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Modelling and optimization of multiple replacement of supplementary cementitious materials for cement composite by response surface method

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A R T I C L E I N F O

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A B S T R A C T

Supplementary cementitious materials are beneficial in improving performance and lessening the cement consumption with highly lessening CO_2 emission. Many researchers used blast furnace slag, bentonite, and active limestone separately or two of them together to improve the performance of cementing materials, however, it is not well known how all react together in cement composite materials. So, the present study used modeling and optimizing the replacement of blast furnace slag, raw bentonite, and active limestone each by the doses of 0 to 20% to maximize strength and minimize the fresh bulk density of cementing materials by central composite design-response surface method (CCD-RSM). The results found, the employment of blast furnace slag, bentonite, and active limestone in the cement composite materials generally lessens the early strength compared to the control mixture. However, the replacement of blast furnace slag and active limestone by 20% significantly improves the 28-days compressive strength while employing raw bentonite by 20% reduced compressive strength by 6.45% compared to the control mixture. However, blending raw bentonite with active limestone by half improved the compressive strength. Besides these, the substitution of bentonite and active limestone reduces the fresh bulk density and flexural strength than the control mixture. Generally, the study optimized depending on the criteria of maximizing strength and minimizing fresh density and found the mix design replacement of blast furnace slag 1.01%, raw bentonite 5.30%, and active limestone 20% that improves 28 days compressive strength simultaneously reduces fresh bulk density in addition to replacing more than 54 different optimized design mix results.

1. Introduction

Employing supplementary cementitious materials in the concrete mixture provides a lot of benefits to improve workability, lessens heat of hydration, and permeability, increases ultimate strength, and durability (Fezzioui et al., 2021; Ahmed, 2017; Tebbal and El Abidine Rahmouni, 2019; Chanakya and Behera, 2016). So, most construction works are highly using supplementary cementitious materials commonly known as pozzolana (Karthikeyan et al., 2015a; Salamatpoor et al., 2018). Pozzolana is mostly two types artificial and natural pozzolana. Most natural pozzolanas are from volcanic ashes like bentonite, kaolin, and pumice; while artificial pozzolanas are a wastage of different products mainly from the combustion of the furnace, and the utilization of waste that can be decomposed into ash which contains reactive silica like silica fume and blast furnace slag (Raggiotti et al., 2018; Walker and Pavía, 2011).

Natural pozzolanas (NP) are promised outcomes in grasping the expansion and strength of cement composite materials (Chihaoui et al., 2022; Paiva et al., 2016). That is through employing as a partial replacement of cement by natural pozzolana to reduce cement consumption and at the same time improve concrete performance (Taklymi et al., 2020; Robalo et al., 2021; Shukla et al., 2020; Pachideh and

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Gholhaki, 2019; Mahmoud et al., 2023). This is due to the pozzolanic reaction of most pozzolana may begin within the first day and its reaction with limestone starts from a little later stage. Hence, this reactions consume free lime produce C-S-H/C-A-S-H gels, and release additional heat of hydration (Du and Pang, 2020).

Bentonite is pozzolana mainly composed of montmorillonite compound and its replacement in cement is used for the reaction of montmorillonite with free calcium hydroxide to form extra C-S-H/C-A-S-H gels. The C-S-H/C-A-S-H gels are the sole cause for the strength development of cement in Portland pozzolana cement production and cement-based concrete (Karthikeyan et al., 2015a; Rathi and Modhera, 2015). Thus, replacing bentonite enhances the durability and compressive strength of concrete in construction works in addition to cost-effective concrete production and reduction of environmental pollution due to Ordinery Portland cement production (Karthikeyan et al., 2015b; Chandrakanth and Rao, 2016; Devi, 2018; Aravindhraj and Sapna, 2016).

The partial replacement of blast-furnace slag is another crucial material for improving the strength, setting time, and permeability of cement composite materials. Also, the incorporation of blast-furnace slag decreases failure strain and improves the ability of the material to withstand deformations without cracking (Macdonald, 2011).

Besides these, the addition of limestone in cement composite materials increases the early strength of cement while blast-furnace slag and bentonite enhance the later strength by the pozzolanic reaction that refines the pores of cement composite materials (Saraya, 2014). In another direction, pozzolana promotes portlandite dissolution and production of monosulfate at earlier ages while limestone can stabilize ettringite and enhance the metastable formation of hemicarbonate followed by the formation of stable monocarbonate (Celik et al., 2019).

Many studies found the positive aspect of employing supplementary cementitious materials in cement composite materials. Mostly, extensive researches were conducted to the benefit of adding blast furnace slag, bentonite, and limestone separately in cementing materials. However, there is no study that optimized simultaneously the replacement effect of blast furnace, bentonite, and active lime stone in the cement composite materials. So, the present study investigated the effect of replacing those three supplementary cementitious materials by modeling and optimizing the independent factors of different replacement doses on the mechanical and physical properties as a response factor using the Central composite design-response surface method and performed its validation experimentally.

2. Materials and methods

2.1. Materials

Mortar specimens were prepared by the mixture of Ordinary Portland cement CEM I 32.5A-P, sand, water, and supplementary cementitious materials blast furnace slag, raw bentonite, and active limestone all having a replacement dose that ranges from 0 to 20% to the cement weight, which was selected based on our previous work recommended for different artificial and natural pozzolana replacement dose up to 20% (Fode et al., 2023). The washed river sand was used and the chemical compositions of used blast furnace slag, bentonite, limestone, and ordinary Portland cement are presented in Table 1, which shows both used pozzolana fulfill the requirement of the ASTM C618 (ASTM C618, 2012) by having SiO_2, Al_2O_3, and Fe_2O_3 more than 70%. The distilled water was used for mixing all ingredients of sampled mortar. Also, the physical color difference of used blast furnace slag, bentonite, and active limestone are shown in Fig. 1.

2.2. Sample preparation

The mixing process started by measuring all mortar ingredients cement, sand, and water as ASTM C109M-02, (ASTM C109, 2002), water to cement ratio of 0.485 and sand to cement ratio 2.75, with 20 different doses of blast-furnace slag, bentonite, and limestone as shown in Table 2, which were prepared three times for each run, a total of sixty separate mixing process undergone immediately one after the other. Then, from all sixty separate mortar mix was taken one individual fresh and hardened mortar cast processed. The correlation of all casted mortar for hardened mix was assessed as ASTM C109M-02, (ASTM C109, 2002), and finally, for all fresh and hardened test average of the three samples within one run was taken as a record of each run.

Table 1
Chemical composition of used ordinary Portland cement and different supplementary cementitious materials.

Compositions	OPC	Blast furnace slag	Bentonite	Active limestone
SiO_2	17.57	42.43	50.51	2.29
Al_2O_3	4.07	17.77	12.62	0.33
Fe_2O_3	2.63	12.34	7.81	0.21
CaO	61.48	14.73	1.91	54.12
MgO	0.41	6.09	6.38	0.43
SO_3	1.79	1.17	0.13	0.14
K_2O	0.11	1.13	2.02	0.01
Na_2O	0.04	1.61	2.88	0.00
TiO_2	0.29	0.79	1.00	0.00
P_2O_5	0.13	0.22	0.34	0.02
SAF	24.27	72.54	70.94	2.83
LoI	10.74	0.84	10.74	42.53
Physical characteristics				
Specific gravity (cm^3/g)	3.06	3.10	2.550	2.673
Blaine (cm^2/g)	4266	3832	8239	6298
Initial setting time (min)	72	–	–	–
Final setting time (min)	121	–	–	–

SAF= Sum (SiO_2, Al_2O_3, Fe_2O_3).

2.3. Fresh and hardened test methods

The fresh physical property of mortar was considered by fresh bulk density test by taking the known volume of mortar in 1 L cylindrical mold. A flexural strength test was conducted by the use of three point loading machine for the mortar specimen at age of 28 days. Also, according to ASTM C349 (ASTM C349, 2002) standard hardened test for compressive strength test was conducted by half prism 40*40*160 mm^3 of mortar specimens used for flexural strength test, which is performed by the use of a digital compressive testing machine at the rate of 2400N/sec. The compressive strength response used in all 20 runs was measured and recorded at the age of 2 and 28 days. For each batch, three samples were casted and a total of 120 samples were exposed to the compressive strength test, and then the average of each run from three samples was recorded. For the last optimized value, the experimental validation was taken from the average of three samples for fresh bulk density and strength, having 40*40*160 mm^3 prism recasted to validate the software optimum suggested value to the actual experimental result.

2.4. Experimental design using CCD-RSM

The Response Surface Method (RSM) is a statistical technique which responds quickly and efficiently, used for the interactive effects of two or more independent factors and optimizes the process with the least number of experimental runs. Besides these, it can analyze multiple independent factor effects on the dependent variables (Cai et al., 2013). This means response surface methods can determine the correlation between one or more response (dependent) variables and a set of quantitative experimental (independent) variables or factors. The most well-known design technique in the RSM is central composite design (CCD), which offers both interaction effects of the independent factors

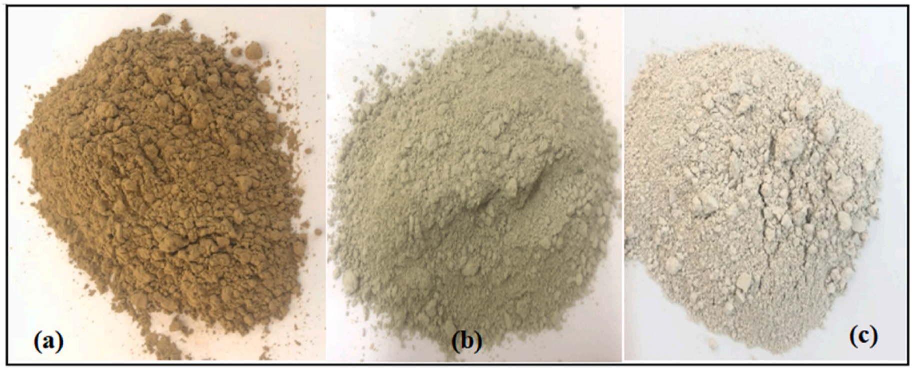

Fig. 1. The colour difference of used (a) blast furnace slag, (b) raw bentonite, and (c) active limestone. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

Table 2
Independent variables and there actual values for CCD-matrix.

Experimental runs	Space type	Experimental conditions		
		A: Blast furnace slag (%)	B: Bentonite (%)	C: Lime stone (%)
1	Factorial	20	20	20
2	Factorial	0	0	0
3	Factorial	20	0	20
4	Factorial	0	20	20
5	Center	10	10	10
6	Center	10	10	10
7	Center	10	10	10
8	Factorial	20	0	0
9	Factorial	0	20	0
10	Center	10	10	10
11	Factorial	0	0	20
12	Factorial	20	20	0
13	Axial	10	20	10
14	Center	10	10	10
15	Center	10	10	10
16	Axial	0	10	10
17	Axial	20	10	10
18	Axial	10	10	20
19	Axial	10	0	10
20	Axial	10	10	0

for a substantial and accurate prediction of the dependent variables (Cai et al., 2013; Gour et al., 2022). The entire number of experimental runs (N) for RSM is determined by applying Eq. (1), where N, C0, and k stand for the necessary experimental runs, center points, and process variables, respectively.

$$N = 2^K + 2K + C_0 \tag{1}$$

The CCD-RSM in Design Expert Software (State-Ease, version 13), which assessed the interacting effects of three independent doses of different supplementary cementitious material substitution variables, blast furnace slag (A), bentonite (B), and active limestone (C), having 0 to 20% replacement ranges, on the response variables.

As presented in Table 2, different configurations of a set of 20 experimental runs were statistically generated by the CCD-RSM. So, the CCD matrices are coded with corresponding response parameters such as Y_1: 2-days compressive strength (MPa), Y_2: 28-days compressive strength (MPa), Y_3: fresh bulk density (Kg/m^3), and Y_4: flexural strength (MPa), are listed in Table 2, which have factorial points, axial points, and center points. As shown in Table 2, from 20 experimental runs, 6 were center runs, 8 factorial runs, and the other 6 were axial runs, which were used for the processes of modeling and optimization. So, to ensure the authenticity and reliability of the response variables all 20 runs were measured three times, and the average of the values was recorded for all response factors, which have 8 factorial, 6 center, and 6 axial points of different response variables. The points at the center are used to check the curvature and the axial points are for estimation of quadratic terms (Coruh et al., 2015).

3. Results and discussion

3.1. Building response surface quadratic models

The CCD-RSM of the Design Expert software experimental data suited to various models, such as linear, logarithmic, quadratic, and cubic models, for modeling and optimizing substitution of active limestone, bentonite, and blast furnace slag in cement composite materials. Therefore, the statistical experiment that can most accurately anticipate the response variables is done by multiple regression analyses. Analysis of variance (ANOVA) was used to assess the quadratic model's significance and compatibility. The response parameters Y_1: 2-days compressive strength (MPa), Y_2: 28-days compressive strength (MPa), Y_3: fresh bulk density (Kg/m3), and Y_4: flexural strength (MPa) were used by the CCD-RSM, as indicated in Table 3. Hence, the generalized Eq. (2) shows the regression models as functions of all independent influencing factors.

$$Y = a_0 + \sum_{i=1}^{n} a_i X_i + \sum_{i=1}^{n} a_{ii} X^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^{n} a_{ij} X_i X_j + \varepsilon \tag{2}$$

where Y is the predicted response variable, α_0 is the constant coefficient, α_i stands for a linear coefficient, α_{ii} a quadratic coefficient, α_{ij} an interaction coefficient, X_i and X_j coded values of variables, and ε the error or unpredicted response variables on the experimental data.

As presented in Table 4, the quadratic models were recommended and selected as statistically significant models for all response variables, with sequential p-values higher than those of the cubic, 2FI, and linear regression models. All replies' cubic models are aliased, which indicates that the model is inappropriate (Myers and Montgomery, 2016). The adjusted R^2 of 0.9038, 0.7763, 0.7763, and 0.9685 as well as the predicted R^2 of 0.8192, 0.7245, 0.7245, and 0.9511 respectively for 2 and 28-days compressive strength, fresh bulk density, and flexural strength which is having less than 0.2 discrepancy that shows a good correlation of the model predicted values (Mohammed et al., 2019; Dahish and Almutairi, 2023). As a result, a substantial quadratic model can be reflected by correlating the anticipated and experimental data based on the quadratic equation's coefficient. Besides these, the model's quality can be assessed through lack of fit. The p-values for all responses, Y_1, Y_2, Y_3, and Y_4, were, respectively, 0.9620, 0.6618, 0.6056, and 0.9620, indicating that the quadratic model's lack of fit is not significant and the model has excellent fitness and robustness (Mohammed et al., 2019).

The acceptable precision can be used to reflect the signal-to-noise ratio, which must be higher than 4.00 in order to be approved. According to Table 5's results, the recommended statistical models have

Table 3
Blast furnace slag, bentonite, and active limestone replacement factors of coded form in the CCD matrix with the corresponding response variables.

Runs	Space type	Factor 1	Factor 2	Factor 3	Response-1	Response-2	Response-3	Response-4
		A: Blast furnace slag (%)	B: Bentonite (%)	C: Active limestone (%)	2-days Compressive strength (MPa)	28-days Compressive strength (MPa)	Fresh bulk density (Kg/m^3)	Flexural strength (MPa)
1	Factorial	1.000	1.000	1.000	4.65	16.20	2417.27	4.85
2	Factorial	-1.000	-1.000	-1.000	20.90	43.40	2543.17	8.02
3	Factorial	1.000	-1.000	1.000	10.25	35.10	2525.18	7.30
4	Factorial	-1.000	1.000	1.000	10.80	38.20	2330.94	6.15
5	Center	0.000	0.000	0.000	10.55	36.20	2489.21	6.90
6	Center	0.000	0.000	0.000	11.30	39.10	2525.18	6.80
7	Center	0.000	0.000	0.000	9.00	36.00	2507.19	6.50
8	Factorial	1.000	-1.000	-1.000	15.55	47.30	2532.37	8.20
9	Factorial	-1.000	1.000	-1.000	15.05	40.60	2446.04	6.45
10	Center	0.000	0.000	0.000	11.25	35.00	2450.00	6.95
11	Factorial	-1.000	-1.000	1.000	19.15	46.40	2550.36	7.80
12	Factorial	1.000	1.000	-1.000	10.65	31.40	2541.35	5.55
13	Axial	0.000	1.000	0.000	10.65	29.00	2515.04	6.05
14	Center	0.000	0.000	0.000	11.50	38.00	2500.00	6.95
15	Center	0.000	0.000	0.000	10.60	37.00	2520.00	6.60
16	Axial	-1.000	0.000	1.316	17.90	44.40	2552.63	7.25
17	Axial	1.000	0.000	0.000	11.45	32.50	2575.19	6.60
18	Axial	0.000	0.000	1.000	11.75	31.50	2500.43	6.30
19	Axial	0.000	-1.000	0.000	17.10	39.30	2605.26	8.30
20	Axial	0.000	0.000	-1.000	18.05	38.90	2575.19	6.80

Table 4
Different model fitness summary for 2 and 28-days compressive strength, fresh bulk density, and flexural strength.

Response	Source	Sequential p-value	Lack of fit p-value	Adjusted R^2	Predicted R^2	Remark
2-days Compressive strength	Linear	<0.0001	0.0762	0.7907	0.7079	
	2FI	0.6510	0.0584	0.7706	0.3844	
	Quadratic	**0.0122**	**0.2293**	**0.9038**	**0.8192**	**Suggested**
	Cubic	0.9274	0.0398	0.8509	-97.1474	Aliased
28-days Compressive strength	Linear	<0.0001	0.0322	0.7127	0.4164	
	2FI	<0.0001	0.3771	0.9359	0.9053	
	Quadratic	**0.00651**	**0.6734**	**0.9602**	**0.9203**	**Suggested**
	Cubic	0.8670	0.2405	0.9422	-16.6563	Aliased
Fresh bulk density	Linear	0.0010	0.2818	0.5812	0.2419	
	2FI	0.7800	0.7403	0.8118	0.8061	
	Quadratic	**0.0053**	**0.6056**	**0.7763**	**0.7245**	**Suggested**
	Cubic	0.9942	0.1279	0.6124	-175.1786	Aliased
Flexural strength	Linear	<0.0001	0.2576	0.8970	0.8219	
	2FI	0.0395	0.4637	0.9341	0.9194	
	Quadratic	**0.0213**	**0.9620**	**0.9685**	**0.9511**	**Suggested**
	Cubic	0.9614	0.5827	0.9490	-2.9839	Aliased

Table 5
Fit statistics for 2 and 28-days compressive strength, fresh bulk density, and flexural strength.

Statistics	2-days compressive strength	28-days compressive strength	Fresh bulk density	Flexural strength
Standard deviation (Std. Dev.)	1.2700	1.4100	27.0200	0.1568
Mean	12.9100	36.7700	2510.100	6.8200
Percentage coefficient of variance (C.V %)	3.8600	3.8400	1.0800	2.3000
Correlation coefficient (R^2)	0.9519	0.9801	0.8882	0.9843
Adjusted R^2	0.9038	0.9602	0.7763	0.9685
Predicted R^2	0.8192	0.9203	0.7245	0.9511
Adequate precision	18.2191	29.6025	13.1845	29.2473

precision rates of 18.2191, 29.6025, 13.1845, and 29.2473. This suggests that the chosen quadratic model is suitable and reliable for predicting the response (Dahish and Almutairi, 2023).

3.2. Analysis of variance for quadratic model response

By the ANOVA of the quadratic models, the interacting influences and statistical significance of the factors on response were identified. Hence, the ANOVA determined the interaction of model fitness, and the significance of independent factors and their interactions using *F* and *P*-values of dependent factors in the cement composite materials. As presented in Table 6, ANOVA for the quadratic models derived from the response variables for all responses *p*-value is less than 5% which is a significant model, indicating that the independent factors have an effect on response parameters (Dahish and Almutairi, 2023). Also, to be a selected quadratic model to well fit the experimental data the lack of fit (LOF) would be not significant. The p-values of LOF given in Table 6, for all responses are not significant compared to the pure error, suggesting good quadratic model adaptability (Mohammed et al., 2019).

Also, as shown in Eq. (3) A, B, C, and BC have a negative impact, and AB, A^2, B^2, C^2, and AC have a positive impact on the 2-days compressive strength response. That shows all of the proposed supplementary

Table 6
ANOVA of the quadratic model developed for all used response variables

Sources	Sum of squares	df	Mean square	*F*-value	*p*-value	Remark
Compressive strength at 2-days						
Block	6.91	1	6.91			
Model	288.68	9	32.08	19.79	<0.0001	Significant
A- Blast furnace slag dose	97.66	1	97.66	60.26	<0.0001	
B- Bentonite dose	97.03	1	97.03	59.88	<0.0001	
C- Active limestone dose	55.70	1	55.70	34.37	0.0002	
AB	1.71	1	1.71	1.06	0.3310	
AC	3.51	1	3.51	2.17	0.1751	
BC	1.28	1	1.28	0.7899	0.3973	
A^2	4.45	1	4.45	2.75	0.1318	
B^2	0.6386	1	0.6386	0.3941	0.5458	
C^2	6.14	1	6.14	3.79	0.0834	
Residual	14.58	9	1.62			
Lack of Fit	10.73	5	2.15	2.22	0.2293	Not significant
Pure Error	3.86	4	0.9644			
Cor Total	310.18	19				
Compressive strength at 28-days						
Block	2.70	1	2.700			
Model	884.96	9	98.330	49.2800	<0.0001	Significant
D- Blast furnace slag dose	255.03	1	255.030	127.8200	<0.0001	
E- Bentonite dose	314.72	1	314.720	157.7400	<0.0001	
F- Active limestone dose	116.96	1	116.960	58.6200	<0.0001	
AB	70.80	1	70.800	35.4900	0.0002	
AC	98.00	1	98.000	49.1200	<0.0001	
BC	8.82	1	8.820	4.4200		
A^2	18.99	1	18.990	9.5200	0.0130	
B^2	7.22	1	7.220	3.6200	0.0895	
C^2	0.935	1	0.935	0.4686	0.5109	
Residual	17.96	9	2.000			
Lack of Fit	8.13	5	1.630	0.6618	0.6734	Not significant
Pure Error	9.83	4	2.460			
Cor Total	905.62	19				
Fresh bulk density						
Block	14403.53	1	14403.53			
Model	52199.88	9	5799.99	7.94	0.0025	Significant
G- Blast furnace slag dose	2830.01	1	2830.01	3.88	0.0805	
H- Bentonite dose	25573.93	1	25573.93	35.02	0.0002	
I- Active limestone dose	9856.65	1	9856.65	13.50	0.0051	
AB	5919.39	1	5919.39	8.11	0.0192	
AC	68.26	1	68.26	0.0935	0.7668	
BC	7151.79	1	7151.79	9.79	0.0121	
A^2	286.09	1	286.09	0.3917	0.5469	
B^2	115.67	1	115.67	0.1584	0.6999	
C^2	668.13	1	668.13	0.9149	0.3638	
Residual	6572.54	9	730.28			
Lack of Fit	3272.19	5	654.44	0.7932	0.6056	Not significant
Pure Error	3300.35	4	825.09			
Cor Total	73175.95	19				
Flexural strength						
Block	0.0216	1	0.0216			
Model	13.85	9	1.54	62.58	<0.0001	Significant
J- Blast furnace slag dose	1.00	1	1.00	40.86	0.0001	
K- Bentonite dose	11.17	1	11.17	454.33	<0.0001	
L- Active limestone dose	0.6864	1	0.6864	27.91	0.0005	
AB	0.4418	1	0.4418	17.97	0.0022	
AC	0.1458	1	0.1458	5.93	0.0377	
BC	0.0018	1	0.0018	0.0732	0.7928	
A^2	0.0064	1	0.0064	0.2602	0.6223	
B^2	0.2397	1	0.2397	9.75	0.0123	
C^2	0.2856	1	0.2856	11.61	0.0078	
Residual	0.2213	9	0.0246			
Lack of Fit	0.0382	5	0.0076	0.1669	0.9620	Not significant
Pure Error	0.1831	4	0.0458			
Cor Total	14.09	19				

cementitious materials can reduce the early strength, due to pozzolanic material's slow hydration reaction at an early age of cement composite materials. In another direction observed from Eq. (4) factors A, B, C, BC, and A^2 have positive impacts while AB, AC, B^2, and C^2 have negative impacts on the improvement of 28-days compressive strength. This shows the employment of pozzolanic materials blast furnace slag and bentonite can be used for late strength improvement and active limestone can increase the strength due to its active reactivity with a pozzolanic material to improve the hydration reaction of cement composite materials.

In addition to these as shown in Eq. (5), factors A, A^2, AB, and B^2 have a positive impact, and B, C, B^2, C^2, AC, and BC have a negative influence on improving the fresh density of cement composite materials. This shows employment of bentonite and active limestone lessens the fresh bulk density of cement composite materials while the substitution of blast furnace slag increases the density. Also, as presented in Eq. (6), factors C, BC, A^2, and B^2 have a positive impact and A, B, AB, AC, and C^2 have a negative impact on the improvement of flexural strength at 28 days of mortar age, which shows employment of blast furnace slag in cement composite materials improves the flexural strength.

$$Y_1 \text{ (2-days compressive strength)} = +11.52 - 3.12A - 3.12B - 2.36C + 0.4625AB - 0.06625AC - 0.4000BC + 1.29A^2 + 0.4877B^2 + 1.51C^2 \quad (3)$$

$$Y_2 \text{ (28-days compressive strength)} = +43.4876 + 0.3895A + 0.16954B + 0.23104C - 0.02975AB - 0.0350AC + 0.0105BC + 0.02659A^2 - 0.0164B^2 - 0.0059C^2 \quad (4)$$

$$Y_3 \text{ (Fresh bulk density)} = +2515.07 + 16.82A - 50.57B - 31.40C + 27.20AB - 2.92AC - 29.90BC + 10.32A^2 + 6.56B^2 - 15.78C^2 \quad (5)$$

$$Y_4 \text{ (Flexural strength)} = +8.1153 - 0.0045A - 0.1435B + 0.051C - 0.0024AB - 0.0014AC + 0.0002BC + 0.0005A^2 + 0.00299B^2 - 0.0033C^2 \quad (6)$$

Besides this, the ANOVA presented in Table 6 shows the *p*-values for all responses are less than 0.05 and 62.58, 7.94, 49.28, 62.58 *F*-values which is very high compared to *p*-values, which shows that the models are significant statistically and indicates the higher influence of the model terms on the response and mostly shows the effectiveness of the model (Gour et al., 2022; Dahish and Almutairi, 2023).

3.3. Quadratic models suitability and adequacy testing

To identify the appropriateness and suitability of the suggested quadratic models the diagnostic graphs for the effect of blast furnace slag, bentonite, and active limestone substitution on early and late strength, fresh bulk density, and flexural strength of cementing material are presented in Figs. 2–7. The suitability and appropriateness of the model can be evaluated using CCD-RSM diagnostic test.

One of the diagnostic tests used to evaluate the normal residual and the validating multivariate regression models is the normal percent probability plot of the studentized residuals. The normal % probability vs internally and externally studentized residuals plots for 2 and 28 days compressive strength, fresh bulk density, and flexural strength by the addition of different supplementary cementitious materials are presented in Fig. 2(a–h), respectively. The result shows that the residual distributions of the various substitution impacts of supplementary cementitious materials on compressive strength, fresh bulk density, and flexural strength are evenly distributed near both sides of the line, which shows the used quadratic models' are realistic. As Mohammed et al. (2019) found the residual data points are near the straight line with minor fluctuations in the normal % probability plots which demonstrates a small normal error distribution of the response variables. Furthermore, the quadratic model that was utilized is adequate, reliable, and valid, as indicated by the random normal distribution of residuals between +3 and −3 and + 2 and −2 sigma values being within the specified limits (Cai et al., 2013).

As shown in Fig. 3(a–h), the dependent valuables are within the prescribed ranges of minimum and maximum values from +3.00 or−3.00 and + 4.33355 or−4.33355 sigma for internal and external studentized residuals versus the predicted values of all the run points respectively.

As shown in Fig. 4(a–d), the DFFITS versus run number of all the values of response points are distributed in between the limit of +2 and−2 which can show the anticipated data can be affected by the experimental data.

The cook's distance can predict the impact of specific data points to uncover data outliers. To be the selected quadratic model accepted and

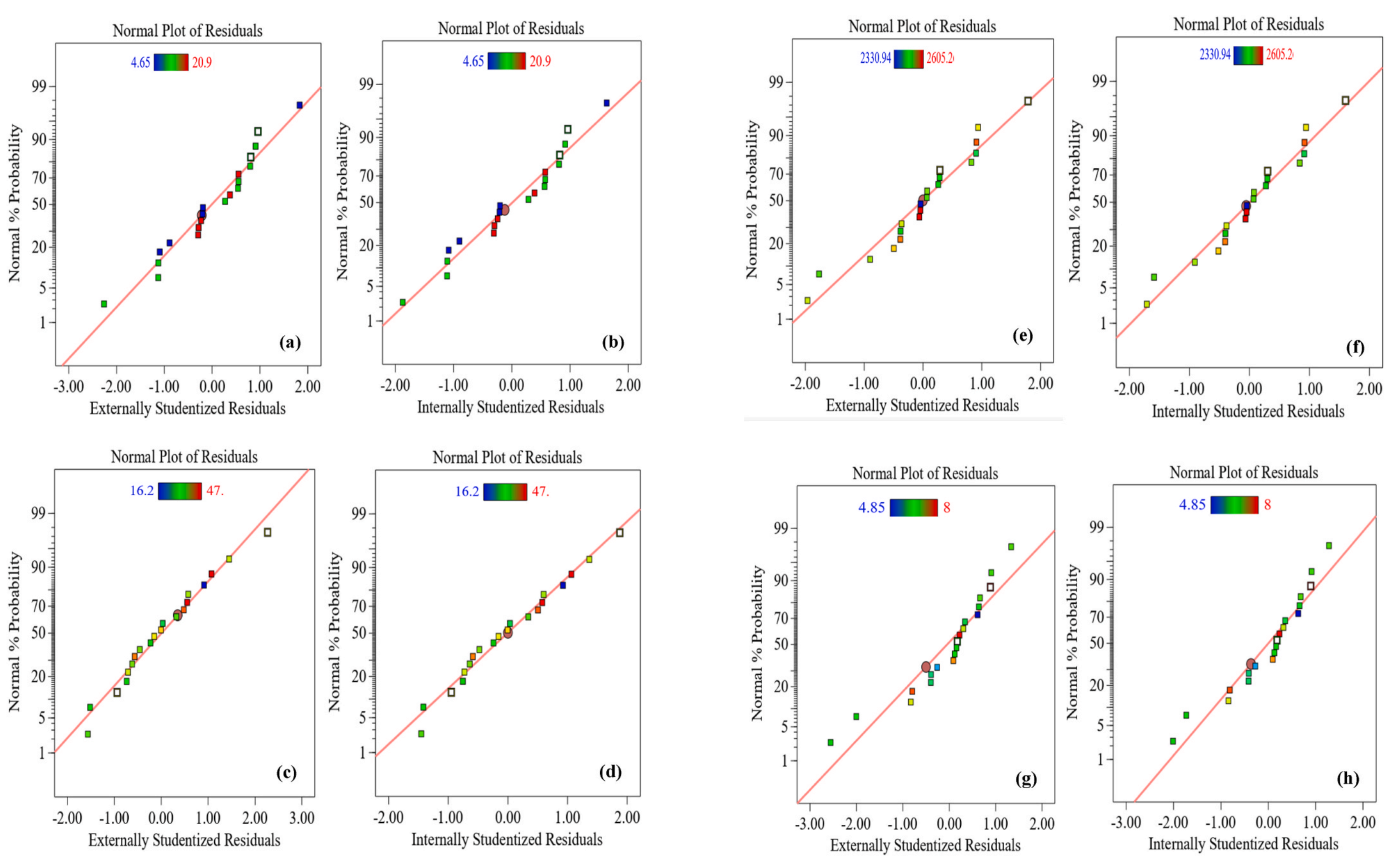

Fig. 2. Normal % probability plot versus external and internal studentized residuals (a,b) 2 days compressive strength, (c,d) 28 days compressive strength, (e,f) fresh bulk density, and (g,h) flexural strength.

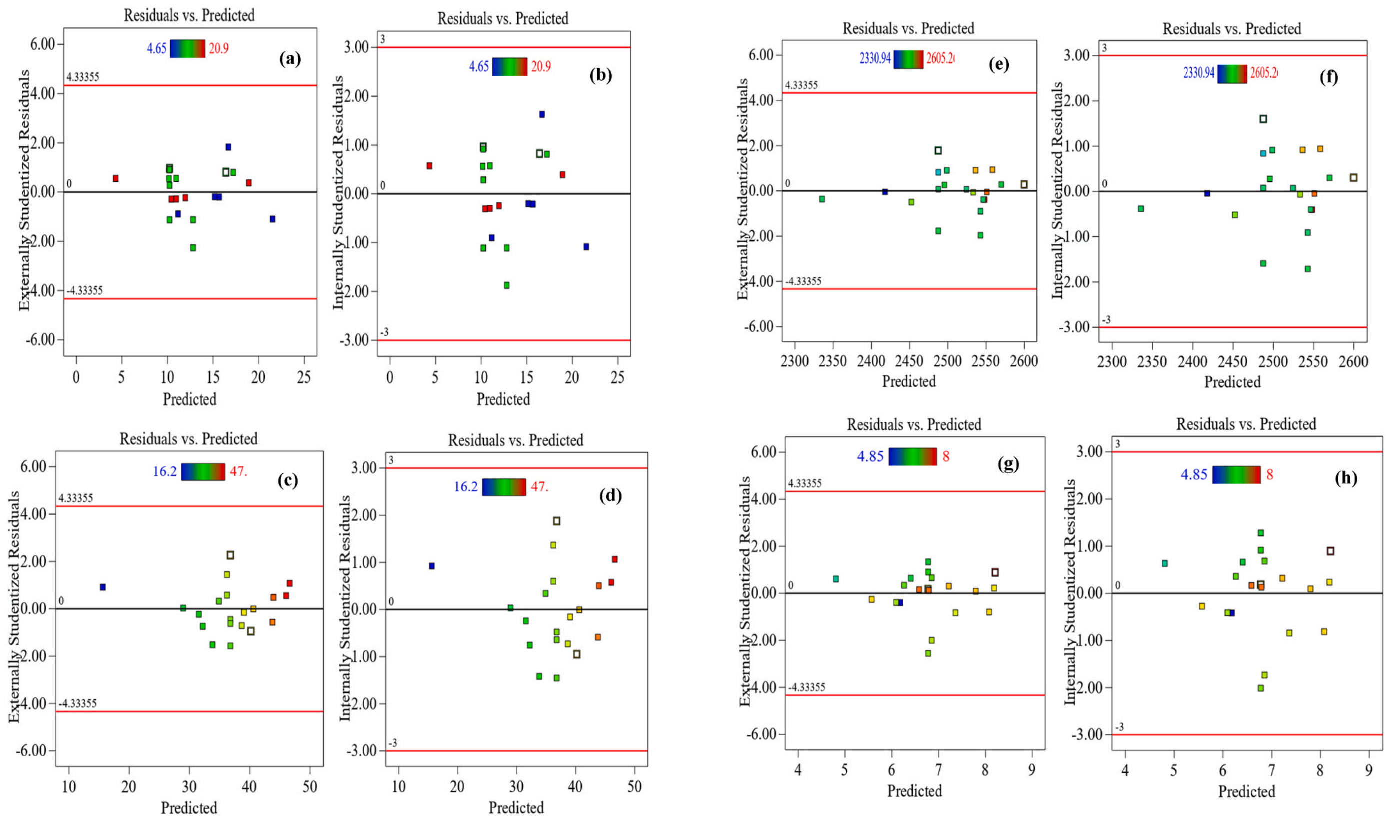

Fig. 3. External and internal studentized residuals versus run predicted (a,b) 2-days compressive strength, (c,d) 28-days compressive strength, (e,f) fresh bulk density, and (g,h) flexural strength.

accurate all the points have to fall within the range of 0 to 1. So, as presented respectively in Fig. 5(a–d), the 2 and 28-days compressive strength, fresh bulk density, and flexural strength changes of all the data points are within the limited cook's points, which indicates that no one of the data points is destructive to the quadratic models.

As shown in Fig. 6(a–d) the lambda values of 2 and 28-days compressive strength, fresh bulk density, and flexural strength were found 1 lambda, which indicates no transformation required, since the quadratic model is accurate and shows the strong fitness of the selected quadratic models (Myers and Montgomery, 2016).

As results presented in Fig. 7(a–d) shows the proposed quadratic models applied can fit the predicted data and the actual data of all responses, respectively as shown in Eqs. (3)–(6). The graph indicates the significant relation between the experimental data and quadratic models' predicted data for all response variables, which all 20'n runs are laying on and near the predicted line graphs. Besides these, the graph shows the regression model best fits the data, due to the variation between the predicted and the actual responses are not significant which shows the suggested response surface quadratic models were accurate and have valid predictions (Dahish and Almutairi, 2023).

3.4. Factors interaction and response surface modeling

As presented in Figs. 8–11, 3D response surfaces and contour plots of response variables are the graphical representations of regression of quadratic models, which are developed to investigate the interactive relationship between the independent variables and responses.

3.4.1. Compressive strength at an early age

The employment of bentonite and blast furnace slag as shown in Fig. 8(a and b) reduces the early strength compared to the control mixture at (0,0) in Fig. 8(b), which is due to pozzolanic materials reduce the hydration reaction at the fresh state (Wang et al., 2019; Fode et al., 2024). However, as shown in Fig. 8(c–f) the substitution of blast furnace slag up to 5% and bentonite up to 10% simultaneously while increasing the active limestone from 0 to 20% can give higher early strength. This means from the three additives active limestone more improves the early strength, which is commonly due to the abundant content of CaO in the active limestone which potentially participates in the hydration reaction to improve the early strength of cement composite materials. Also, from the result observed that bentonite is more reactive than blast furnace slag at the early age of cementing materials that can replace cement than blast furnace slag to get higher early strength.

The same observation with Makhloufi et al. (2015) as blast furnace slag and natural pozzolana lessen the early strength because of their slow hardening kinetics, that has almost proportional to the increase of the percentage of the two additives, however, the study reported that the strength of mortar with a high percentage of blast furnace slag is almost comparable to that of the control mortar at a late age. Also, Ghrici et al. (2007) reported the point of maximum early strength is around 10% of limestone and low NP replacement level to the cement composite materials. But after 28 days, this point moves toward the high level of NP substitution and low limestone, which is commonly due to pozzolanic reaction gradually increases the strength of cementing materials.

3.4.2. Compressive strength at late age

As shown in Fig. 9(a–d) up to 5% replacement of blast furnace slag while increasing the content of bentonite from 0 to 20% can increase the strength, which is mostly due to the pozzolanic reaction of bentonite and blast furnace slag at late age which can more consume the free calcium hydroxide and produce C-S-H/C-A-S-H that can participate for the improvement of the strength (Voit et al., 2020; Ahad et al., 2018; Borg et al., 2018; Trümer et al., 2019). Also, increasing the content of limestone from 0 to 20% and the blast furnace up to 5% can give more than 40 MPa of compressive strength which shows the pozzolanic reaction of the blast furnace slag and CaO from limestone can participate in the

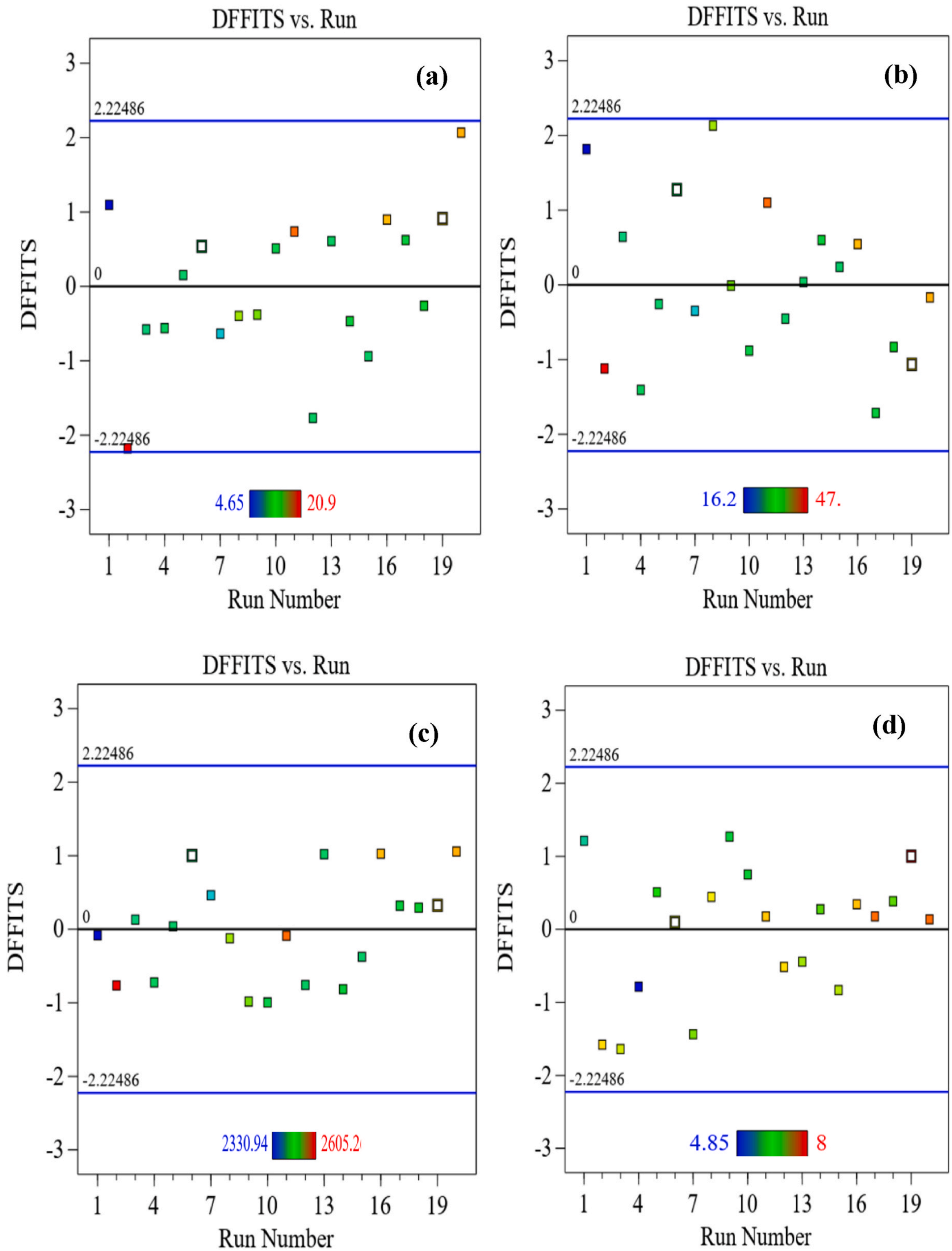

Fig. 4. Plot of DFFTTS versus run number (a) 2-days compressive strength, (b) 28-days compressive strength, (c) fresh bulk density, and (d) flexural strength.

strength development due to formation of extra entringite (Karthikeyan et al., 2015a; Rathi and Modhera, 2015).

Besides these, as shown in Fig. 9(e and f) employment of limestone and blast furnace slag both separately up to 10% can give strength of more than 40 MPa. However, increasing both limestone and blast furnace slag up to 20% significantly lessens the compressive strength than the lower replacement.

Also, increasing the replacement of blast furnace slag up to 20% with the increasing bentonite dose improves the compressive strength. That is due to at late age, blast furnace slag and bentonite, undergo hydration reactions in the presence of water with free calcium hydroxide. Hence, this secondary pozzolanic reaction makes a denser microstructure because the free calcium hydroxide was consumed and C–S–H gel produced, thus leading to enhanced later strength (Deboucha et al., 2015;

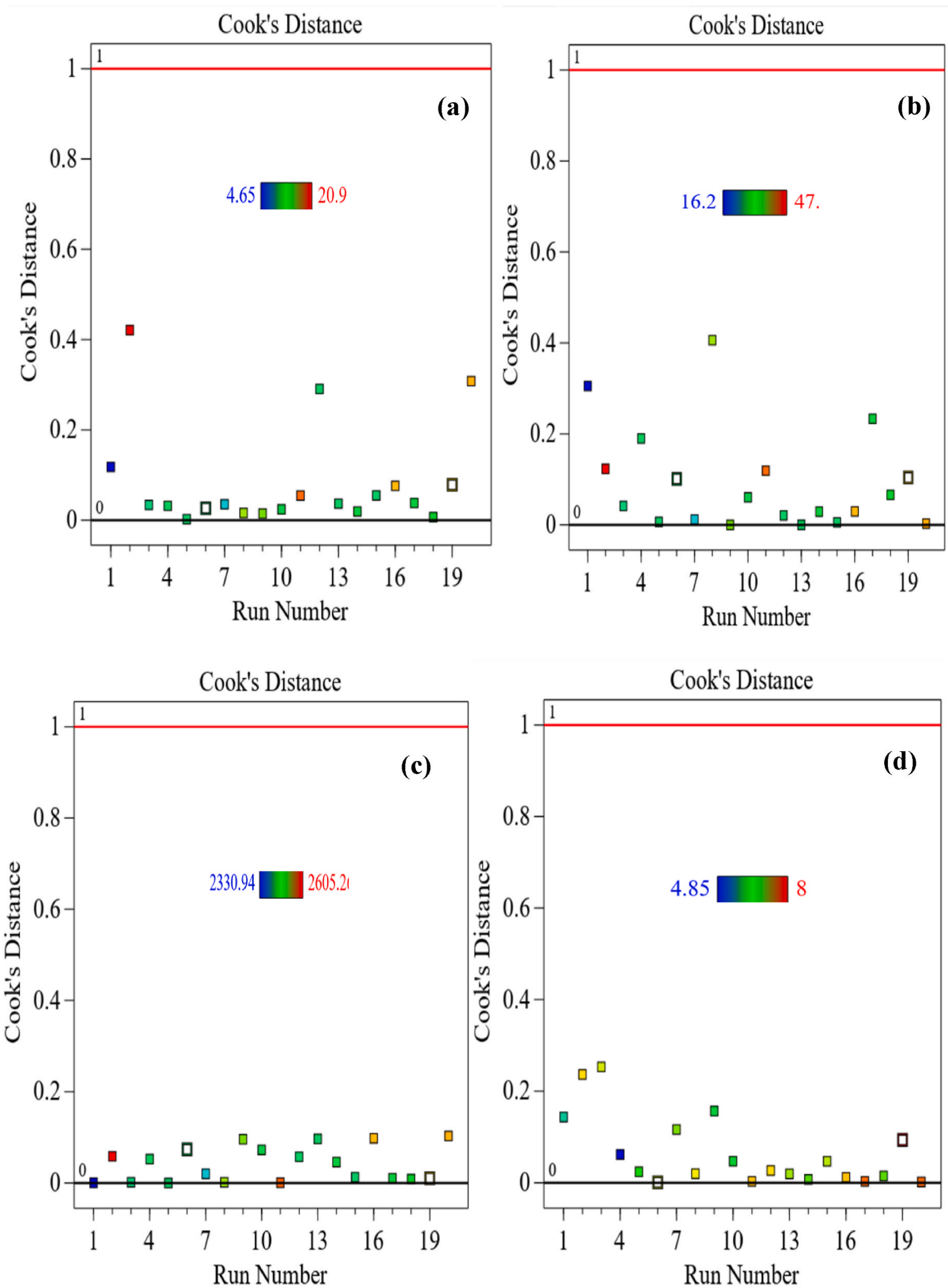

Fig. 5. Plot of cook's distance versus run number (a) 2-days compressive strength, (b) 28-days compressive strength, (c) fresh bulk density, and (d) flexural strength.

Chen et al., 2019; Zeyad et al., 2019).

The same observation as mortar containing the high content of blast furnace slag and natural pozzolana 30% and 20% respectively gives low early strength compared to the control mixture. However, the study found that the mortar specimens with a higher content of limestone have higher early compressive strengths than pozzolanic materials (Makhloufi et al., 2015). That is mainly due to the higher content of calcium hydroxide in limestone which can contribute occurrence of good early strength, but in the long term, the pozzolanic materials have higher compressive strength than the limestone blended cement composite materials. So, the employment of pozzolanic materials both artificial and natural like blast furnace slag and bentonite respectively,

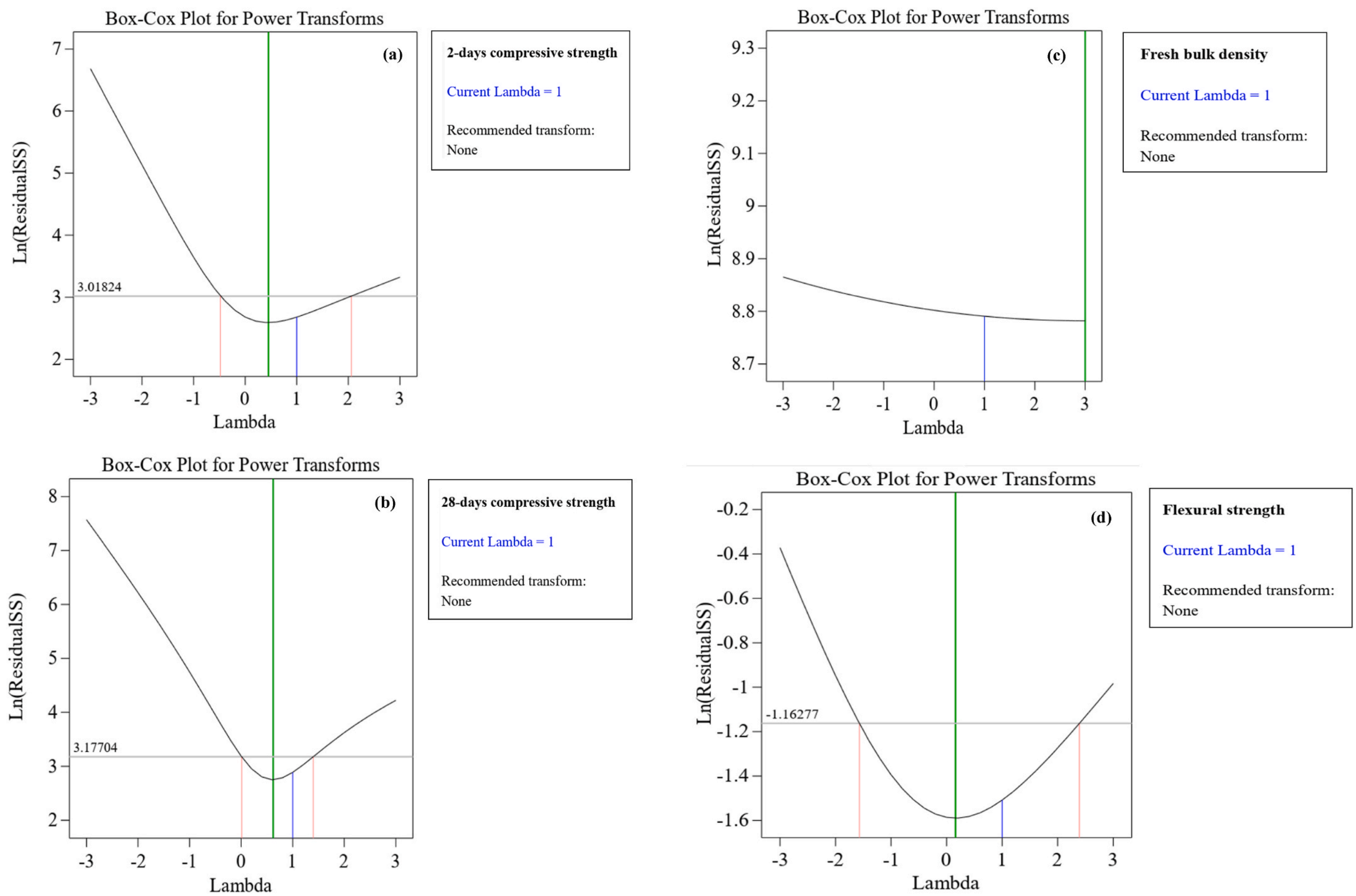

Fig. 6. Box cox plot for power transform (a) 2-days compressive strength, (b) 28-days compressive strength, (c) fresh bulk density, and (d) flexural strength.

can play an important role in the compressive strength of limestone cement composite materials at a late age.

3.4.3. *Fresh bulk density*

As presented in Fig. 10(a and b) the employment of blast furnace slag increases the density of the cement composite materials. However, increasing the content of bentonite substitution significantly reduces the fresh bulk density of the cement composite materials which is mostly due to most natural pozzolanic materials have lower specific density than Ordinary Portland cement and as the result of the dilution effect of pozzolanic materials (Saraya, 2014).

However, as shown in Fig. 10(c–f), increasing the replacement of active limestone with increasing the bentonite substitution significantly reduces the fresh bulk density. Besides these, employing 20% of raw bentonite gives the lowest fresh bulk density than all replacements. This is due to the pozzolanic materialsmainly can react and consume hydrated calcium hydroxide $Ca(OH)_2$ and produce the hydrated calcium silicate, which is responsible for the bulk density of hydrated cement pastes (Saraya, 2014). However, the employment of blast furnace slag isolately can significantly increase the fresh bulk density compared to all sampled mixtures, which is maybe due to the used slag has a higher density than cement particles. Hence, blinding blast furnace slag with bentonite or active limestone is beneficial to reduce its fresh bulk density while at the same time improving the strength of cement composite materials.

3.4.4. *Flexural strength*

As shown in Fig. 11(a–f) the employment of blast furnace slag increases the flexural strength more than bentonite replacement, hence, the addition of blast furnace slag by 20% has higher flexural strength than the employment of bentonite by 20% in cement composite materials. Therefore, increasing both substitutions up to 20% significantly reduces the flexural strength of cementing materials, which because raw bentonite may need activation since mostly bentonite exists in a consolidated forms (Reddy and Reddy, 2021). However, blending the blast furnace slag and active limestone by 10% have achieved the highest flexural strength than all sampled mixtures.

3.5. *Numerical optimization for replacement of different supplementary cementitious materials using CCD-RSM*

The CCD-RSM can perform numerical optimization through established goals to reach the best performance from the desired output.

3.5.1. *Optimized value selection criteria*

The optimization criteria was employed as presented in Table 7, which is to achieve the maximum compressive and flexural strength with a minimum fresh bulk density of the investigated different supplementary cementitious materials replacement are “in range”. Depending on the selected criteria shown in Table 7, 54 solutions were generated which consists of different replacement of supplementary cementitious materials dose that can give various response values. So, from 54 different optimum generated values one is selected based on; i) the highest strength and lower fresh bulk density, ii) higher sum of blast furnace slag, bentonite, and limestone dose which can replace more and reduces production cost and environmental pollution raising due to ordinary Portland cement production, and iii) higher desirability of the suggested value which means the highest desirability indicates the best combination between the predicted and actual values (Myers and Montgomery, 2016). Hence, the optimum values of factor were selected

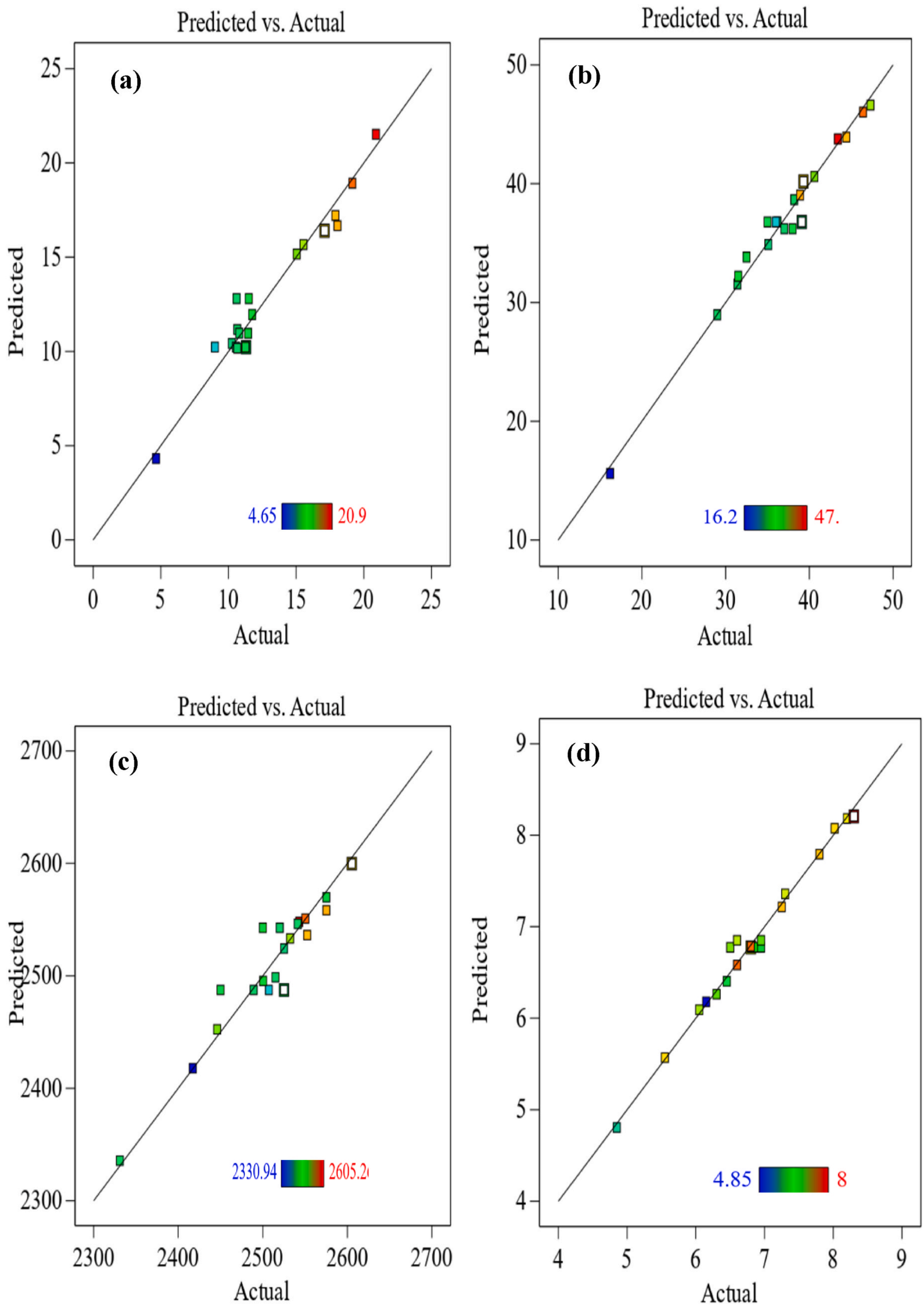

Fig. 7. Plot of predicted versus actual values (a) 2-days compressive strength, (b) 28-days compressive strength, (c) fresh bulk density, and (d) flexural strength.

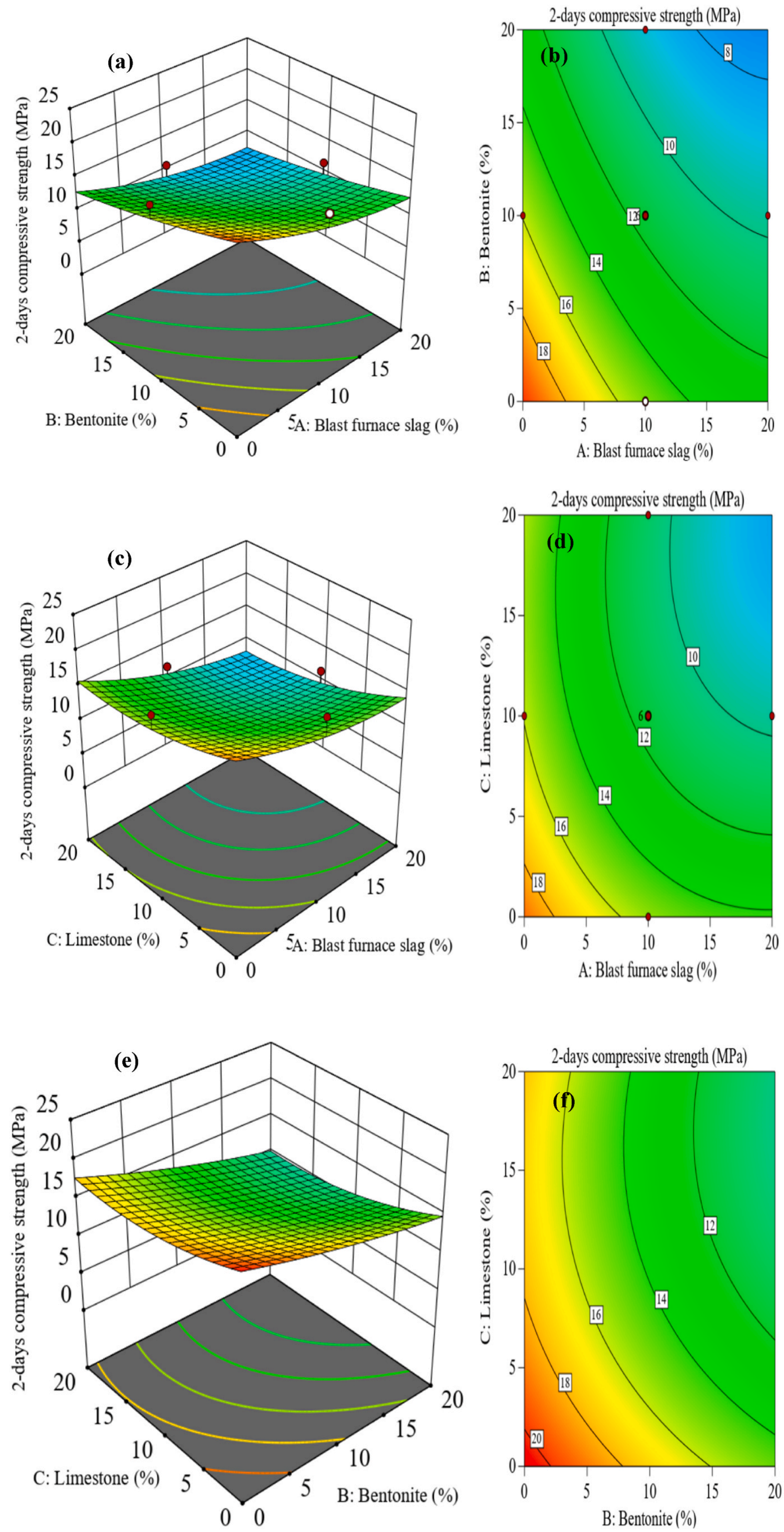

Fig. 8. 3D surface response and contour plots of interaction model terms for 2-days compressive strength response variable.

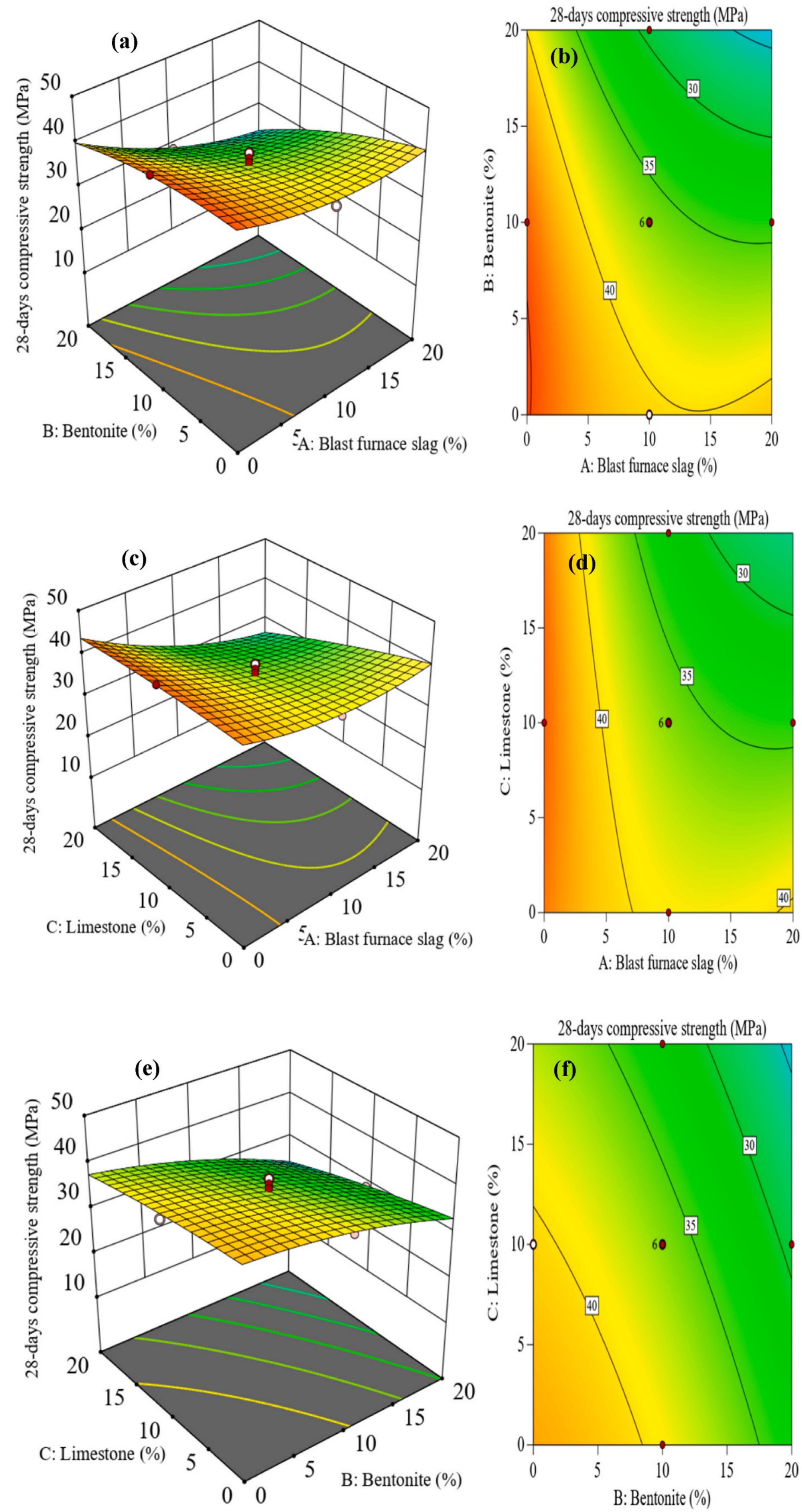

Fig. 9. 3D surface response and contour plots of interaction model terms for 28-days compressive strength response variable.

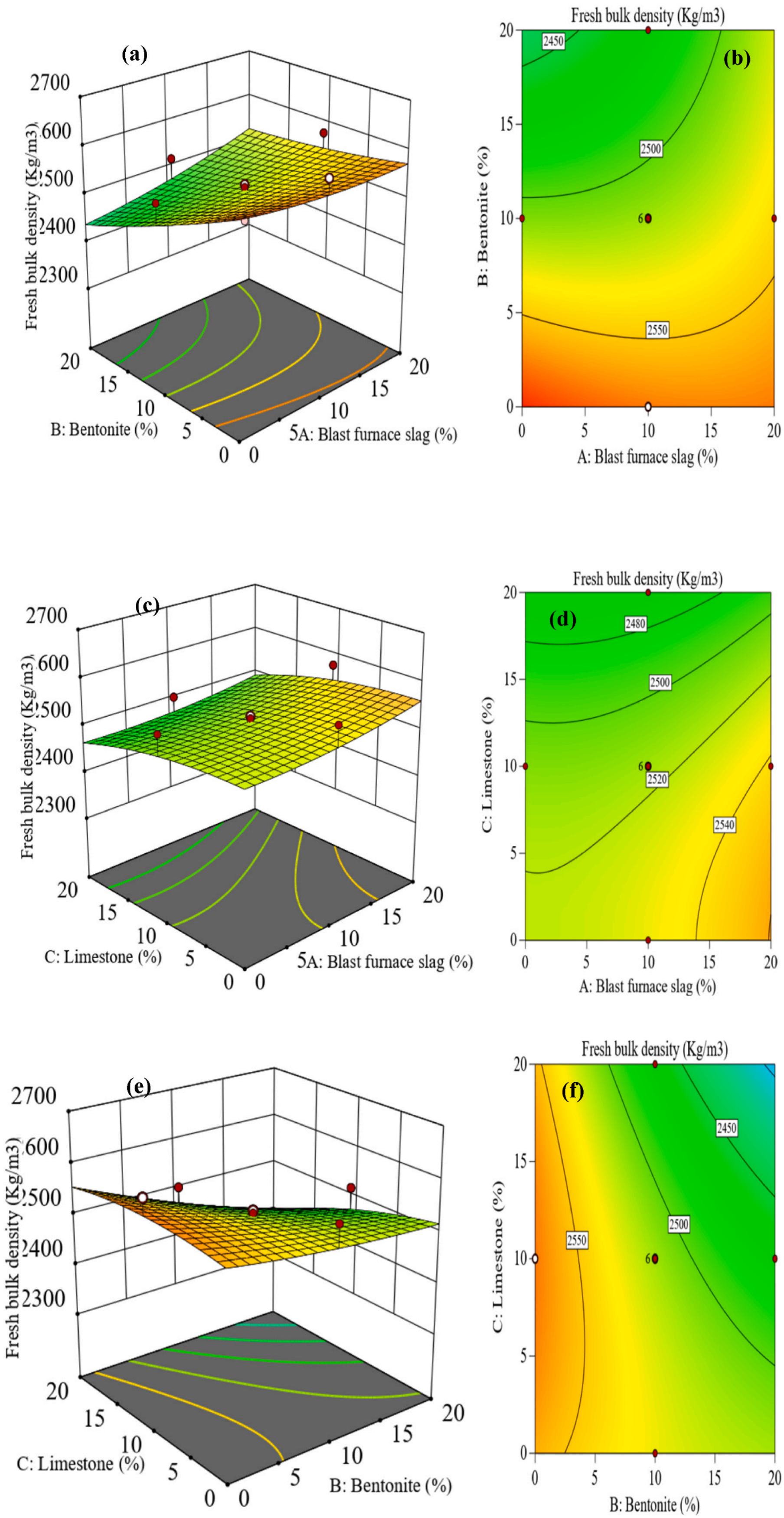

Fig. 10. 3D surface response and contour plots of interaction model terms for fresh bulk density response variable.

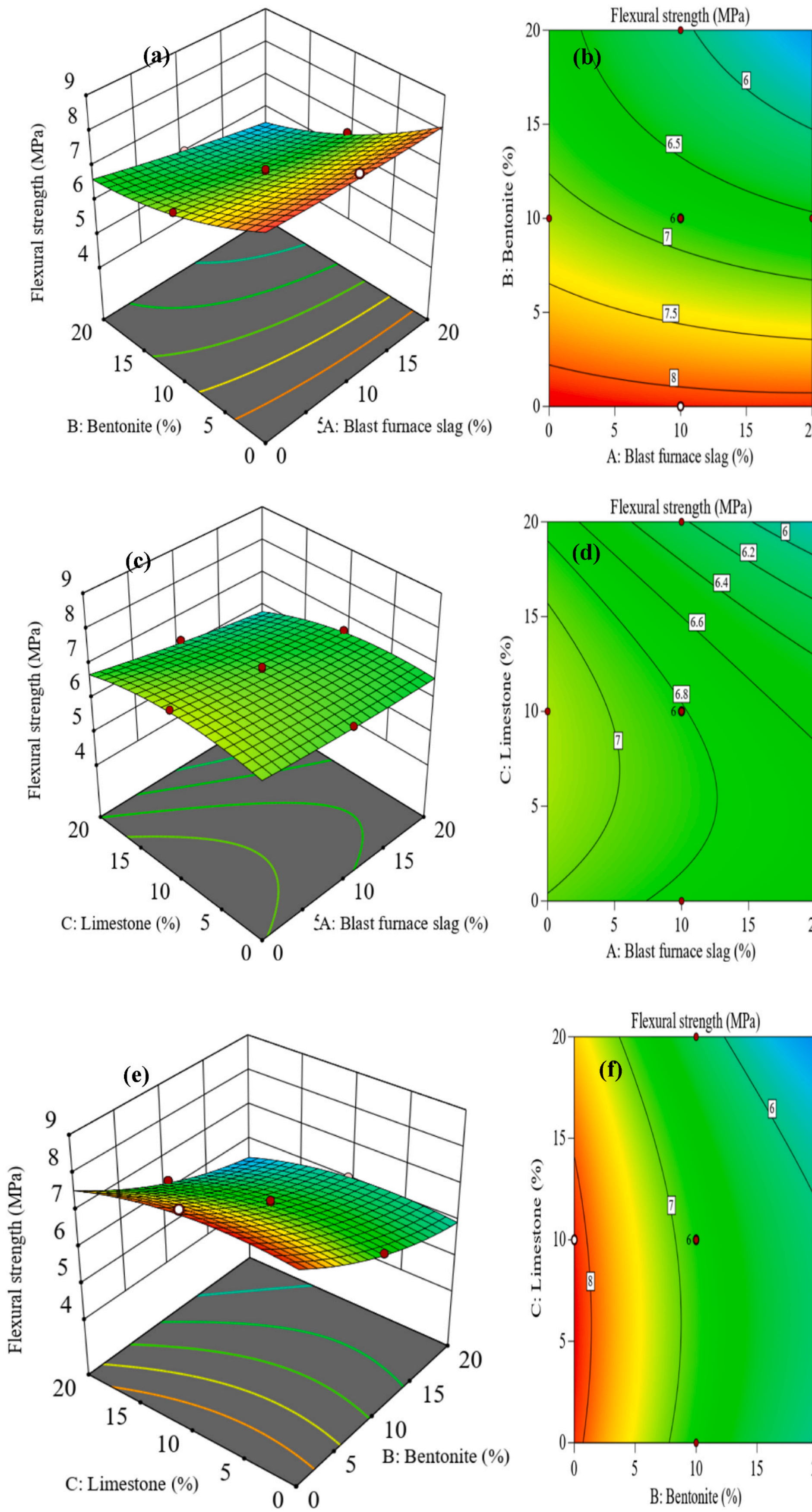

Fig. 11. 3D surface response and contour plots of interaction model terms for flexural strength response variable.

Table 7
Optimization constraints for all factor and response variables.

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
A: Blast furnace slag dose	Is in range	5.000	20.000	1.000	1.000	3.000
B: Bentonite dose	Is in range	5.000	20.000	1.000	1.000	3.000
C: limestone dose	Is in range	5.000	20.000	1.000	1.000	3.000
Compressive strength at 2days	Maximize	4.65	20.9	1.000	1.000	3.000
Compressive strength at 28days	Maximize	16.2	47.3	1.000	1.000	3.000
Fresh bulk density	Minimize	2330.94	2605.26	1.000	1.000	3.000
Flexural strength at 28days	Maximize	4.85	8.3	1.000	1.000	3.000

for the blast furnace slag 1.01%, bentonite 5.296%, and limestone 20% can give 17.0701 MPa, 43.8351 MPa, 2514.54 kg/m^3, and 7.1271 MPa of 2-days compressive strength, 28-days compressive strength, fresh bulk density, and flexural strength respectively as shown in Fig. 12.

Generally, from all the results have observed that a mixture of different blended supplementary cementitious materials such as blast furnace slag, bentonite, and active limestone total addition of more than 26% replacement to the cement weight has improved compressive strength at 28 days and reduce the fresh bulk density of cement composite materials compared to the control mixture without any calcination temperature. However, the cement clinker requires calcination at 1450 °C (Taylor-Lange et al., 2015); that shows the various supplementary cementitious materials designed by this study, not only improve the performance of cementing materials, but also highly reduces production cost, CO_2 emission, and energy consumption of ordinary Portland cement production.

3.6. Post-analysis of quadratic models and optimization results validation

For all responses the developed model was validated experimentally to check their practical applicability by inputting the optimum value suggested by the model blast furnace slag 1.01%, bentonite dose 5.3%, and active limestone 20% was evaluated experimentally as it can give the optimum numerical value from the model or near up to ±5% error. The error was calculated as shown in Adamu et al. (2022) to find the variation between the model-suggested values and the experimental responses as Eq. (7).

$$E = \frac{(x - y)}{x} * 100 \quad (7)$$

where E is the percentage of error, x and y represent the experimental and model response.

A similar method of error finding was used for all response variables. Hence, as presented in Table 8, the value of experimental validation was checked as within the 95% confidence level to the optimized results found 17.261 MPa, 43.811 MPa, 2529.73 kg/m^3, and 7.150 MPa respectively to 2-days compressive strength, 28-days compressive strength, fresh bulk density, and flexural strength which is very close to the optimum predicted value by CCD-RSM that confirms the validity and accuracy of the quadratic model.

4. Conclusions

The present study modelled and optimized the effects of blast furnace slag, bentonite, and active limestone replacement by 0 to 20% on physical and mechanical properties of cement composite materials using the statistical method CCD-RSM and the following have been concluded.

- The employment of blast furnace slag, bentonite, and active limestone to the cement composite materials generally lessens the early strength compared to the control mixture. However, from the three additives active limestone more improves the early strength.
- The addition of blast furnace slag and active limestone by 20% significantly improves the 28 days compressive strength while employing raw bentonite by 20% reduced compressive strength by 6.45% compared to the control mixture. However, blending raw bentonite with active limestone by half improves the compressive strength.
- Besides these, the substitution of bentonite and active limestone reduces the fresh bulk density and flexural strength than the control mixture. However, the employment of blast furnace slag increases

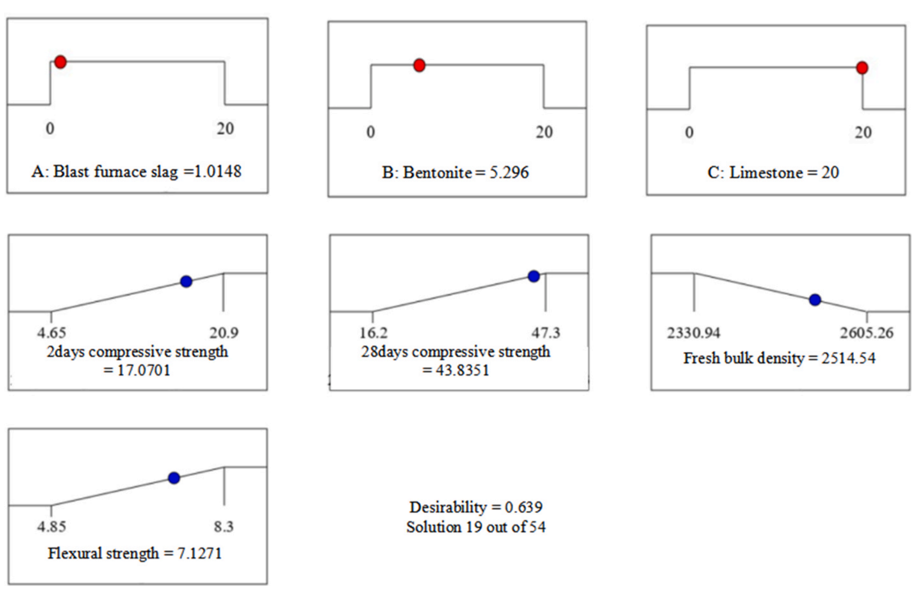

Fig. 12. Optimization ramps containing optimal experimental conditions for factor variables and maximized response variables.

Table 8
Verification of model prediction with the experimental data.

Response variables	CCD-RSM Optimum predicted value	95% lower bound to predicted value	Experimental value	95% upper bound to predicted value
2-days compressive strength (MPa)	17.0701	16.2166	17.261	17.9236
28-days compressive strength (MPa)	43.8351	41.6433	43.811	46.0268
Fresh bulk density (Kg/ m^3)	2514.5400	2388.81	2529.730	2640.2670
Flexural strength (MPa)	7.1271	6.7707	7.150	7.483

the fresh bulk density and flexural strength of cement composite materials. Also, blending the blast furnace slag and active limestone by half of 20% each most improved the flexural strength than all sampled mixtures.

Generally, the aim of the present study was to improve the strength and reduce the fresh bulk density of cementing materials to be effectively used in mass concrete production besides improving environmental pollution and production cost of ordinary Portland cement. So, the study optimized depending on the criteria of maximizing strength and minimizing fresh density and found the mix design of replacement; blast furnace slag 1.01%, raw bentonite 5.296%, and active limestone 20% that improves 28 days compressive strength and reduces fresh bulk density in addition to replacing more content than 54 different optimized design mix results and which is also validated with the experimental work. Finally, the authors recommend the future researchers to consider more than 28 days longer term performance of the optimized supplementary cementitious materials replacement in cementing materials for the effective use of blast furnace slag, bentonite, and limestone simultaneously in cement composite materials.

CRediT authorship contribution statement

Tsion Amsalu Fode: Writing – original draft, Validation, Methodology, Conceptualization. **Yusufu Abeid Chande Jande:** Writing – review & editing, Supervision. **Thomas Kivevele:** Writing – review & editing, Supervision.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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