model can be considered to be statistically significant according to the F test with 95% confidence and F value of 38.75.

Table 5: Model Fit Statistics for CCD & BBD for strength prediction of OPA-ESP cement blends

Source	<u>CCD</u>	BBD
Sum of Squares	<u>646.10</u>	604.82
DF	<u>9</u>	3
Mean square	<u>71.79</u>	201.61
F value	<u>38.75</u>	8.87
Prob. > F	<u>< 0.0001</u>	0.0028
Std. Dev.	<u>1.36</u>	4.77
R^2	<u>0.9803</u>	0.7075
Adj. R^2	<u>0.9550</u>	0.6277
Pred. R^2	<u>0.8464</u>	0.3650
PRESS	<u>101.26</u>	542.85

The strength prediction for the OPA-ESP cement blend via CCD produced R^2 values

greater than 80% whereas BBD model was less than 80% as illustrated in Table 5.

Olubajo et al. (2019), Olubajo et al. (2022), Koocheki et al. (2009) and Chauhan & Gupta, (2004) suggested that a fitted model with above R^2 of 80% and $> 75\%$ can be considered acceptable and the predicted values for the developed models should have a good relationship with the experimental data. Amongst the models, CCD model best suited to predict the strength for OPA-ESP cement blend. The regression and adjusted regression values for CCD model were 0.9803 and 0.9550 respectively demonstrating that the developed model's appropriateness to predict the mortar compressive strength for OPA-ESP cement blends by its R^2 and R^2_{adj} value near 1.

According to Zaibunnisa et al. (2009) and Arsenovic et al. (2012), they suggested that the most reliable empirical equation which fit the experimental data which is considered adequate if the regression value is near unity and having a relatively high regression value does not necessarily suggest that the model is adequate. They recommended that the adjusted regression value should be above 0.90 which should be near unity to be considered appropriate in determining how adequate the model is.

Employing the ANOVA on the experimental results to predict the mortar strength of OPA-ESP- cement blends indicated that the CCD model satisfied quadratic equation with F value of 38.75 while BBD model satisfied linear

equation with a low F value of 8.87 respectively. Table 4 presents the ANOVA results for the prediction of the mortar strength of OPA-ESP cement blends for CCD and BBD (S_{CCD}) and (S_{BBD}) respectively. The model developed for CCD indicated that the model terms such as A_1 , C_1 , B_1C_1 , B_1^2 and C_1^2 were considered to be significant with the other model terms having p values greater than 0.10 were considered not significant. Whereas, the model develops via BBD indicated that the model terms such as A_2 and C_2 were considered to be significant with the other model terms having p values greater than 0.10 were considered not significant.

The F values for CCD and BBD models for curing age (A) were 240.29 and 22.14; F values for both models for blending ratio (B) were 3.32 and 1.09; F value for CCD and BBD models for replacement level (C) were 27.46 and 3.38 for mortar strength respectively. The quadratic terms of blending ratio, replacement level independently and the interaction of blending ratio and replacement level all fell within $p < 0.05$ or $p < 0.10$ for CCD respectively and thus considered significant.

The quadratic term of curing age on the model for the prediction of mortar strength of OPA-ESP PLC was found to be insignificant evident by the low F values of 0.0246 for CCD which did not fall within $p < 0.05$ or $p < 0.10$ respectively. According to the ANOVA, the significant high F value of

curing age is a strong indication that the curing age has a significant influence on the mortar strength of OPA-ESP-cement blends in comparison with other factors such as blending ratio and replacement level. CCD and BBD models were found to be statistically significant with F values of 38.75 and 8.87 respectively.

Graphs for Predicted versus Actual and Normal Probability

Figure 6b illustrates a strong correlation between the experimental and predicted value for the mortar strength of OPA-ESP cement blends for CCD.

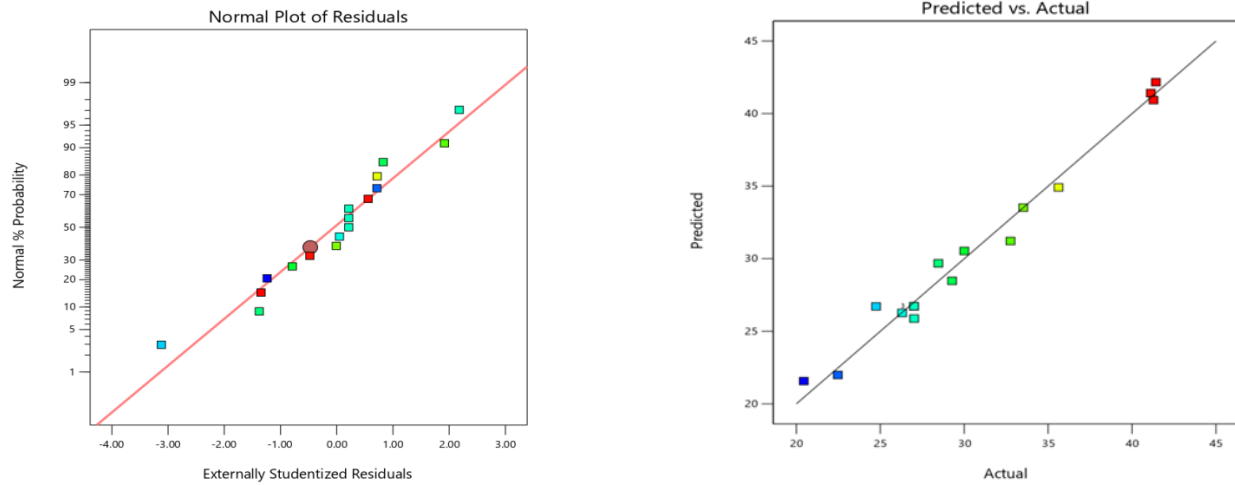


Figure 6: (a) Normal probability plot of residuals indicating significance of the model developed for mortar strength via CCD (b) Predicted against actual plot of the model developed for mortar strength via CCD

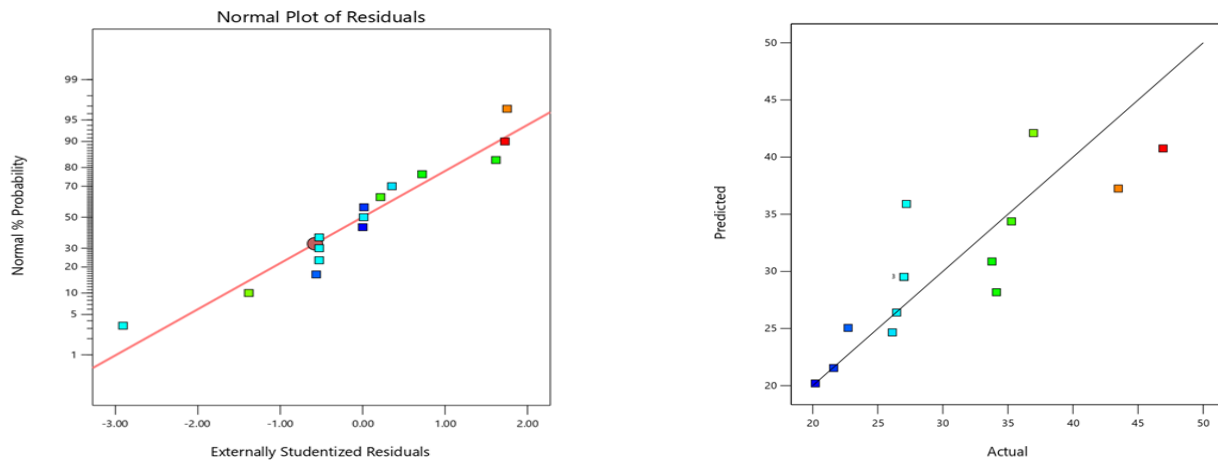


Figure 7: (a) Predicted against actual plot of the model developed for mortar strength via BBD (b) Normal probability plot of residuals indicating significance of the model developed for mortar strength via BBD

Table 6: Diagnostic Case Statistics for mortar strength of OPA-ESP-cements for CCD & BBD

Curing age Days	Blending ratio B	Replacement level wt.% C	Actual Value N/mm ² CCD	Predicted Value N/mm ² CCD	Curing age Days A	Blending ratio B	Replacement level wt.% C	Actual Value N/mm ² BBD	Predicted Value N/mm ² BBD
28	0.25	4	28.46	29.68	7	0.5	2	26.45	26.40
7	0.5	4	20.44	21.57	28	0.5	4	27.01	29.52
28	0.5	4	27.01	26.73	7	0.75	4	21.61	21.54
60	0.5	4	35.62	34.91	60	0.25	4	46.92	40.76
28	0.5	6	24.75	26.71	7	0.5	6	20.2	20.20
60	0.25	2	41.12	41.42	28	0.25	6	34.12	28.18
7	0.25	2	26.3	26.27	28	0.75	6	26.12	24.66
28	0.75	4	29.28	28.48	60	0.5	2	36.96	42.10
7	0.25	6	27.02	25.88	60	0.5	6	27.2	35.90
60	0.75	6	33.52	33.52	28	0.5	4	27.01	29.52
28	0.5	4	27.01	26.73	7	0.25	4	22.72	25.05
7	0.75	2	30.01	30.54	28	0.25	2	35.27	34.38
28	0.5	4	27.01	26.73	28	0.5	4	27.01	29.52
60	0.25	6	41.28	40.93	28	0.75	2	33.77	30.87
60	0.75	2	41.42	42.17	60	0.75	4	43.48	37.25

Table 6 presents the variation between the predicted and the actual values in the prediction of the mortar strength for OPA-ESP cement blends for CCD and BBD respectively.

Response Surface Plots for OPA-ESP cement blends

The response surface plot illustrates the correlation between the factors (curing age, blending ratio, replacement level) and the response (mortar strength) of OPA-ESP cement blend. Figures 8-9 show the diagnostic plots which determines the regression model adequacy showing the

response surface plots for the influence of following factors; A (curing age), B (blending ratio) C (replacement level) on the first response S_{CCD} (mortar strength of cement blend via CCD), second response S_{BBD} (mortar strength of cement blend via BBD) respectively. These response surface plots elucidate the interactions between the responses and their various factors; as well as determine the optimum for the various factors in order to obtain maximum responses. The parabolic nature of the contour observed in model obtained from CCD agrees with works by Dockery (2017), Silva et al. (2011), Olubajo et al.

(2022) implying that there exists an interaction between the factors considered which significant showed in Figures 8 (a) -(c).

The experimental design was employed to conduct the experiments in order to

investigate the correlation between the factors and the various responses. The experimental results of the full factorial Central composite design and Box and Behnken design were represented by equations 3 and 4 respectively.

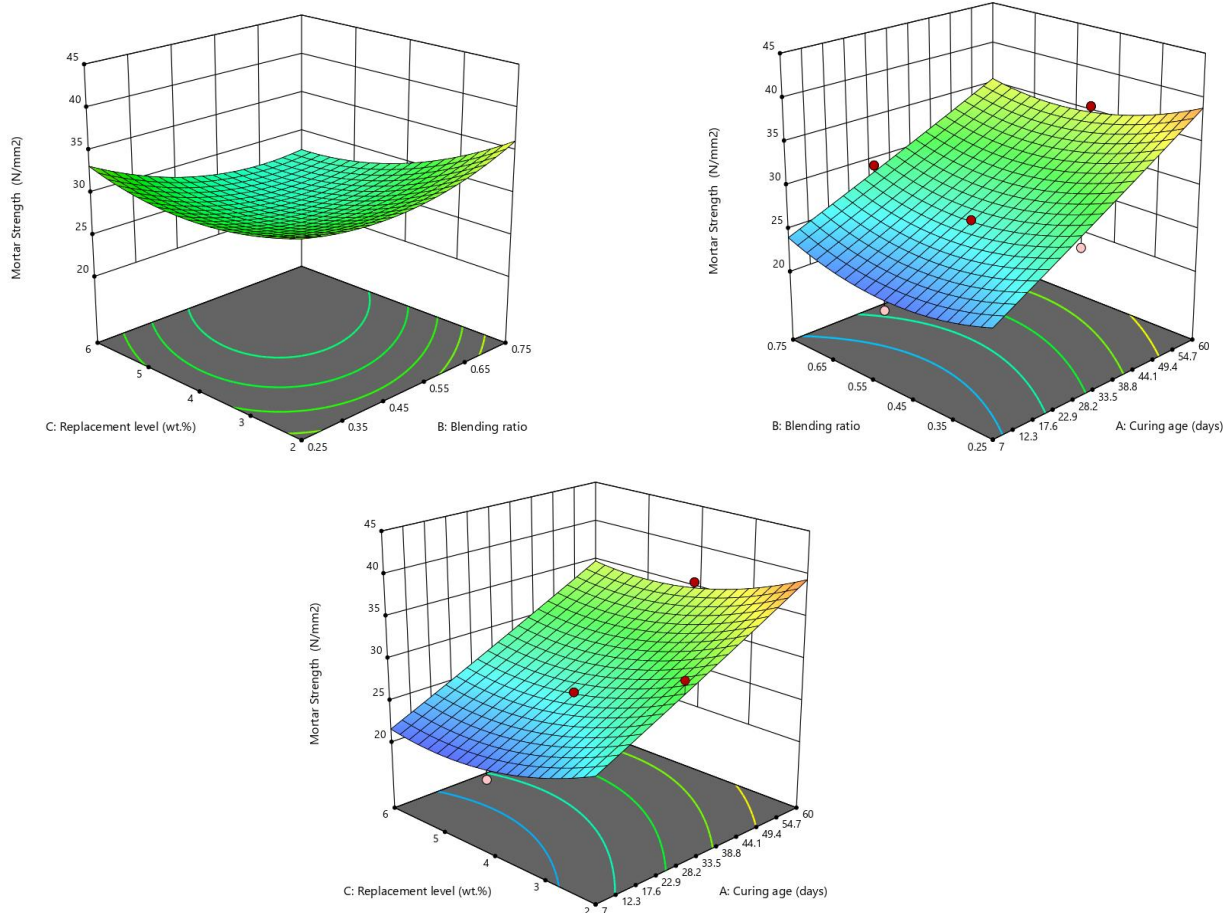


Figure 8: (a) Response surface plot for interaction on mortar strength of OPA-ESP-cement blends between blending ratio and curing age. (b) Response surface plot for interaction on mortar strength of OPA-ESP-cement blends between replacement level and curing age (c): Response surface plot for interaction on mortar strength of OPA-ESP-cement blends between replacement level and blending ratio.

It was seen that the equation obtained from the analysis indicated the parameter that significantly influencing the prediction of the mortar strength of OPA-ESP cement blends are curing age A, followed by replacement level C and finally

blending ratio B respectively. The curing age was found to produce the most significant effect on the response with a high F value of 240.29 followed by replacement level whose effect was quite significant on the response with a

moderate F value of 27.46 followed by the interaction between blending ratio and replacement level with a F value of 17.97 and quadratic term of blending ratio had a F value of 8.03 and replacement level had a F value of 7.26 which had a least significant effect on the mortar compressive strength of OPA-ESP cement blend according to ANOVA. Figures 8 and 9 illustrate the response surface plot illustrating the interaction between the response and the factors for CCD and BBD respectively. From figure 7 (a), it was observed that as the blending ratio and replacement level were increased simultaneously at constant curing age at 7 days, the mortar strength experienced a decrease at 0.5 and beyond 0.5 led to a slight increase. Whereas when the curing ages were held constant at 28 and 60 days, their mortar strengths experienced a decrease by 17.77 and 19.06% as the blending ratio and replacement level were increased respectively.

Figure 8 (b) showed that the mortar strength increased by 60.54%, 49.41% and 29.55% when the blending ratio and curing ages were increased simultaneously at constant replacement level at 2 - 6wt. % respectively. Figure 8 (c) showed that mortar strength of cement blends increased by 55.84% and 33.89% as the curing age and replacement levels were increased at constant blending ratios for 0.25 and 0.5 respectively. Whereas as the

blending ratio was increased between 0.25 to 0.5 led to a decrease in the mortar strength by 6.73% while as beyond blending ratio of 0.5 resulted in an increase in the mortar strength by 9.78% as the curing age and cement replacement was increased.

Similarly, it was observed that the most significant factor was the curing age and if other factors are held constant and the curing age was increased resulting in an increase in the mortar compressive strength, thus indicating has a positive effect related to the hydration reactions coupled with pozzolanic reactions stemming from silica present in OPA and excess lime present. Thus, production of more CSH as the curing days progressed. The trend agrees with the fact that the time of hydration determine the rate of hydration of cement (Jarumi et al., 2022; Olubajo et al., 2020a, Olubajo and Osha, 2013; Olubajo et al., 2017; Olubajo et al., 2020b, Olubajo et al., 2019; Olubajo et al., 2020c, Olubajo, 2020d). Figure 7 indicated an increase in the mortar compressive strength as the curing age and the blending ratio were increased while the cement replacement was held constant. Similarly, as the cement replacement and blending ratio were held constant and curing age was progressed the mortar compressive strength of OPA-ESP cement blend experienced an increase for BBD model.

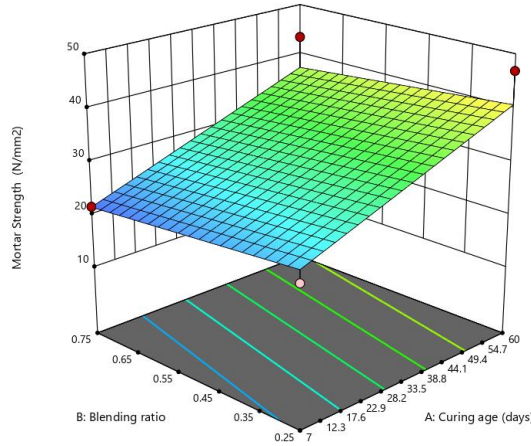


Figure 9: Response surface plot for interaction on mortar strength of OPA-ESP-cement blends between curing age and blending ratio via BBD.

Optimization of Mortar compressive strength of cement blends

Optimization of the mortar strength of OPA-ESP-cement blends were conducted and the optimal conditions were blending ratio of 0.75, replacement level of 2.0 wt. % and curing age of 60 days with a mortar compressive strength of 42.17 N/mm² with a desirability of 1.0 for CCD whereas BBD optimal conditions with

blending ratio of 0.25 replacement level of 2 wt. %, curing age of 60 days and mortar compressive strength of 43.86 N/mm² and desirability of 0.885. Figures 10 (a) and (b) depict graphs of the effect of replacement level and blending ratio as a function of mortar strength via CCD and the effect of curing age and blending ratio as a function of mortar strength via BBD respectively.

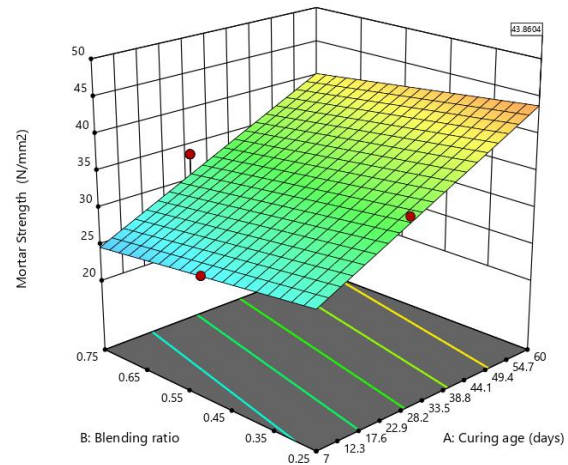
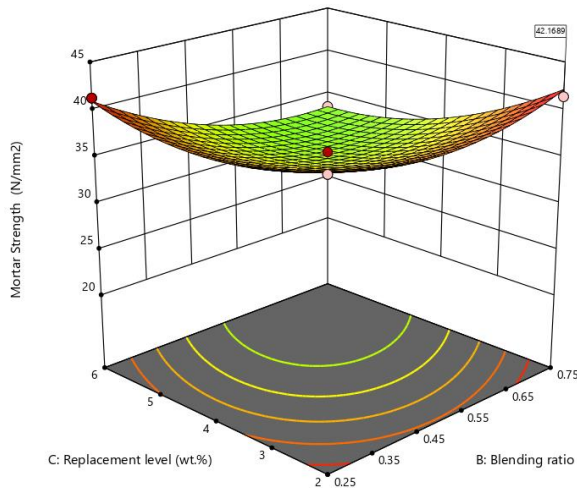


Figure 10 (a) Response surface plot indicating the optimal conditions for interaction on mortar strength of OPA-ESP-cement blends between replacement levels and blending ratio via CCD (b) Response surface plot indicating the optimal conditions for interaction on mortar strength of OPA-ESP-cement blends between blending ratio and Curing age via BBD

Conclusion

Chemical analysis revealed that the cement employed was classified as CEM II A-L 42.5R composed of four major oxides greater than 85 wt. % with Fe_2O_3 , MgO , Al_2O_3 , SO_3 , K_2O except CaO and SiO_2 which fell within the approximate amount for Portland limestone cement. OPA obtained by calcined at 650°C for 2 hours comprised mainly of lime, silica and potassium oxide with $\text{SiO}_2+\text{Al}_2\text{O}_3+\text{Fe}_2\text{O}_3$ (19.77 %) contained less than 70 % with CaO content > 10 % at 13.74 % which met Class C; hence the ash does not satisfy as a pozzolan according to ASTM C618-19 whereas ESP classified a filler with high lime content (54.09 wt.%) and CaCO_3 content of 96.55 wt.% with a loss of ignition of 15.81 % which indicates a high degree of unburnt carbon of ash. The X-ray pattern of OPA primarily reveals the presence of crystalline minerals like CaCO_3 represented by Calcite and Lime as the dominant peak at (CaCO_3 $2\theta= 36.2, 29.8$).

The dominant peaks in the XRD pattern corresponds to the crystalline calcium carbonate due to the high calcium content in citrus fruits. Minor peaks correspond to Potassium, Magnesium and silica which varies of the soil composition in which the oranges were grown. Other components include Fairchildite, Osumlite and muscovite. Results indicated that the XRF analysis of OPA seemed to tally with the components revealed from XRD pattern.

This work investigated the factors influencing the mortar strength of OPA-ESP-PLC conducted at laboratory conditions. The use of RSM 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 2 - 6 wt. % and curing age between 7 and 60 days.

The process optimization was carried out using RSM and the model equations for the strength prediction were obtained from 14 runs for both CCD and BBD respectively with CCD being the most reliable. The optimum blending ratio, replacement level and curing age were 0.75, 2.0 wt. % and 60 days producing the best strength of 42.17 N/mm^2 and desirability of 1.0 for CCD respectively while the optimum blending ratio, replacement level and curing age were 0.25, 2.0 wt. % and 60 days producing the best strength of 43.86 N/mm^2 and desirability of 0.885 for BBD respectively.

Amongst the factors, the curing age significantly influenced the strength gain due to increase in the hydration reaction time during curing from 7 - 60 days. The model equation developed via CCD can adequately predict the mortar strength of ternary cement blended with OPA and ESP between replacement level of 2 -6 wt. %. The regression and adjusted regression values for CCD were 0.9803 and 0.9550 respectively demonstrating that the developed model's appropriateness to

predict the mortar compressive strength for OPA-ESP cement blends. The optimal OPA content to obtain strength slightly above the control was 2 wt.% beyond which produced lower strengths in comparison to control.

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