



Modeling and optimization of calcined bentonite replacement in the mechanical and durability properties of mortar

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ABSTRACT

Currently, pozzolanic materials are mostly recommended to improve the properties of cement composite materials and reduce environmental pollution, challenging the world owing to ordinary Portland cement (OPC) production. Bentonite is mostly available natural pozzolana, however, extensive studies conducted on other clays like kaolin and some studies reported that bentonite exists in a consolidated form which requires heating activation methods. Therefore, it is essential to investigate the properties of bentonite in detail for its sustainable use, and it is novel to model and optimize the optimum bentonite calcination temperature and time for the best performance replacement in mortar. Hence, the present study investigates the optimum bentonite calcination temperature, calcination time, and replacement dose for mortar strength and free lime using the central composite design-response surface method (CCD-RSM). The mortar was prepared by replacing the calcined bentonite with cement weight with different values of the factor variables, bentonite dose, calcination temperature, and calcination time. Durability tests were conducted after 56 days. Thus, the results indicate that the selected model of response variables for compressive strength and free lime were significant, accurate, reliable, and had excellent fitness to the experimental work. Hence, CCD-RSM predicted the optimum for independent factors of bentonite dose 19.99 %, calcination temperature 799.99 °C, and calcination time 135.04 min and experimentally validated, which improved the strength by 24.94% and reduced free lime by 3.08% compared to the control mortar, besides reducing CO₂ emissions compared to OPC production, which requires 1450 °C. Furthermore, the optimized bentonite replacement parameters have highly enhanced durability in different environments such as water, acids, salt, and elevated temperature compared to the control mixture at the age of 56 days.

1. Introduction

Ordinary Portland cement is the most commonly fabricated product and is widely used worldwide after water (Scrivener et al., 2018). However, it creates environmental pollution because it consumes large amounts of energy and natural resources, and every ton of ordinary Portland cement releases equal ton of CO₂ to the environment (Hamada

et al., 2021). However, because of the inflation of population and development, the use of ordinary Portland cement (OPC) is increasing globally (Channa et al., 2022; Milović et al., 2022). Therefore, many researchers recommend the use of supplementary cementitious materials commonly reached in SiO₂, Al₂O₃, and Fe₂O₃ in concrete to reduce cement content, improve concrete properties, produce cost-effective cementitious material, and potentially decrease environmental

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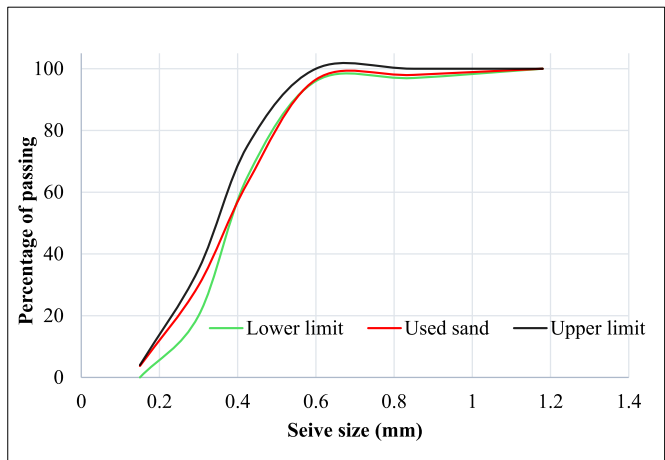


Fig. 1. Size distribution of used sand.

pollution of air owing to OPC production (Raghav et al., 2021). Supplementary cementitious materials (SCM) in cement composite materials have several benefits, including enhancing workability, reducing permeability, lowering the heat of hydration, and enhancing durability and strength (Küçükyıldırım and Uzal, 2014; Fezzioui et al., 2021; Ahmed, 2017; Nikhil K and Ajay A, 2015; Tebbal and El Abidine Rahmouni, 2019; Karthikeyan et al., 2015). Hence, it is highly used in one or another way in the construction industry commonly identified as pozzolana (Salamatpoor et al., 2018). Pozzolana is used as a cement substituent supplementary cementitious material with significant importance in the enhancement of concrete performance and lessening the consumption of cement while significantly reducing CO₂ emissions (Channa et al., 2022; Salamatpoor et al., 2018; Hossain et al., 2018; Ruan and Unluer, 2017; Deboucha et al., 2015; Khan et al., 2017; Park et al., 2021; Jaskulski and Daria, 2020; Costafreda et al., 2021; Firdous and Singh, 2021; Pacewska and Wilińska, 2020; Tayeh, 2018; Al-Hammood et al., 2021a; Taklymi et al., 2020; Trümer et al., 2019; Sriwattanapong Photisan, 2018; Zhang et al., 2019; Arrigoni et al., 2020; Cheng et al., 2018; Hu et al., 2020). Thus, this is an indication of SCM beneficiaries in encouraging the economic sustainability of the countries, since the employment of SCM in cement composite materials favors the reduction of energy expenditure and production cost of mortar

or concrete. Bentonite mostly fulfills pozzolanic properties and is rich in alumina-siliceous material, commonly categorized in the natural pozzolana (Anand and Kumari, 2022; Chamundeewari, 2012; Ahad et al., 2018). Calcination of natural pozzolana is beneficial for the activation of pozzolanic reactions and for creating a denser microstructure of concrete (Elbar et al., 2018). In particular, at higher calcination temperatures, bentonite exhibits appreciable pozzolanic properties (Adjei et al., 2021). The pozzolanic properties of bentonite contribute to several benefits in the construction industry as a partial substitution for cement for the improvement of concrete properties. In addition, kaolin is a well-known natural pozzolana that potentially differs from bentonite because of its higher Al content and white color, whereas most bentonite is gray (Fode et al., 2024a; Shafiq et al., 2015). Therefore, it was found that bentonite in cement composite materials can consume more portlandite than kaolin in concrete mixtures. This is because kaolin has high Al, which reduces the Ca/(Si + Al) ratios while increasing the cement replacement; consequently, it reduces the consumption of portlandite to form C-S-H compared to bentonite (Wei and Gencturk, 2019). However, extensive studies have been conducted on kaolin replacement in cementing materials compared to bentonite, although bentonite mostly exists in many countries. Hence, this took the attention of the present study authors for a detailed investigation of bentonite for the sustainability of construction materials.

The bentonite in cementitious materials is crucial for improving the durability and mechanical properties of cementing materials by increasing the resistance of mortar/concrete structures to acidic attacks (Haq, 2022; Rehman et al., 2019a; Xie et al., 2019; AKBAR et al., 2012; Andrade et al., 2021). Furthermore, the addition of bentonite to cementing materials reduces bleeding and enhances the cohesiveness of cementitious materials with low-intensity levels (Yu et al., 2013). Generally, employing bentonite in construction materials is a good option for reducing environmental pollution and technical and financial aspects compared to conventional mortar or concrete (Lima-Guerra et al., 2014; Ali et al., 2012; Mushtaq et al., 2022; Khan et al., 2022; Dunuweera and Rajapakse, 2018; Fode et al., 2023). However, bentonite exists naturally in a consolidated form without the required treatment or activation methods. Therefore, the heating of bentonite is crucial for the activation of its pozzolanic properties to be used as a supplementary material in cement composites (Rehman et al., 2019a). However, much

Table 1
For each run used calcined bentonite and OPC chemical composition.

Samples	Calcination		SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	K ₂ O	Na ₂ O	TiO ₂	P ₂ O ₅	SAF	LOI
	Temperature (°C)	Time (min)												
OPC	–	–	17.57	4.07	2.63	61.48	0.41	1.79	0.11	0.04	0.29	0.13	24.27	10.74
Run-1	200.00	180.00	52.85	12.34	6.53	3.67	5.42	0.08	5.27	3.08	0.87	0.21	71.73	8.73
Run-2	200.00	120.00	53.15	12.92	6.82	3.51	5.68	0.07	5.53	3.22	0.91	0.22	72.89	7.45
Run-3	500.00	150.00	53.96	13.12	6.95	3.67	5.79	0.07	5.57	3.24	0.92	0.22	74.03	5.67
Run-4	500.00	150.00	53.96	13.12	6.95	3.67	5.79	0.07	5.57	3.24	0.92	0.22	74.03	5.67
Run-5	500.00	150.00	53.96	13.12	6.95	3.67	5.79	0.07	5.57	3.24	0.92	0.22	74.03	5.67
Run-6	800.00	180.00	56.19	13.37	7.46	3.94	6.04	0.05	5.79	3.39	0.96	0.24	77.01	1.77
Run-7	800.00	120.00	55.34	13.49	7.23	5.55	5.94	0.14	5.71	3.30	0.96	0.25	76.06	1.85
Run-8	800.00	120.00	55.34	13.49	7.23	5.55	5.94	0.14	5.71	3.30	0.96	0.25	76.06	1.85
Run-9	800.00	180.00	56.19	13.37	7.46	3.94	6.04	0.05	5.79	3.39	0.96	0.24	77.01	1.77
Run-10	200.00	120.00	53.15	12.92	6.82	3.51	5.68	0.07	5.53	3.22	0.91	0.22	72.89	7.45
Run-11	200.00	180.00	53.25	13.92	6.92	3.50	5.58	0.07	5.43	3.22	0.91	0.22	74.09	7.45
Run-12	500.00	150.00	53.96	13.12	6.95	3.67	5.79	0.07	5.57	3.24	0.92	0.22	74.03	5.67
Run-13	500.00	150.00	53.96	13.12	6.95	3.67	5.79	0.07	5.57	3.24	0.92	0.22	74.03	5.67
Run-14	500.00	110.52	53.65	13.08	6.88	3.84	5.75	0.07	5.56	3.25	0.92	0.22	73.61	6.75
Run-15	500.00	150.00	53.98	13.12	7.03	3.99	5.78	0.07	5.58	3.24	0.93	0.22	74.13	5.13
Run-16	500.00	189.48	53.40	12.97	6.95	3.77	5.74	0.07	5.52	3.27	0.92	0.24	73.31	6.37
Run-17	500.00	150.00	53.96	13.12	6.95	3.67	5.79	0.07	5.57	3.24	0.92	0.22	74.03	5.67
Run-18	894.82	150.00	56.47	13.49	7.40	4.25	6.15	0.14	5.90	3.39	0.96	0.24	77.36	0.68
Run-19	500.00	150.00	53.98	13.12	7.03	3.99	5.78	0.07	5.58	3.24	0.93	0.22	74.13	5.13
Run-20	105.18	150.00	51.62	12.21	6.51	3.64	5.38	0.07	5.23	3.06	0.87	0.22	70.36	10.33

SAF= Sum (SiO₂, Al₂O₃, Fe₂O₃).

Table 2

Mortar mix design for all runs.

Experimental runs	Space type	OPC Cement (g)	Bentonite			Sand (g)	W/C
			A: Replacement dose (g)	B: Calcination temperature (°C)	C: Calcination time (Min)		
1	Factorial	400	100	200.00	180.00	1375	0.485
2	Factorial	400	100	200.00	120.00	1375	0.485
3	Center	437.5	62.5	500.00	150.00	1375	0.485
4	Center	437.5	62.5	500.00	150.00	1375	0.485
5	Center	437.5	62.5	500.00	150.00	1375	0.485
6	Factorial	475	25	800.00	180.00	1375	0.485
7	Factorial	475	25	800.00	120.00	1375	0.485
8	Factorial	400	100	800.00	120.00	1375	0.485
9	Factorial	400	100	800.00	180.00	1375	0.485
10	Factorial	475	25	200.00	120.00	1375	0.485
11	Factorial	475	25	200.00	180.00	1375	0.485
12	Center	437.5	62.5	500.00	150.00	1375	0.485
13	Center	437.5	62.5	500.00	150.00	1375	0.485
14	Axial	437.5	62.5	500.00	110.52	1375	0.485
15	Axial	388.15	111.85	500.00	150.00	1375	0.485
16	Axial	437.5	62.5	500.00	189.48	1375	0.485
17	Center	437.5	62.5	500.00	150.00	1375	0.485
18	Axial	437.5	62.5	894.82	150.00	1375	0.485
19	Axial	486.85	13.15	500.00	150.00	1375	0.485
20	Axial	437.5	62.5	105.18	150.00	1375	0.485

Table 3

Variables with their level.

Response factors	Symbol	Units	Level of factor		
			Lower	Middle	Higher
Bentonite dose	A	%	5	7.5	20
Bentonite calcination temperature	B	°C	200	500	800
Calcination time	C	Min	120	150	180

of the calcination of montmorillonite clay such as bentonite can be affected by the high calcination temperature, which depends on the particle size distribution and the specific surface area of the clay (Fernandez et al., 2011).

Laidani et al. (2022) and Taylor-Lange et al. (2015) studied bentonite calcination and found maximum bentonite pozzolanic reactivity achieved in between calcination temperatures of 650–900 °C, which can alter bentonite crystallization to amorphousness. However, the study reported that above 900 °C, the reactivity rapidly decreased because of bentonite re-crystallization. Reddy and Reddy (2021) investigated bentonite employment in concrete by raw and calcined at 700 °C and 800 °C to determine its effect on the mechanical and physical properties of concrete and found that calcined bentonite provides higher performance than raw bentonite in concrete. In addition, Fode et al. (2024b) studied different bentonite replacement doses at 5, 10, 15, and 20 % to the weight of cement and found 5% replacement of calcined bentonite significantly improved flexural strength.

Various researchers have investigated the effect of a limited constant range of bentonite calcination temperatures and replacement doses on concrete and mortar properties. However, to the best of our knowledge, no studies have modeled and optimized the effects of calcination temperature, calcination time, and replacement dose on the mechanical properties of mortar/concrete. Therefore, the present study investigates the optimum bentonite calcination temperature, calcination time, and replacement dose to employ in mortar using multivariate experimental designs known as response surface methodology (RSM)- Design Expert (Stat-Ease, version 13). This statistical method optimizes experimental runs and can reduce experimental inconsistency and excessive time demand. In addition, this statistical experimental procedure can be used as an effective tool and valuable in optimizing the content of bentonite to achieve high performance of mortar that can provide high strength and low free lime, which can reduce expansion and cracks. In addition,

the optimized bentonite replacement parameters were experimentally verified and assessed in different adverse environments.

2. Materials and methods

2.1. Materials

The raw bentonite was extracted from Arusha, Tanzania, which has a mineral composition of quartz, montmorillonite, illite, and calcium carbonate, as described in the X-ray diffraction results of our previous study (Fode et al., 2024b). Bentonite calcination was conducted after grinding the bentonite sample that could pass 45 µm, which was calcined as governed by the CCD-RSM at different input calcination temperatures from 200 to 800 °C and calcination time of 120–180 min at a fixed calcination rate of 5 °C/min, as reported in previous studies (Ali et al., 2012). That is, bentonite was heated at 200 °C and most other authors used 800 °C for 2–3 hours and found that bentonite had the highest reactivity (Fode et al., 2024b; Laidani et al., 2020; Rehman et al., 2019b; Keke et al., 2022). However, the CCD-RSM was observed below and above the lower and upper limits. In addition, different substitutions of the calcined bentonite dose from 5 to 20% to the ratio of cement were considered, based on our previous study (Fode et al., 2024c). Washed river sand was used, and its gradation is shown in Fig. 1. The chemical compositions of the different calcined bentonite for each run and OPC used are presented in Table 1, which shows that all the calcined bentonite in each run fulfills the requirements of ASTM C618 (A. C618, 2012) by having SiO₂, Al₂O₃, and Fe₂O₃ more than 70% of the overall composition. Distilled water was supplied by Tanga Cement PLC, where this study was conducted.

2.2. Sample preparation

The mortar samples were prepared using cement, sand, water, and 20 calcined bentonite samples with different replacement doses, calcination temperatures, and calcination times as presented in Table 2. The ordinary Portland cement (32.5 MPa) and washed river sand used for sand gradation as shown in Section 2.1. Therefore, the mixing process was performed using a laboratory mixing machine JJ-5 model. Fresh mortar as ASTM C109 (ASTM C109/C109M-02, 2002), was used to prepare mortar cubes of size 50 × 50 × 50 mm³. A laboratory shaking table Model HC-3255.4F was used to compact the mortar specimens. All cast mortar samples were kept in a humidity chamber for 24 hours to prevent moisture gain or loss in the mortar samples. Subsequently, the

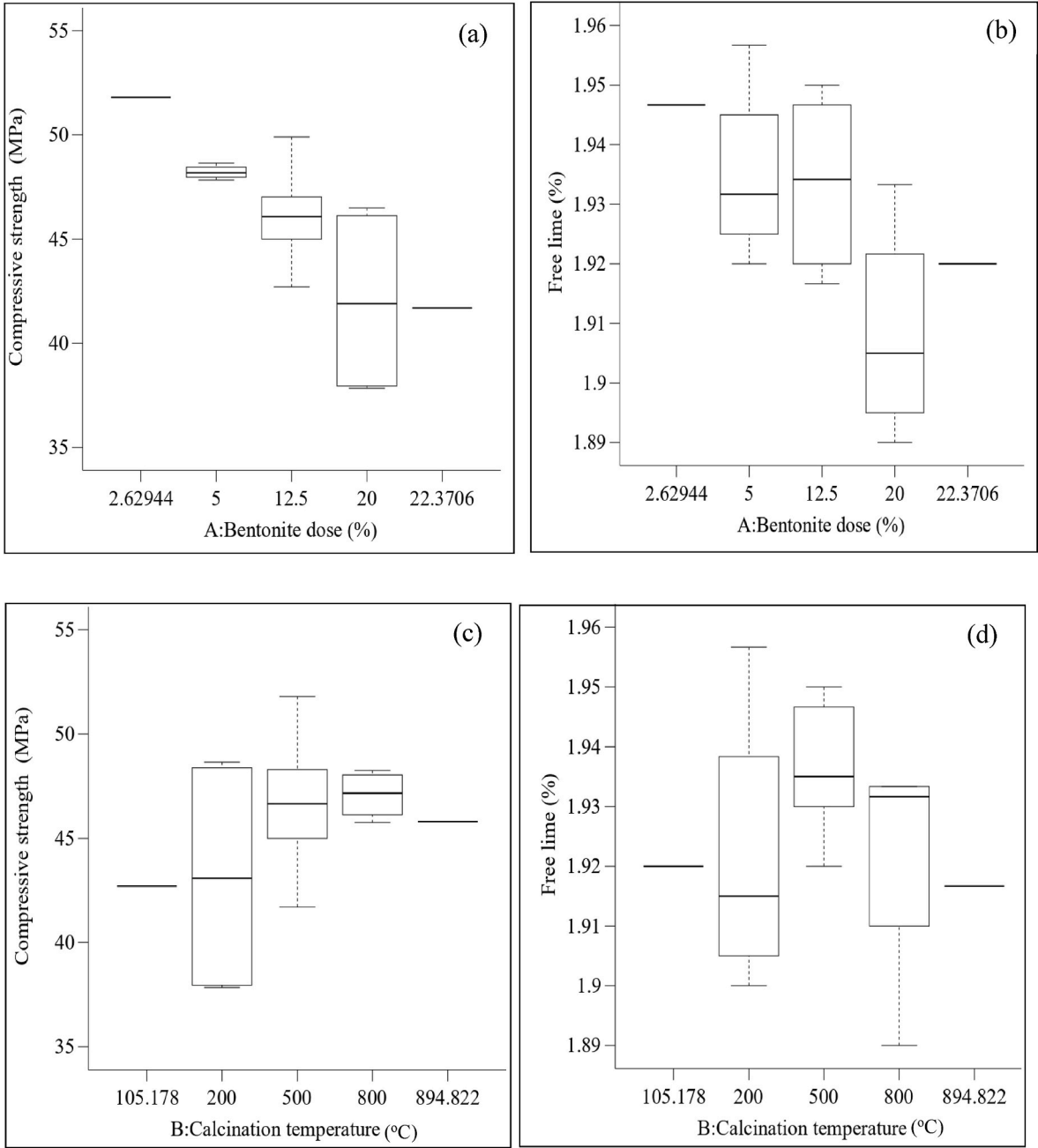


Fig. 2. The box plot of experimental runs response variables (compressive strength and free lime) versus independent factors (a, b) bentonite dose, (c, d) calcination temperature, and (e, f) calcination time, respectively.

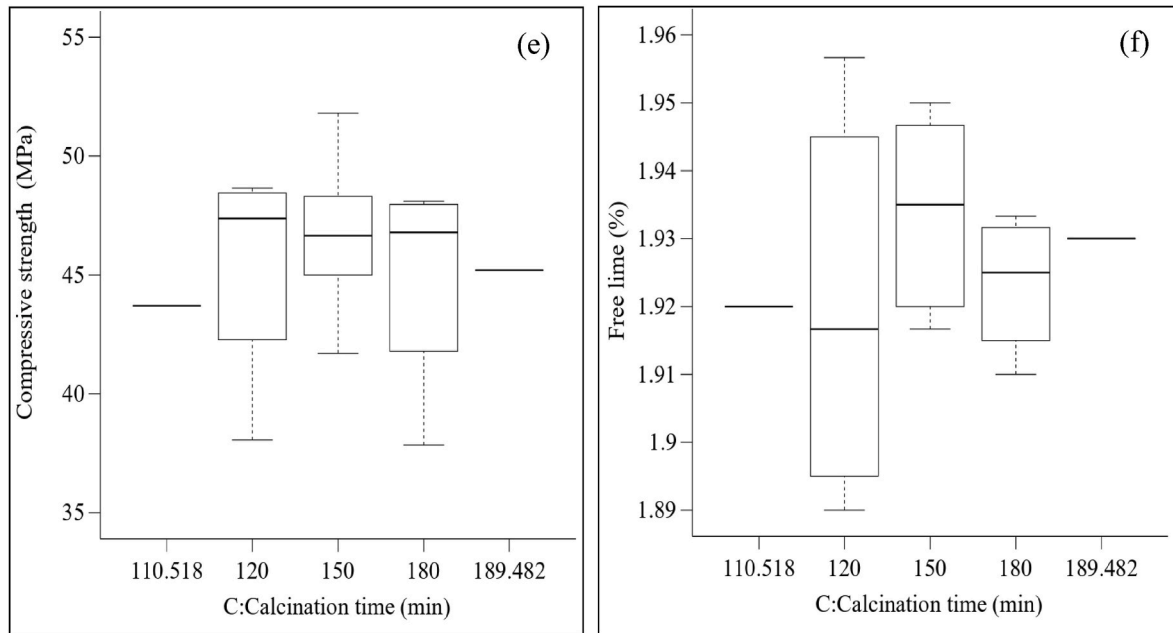


Fig. 2. (continued).

Table 4

The coded form of bentonite substitution in the CCD matrix with respective response variables.

Runs	Space type	Factor 1	Factor 2	Factor 3	Response 1	Response 2
		Bentonite dose (%)	Calcination temperature (°C)	Calcination time (min)	Compressive strength (MPa)	Free lime (%)
1	Factorial	1.00	−1.00	1.00	37.84	1.91
2	Factorial	1.00	−1.00	−1.00	38.05	1.90
3	Center	0.00	0.00	0.00	45.00	1.95
4	Center	0.00	0.00	0.00	49.90	1.94
5	Center	0.00	0.00	0.00	47.02	1.95
6	Factorial	−1.00	1.00	1.00	47.83	1.93
7	Factorial	−1.00	1.00	−1.00	48.25	1.93
8	Factorial	1.00	1.00	−1.00	46.50	1.89
9	Factorial	1.00	1.00	−1.00	45.75	1.93
10	Factorial	−1.00	−1.00	−1.00	48.65	1.96
11	Factorial	−1.00	−1.00	1.00	48.10	1.92
12	Center	0.00	0.00	0.00	48.30	1.95
13	Center	0.00	0.00	0.00	46.35	1.94
14	Axial	0.00	0.00	−1.32	43.70	1.92
15	Axial	1.32	0.00	0.00	41.70	1.92
16	Axial	0.00	0.00	1.32	45.20	1.93
17	Center	0.00	0.00	0.00	46.95	1.93
18	Axial	0.00	1.32	0.00	45.80	1.92
19	Axial	−1.32	0.00	0.00	51.80	1.95
20	Axial	0.00	−1.32	0.00	42.70	1.92

Table 5

Different model fitness summary for compressive strength.

Source	Sequential <i>p</i> -value	Lack of fit <i>p</i> -value	Adjusted R ²	Predicted R ²	Remark
Linear	0.01	0.28	0.60	0.36	
2FI	0.06	0.46	0.72	0.42	
Quadratic	0.03	0.91	0.85	0.73	Suggested
Cubic	0.78	0.96	0.81	0.84	Aliased

Table 6

Different model fitness summary for free lime.

Source	Sequential <i>p</i> -value	Lack of fit <i>p</i> -value	Adjusted R ²	Predicted R ²	Remark
Linear	0.14	0.03	0.16	−0.34	
2FI	0.07	0.05	0.39	−0.69	
Quadratic	0.01	0.72	0.89	0.75	Suggested
Cubic	0.71	0.42	0.86	−7.88	Aliased

mortar specimens were de-molded and placed in a water-curing chamber at room temperature.

As shown in Table 2, the configuration difference of 20 experimental runs was generated statistically using the CCD-RSM with respect to the response variables: Y₁, compressive strength (MPa), and Y₂, free lime

(%) that used for the modeling and optimization processes, which have eight factorial, six center, and six axial points of various response variables.

Table 7

Fit statistics for compressive strength and free lime.

Statistics	Compressive strength	Free lime
Standard deviation (Std. Dev.)	1.40	0.01
Mean	45.77	1.93
Percentage coefficient of variance (C.V%)	3.05	0.28
Correlation coefficient (R ²)	0.93	0.95
Adjusted R ²	0.85	0.92
Predicted R ²	0.73	0.86
Adequate precision	12.79	13.98

Table 8

ANOVA of the quadratic model developed for compressive strength.

Sources	Sum of squares	df	Mean square	F-value	p-value	Remark
Block	0.80	1	0.80	–	–	Significant
Model	223.02	9	24.78	12.71	0.01	
A- Bentonite dose	125.81	1	125.81	64.53	<0.01	
B- Calcination temperature	34.08	1	34.08	17.48	0.01	
C- Calcination time	0.01	1	0.01	0.01	0.99	
AB	36.23	1	36.23	18.58	0.01	
AC	0.00	1	0.00	0.00	0.99	
BC	0.02	1	0.02	0.01	0.92	
A ²	0.07	1	0.07	0.04	0.85	
B ²	12.28	1	12.28	6.30	0.03	
C ²	10.25	1	10.25	5.26	0.05	
Residual	17.55	9	1.95	–	–	Not significant
Lack of Fit	4.50	5	0.91	0.28	0.91	
Pure Error	13.04	4	3.26	–	–	
Cor Total	241.37	19	–	–	–	

Table 9

ANOVA of the quadratic model developed for free lime.

Sources	Sum of squares	df	Mean square	F-value	p-value	Remark
Block	0.00	1	0.00	–	–	Significant
Model	0.01	9	0.01	23.69	<0.01	
A-Bentonite dose	0.01	1	0.01	69.81	<0.01	
B- Calcination temperature	1.7E-06	1	1.7E-06	0.07	0.80	
C- Calcination time	0.01	1	<0.01	2.43	0.15	
AB	0.01	1	<0.01	3.53	0.09	
AC	0.01	1	<0.01	43.31	<0.01	
BC	0.01	1	<0.01	22.09	<0.01	
A ²	0.01	1	<0.01	2.39	0.16	
B ²	0.01	1	<0.01	36.77	<0.01	
C ²	0.01	1	<0.01	16.45	<0.01	
Residual	0.01	9	0.00	–	–	Not significant
Lack of Fit	0.01	5	0.00	0.38	0.84	
Pure Error	0.01	4	0.00	–	–	
Cor Total	0.01	19	–	–	–	

2.3. Methods

According to ASTM C109/C109M-02 (2002), a 50 × 50 × 50 mm³ mold was used for the compressive strength test of the mortar samples using a 1543–08 Zwick-Roell digital compressive strength testing machine at a rate of 1800 N/s. For all 20ⁿ runs, the compressive strength response was conducted at a mortar age of 28 days. A total of 63 samples with a control mortar mixture were cast, with three for each batch used in the compressive strength test, and the average results of each run were recorded. For the experimental validation, a 50 × 50 × 50 mm³

cube of the optimized value was recasted to validate the selected software optimum value based on the experimental results. Subsequently, the optimized bentonite replacement parameters were evaluated in different adverse environments to verify the durability of the optimized bentonite compared to the control mixture. The free lime was investigated for all 20 runs and for the optimized sample using the iteration method following ISO 4032-1985.

2.4. Experimental design using CCD-RSM

The response surface method (RSM) is a statistical method that optimizes experimental runs and is rapid and effective for investigating the effects of various independent factors. In addition, comparing conventional methods to multivariate experimental designs has many key advantages, including low cost, fast implementation, and concurrent finding of the effects of multiple independent factors on a response variable (Cai et al., 2013). Therefore, the response surface method is employed to evaluate the effect of a set of experimental independent variables on a single or many response variables. Hence, this method is often used to find the factor place that optimizes the response after identifying a “vital few” controllable factors and is usually good for the response surfaces suspected by a factorial experimental design (Coruh et al., 2015). In RSM, central composite design (CCD) is one of the best designs to provide an accurate and significant prediction of the response variables as well as the interactive consequences of the independent variables (Cai et al., 2013; Gour et al., 2022). The total experimental runs (N) of the RSM were calculated using Eq. (1), where K, C₀, and N are the process variables, center points, and required experimental runs (Fode et al., 2024a).

$$N = 2^K + 2K + C_0 \quad (1)$$

The CCD-RSM in Design Expert Software (State-Ease, version 13) consists of the interacting effects of three independent bentonite substitution variables, including bentonite dose (A), bentonite calcination temperature (B), calcination time (C), independent factors, their ranges, and the response variables listed in Table 3, taken from previous literature (Reddy and Reddy, 2021; Vijay and Achyutha Kumar Reddy, 2021).

2.5. Assessment of durability in different adverse environments

Evaluation of the durability of the optimized bentonite replacement in mortar is crucial for checking its life in different adverse environments. Therefore, the optimized bentonite replacement dose, calcination temperature, and calcination time given by CCD-RSM were verified by experimental methods; then assessed in 10% sodium chloride (NaCl), 5% hydrochloric acid (HCl), and nitric acid (HNO₃), each cured separately for 56 days. These effects were measured using a mortar cube of 50 mm³ for the optimized bentonite replacement and control mixture using the methods of strength and mass loss. The effect of mass loss by elevated temperature at 200 °C for 2 hours for both the optimized bentonite employment and control mix was conducted at 56 days of mortar specimen age. In addition, the water absorption of the optimized bentonite employment and control mix for the cubic mortar specimen was conducted at 56 days by drying the mortar specimen in an oven at 75 °C until it reached a constant mass. The samples were then placed in 5 mm deep water after covering all lateral parts of the cube with paraffin oil to prevent lateral moisture absorption (Deboucha et al., 2015).

3. Results and discussion

3.1. Cementitious materials strength and free lime assessment

Based on the CCD-RSM, three independent variables were examined: bentonite dose, bentonite calcination temperature, and calcination time

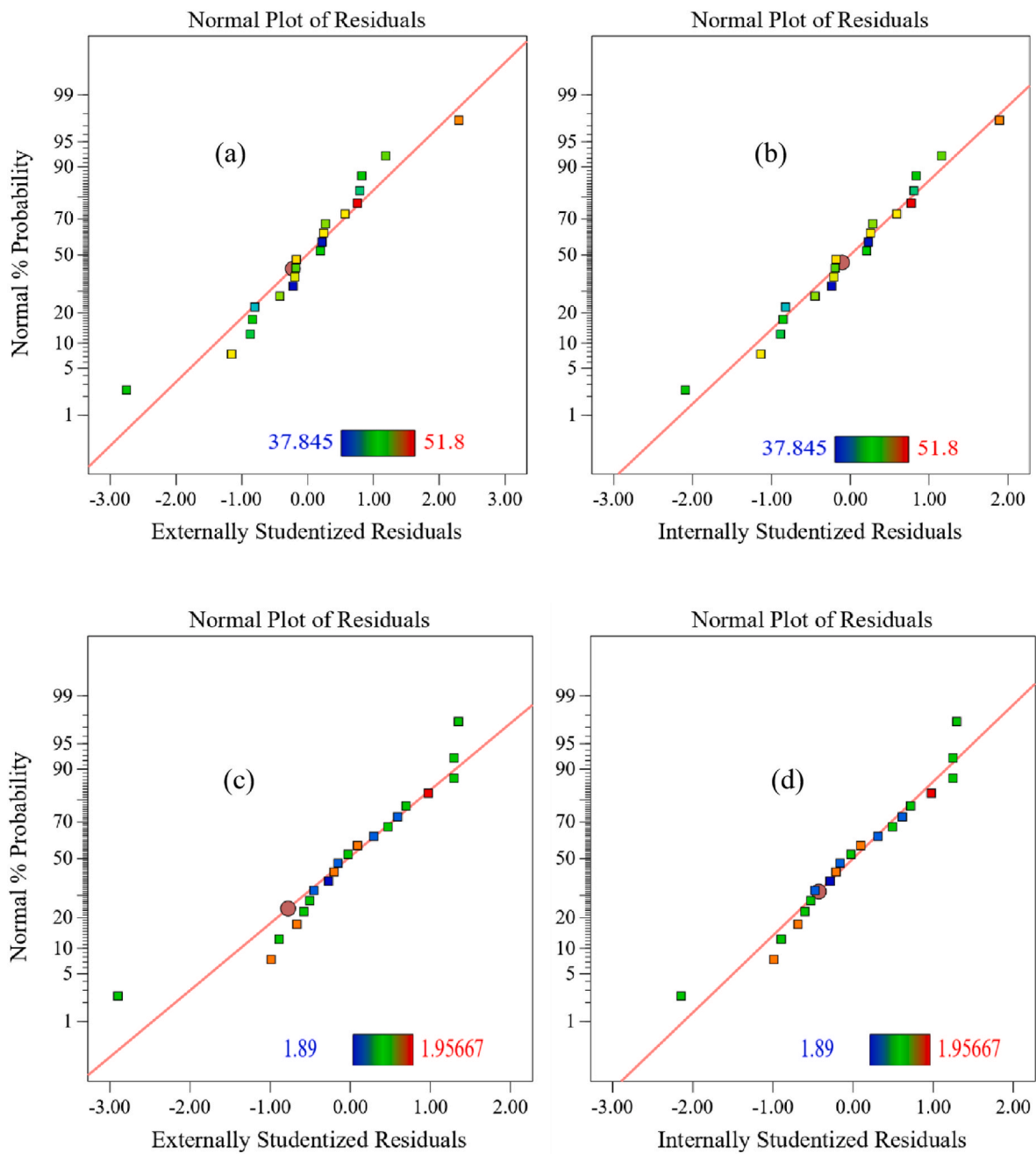


Fig. 3. Normal % probability plot versus external and internal studentized residuals for (a,b) compressive strength and (c,d) free lime.

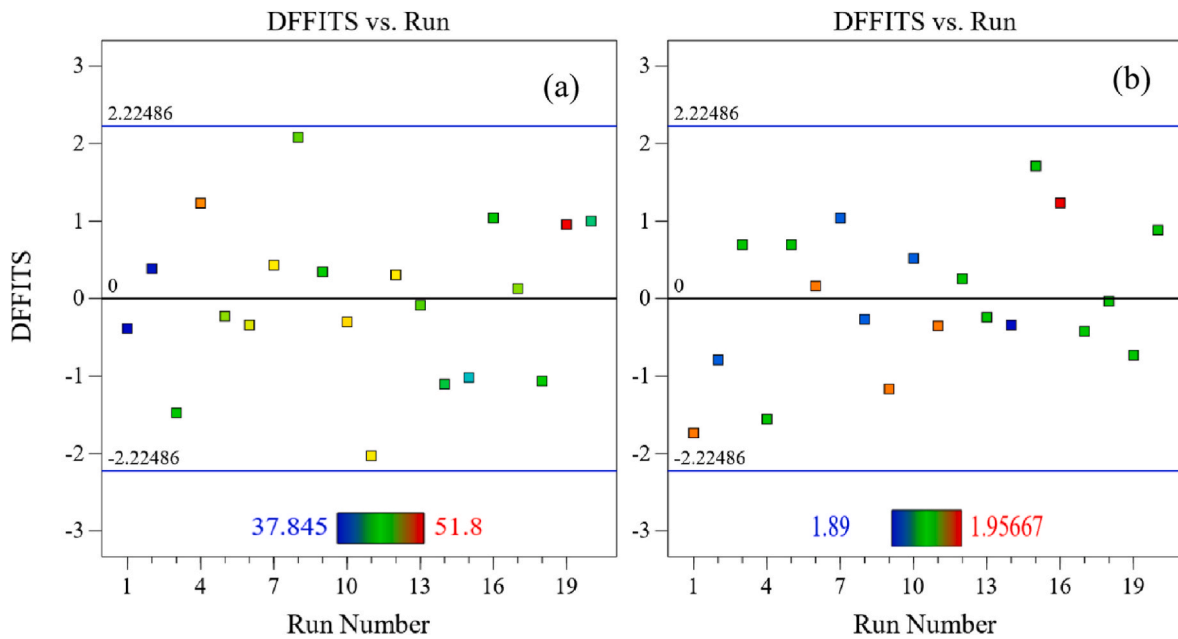


Fig. 4. Plot of DFFITS versus run number for (a) compressive strength and (b) free lime.

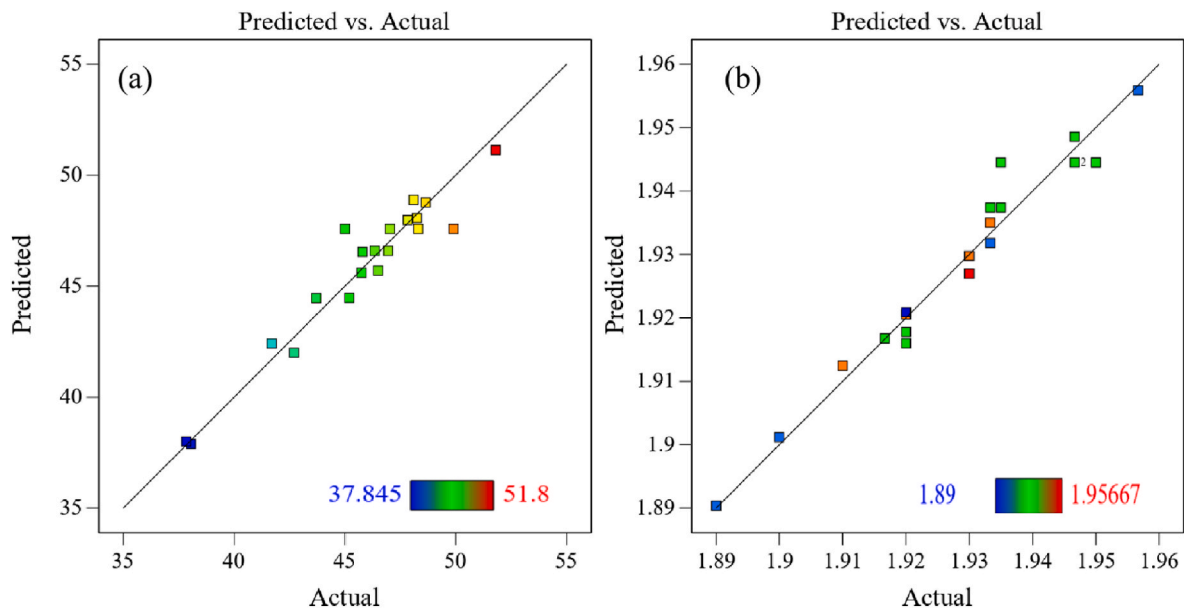


Fig. 5. The graph of predicted versus actual values (a) compressive strength and (b) free lime.

on the compressive strength and free lime content. Therefore, the CCD matrices are coded with respect to response parameters, such as Y_1 -compressive strength (MPa) and Y_2 -free lime (%), as presented in Table 3. The box plot for different responses of the experimental runs, including the compressive strength and free lime, against independent factors such as bentonite dose, bentonite calcination temperature, and calcination time are shown in Fig. 2(a–f), respectively. This result indicates that the comparison of only two variables at a time, such as compressive strength, reduces with increasing bentonite content, and 20% bentonite replacement can provide a large variation range of strength compared to other bentonite replacement doses. However, free lime decreased with increasing bentonite content; in particular, the 20% replacement showed the least free lime. This is commonly due to the substitution of bentonite in concrete, which improves the strength and reduces the free lime by consuming calcium hydroxide and forming CSH

gel, which is responsible for the strength (Taylor-Lange et al., 2015). As shown in Fig. 2(c and d), the compressive strength increases as the calcination temperature of bentonite increases; in contrast, it lessens the existence of free lime. This is because the high calcination of bentonite enhances the amorphous content, which consumes portlandite and improves compressive strength (Taylor-Lange et al., 2015). In addition, as presented in Fig. 2(e and f) the calcination time of bentonite substitution between 120 and 180 min increases the strength, and the low calcination time of 120 min significantly reduced the occurrence of free lime compared to the other sampled calcination times of bentonite. Similar results were found by Wahab (2008), who reported that longer exposure of pozzolanic materials to temperature causes re-crystallization, and hence, can lose its pozzolanic reactivity.

Table 10

Numerical value from CCD-RSM for actual, predicted residual and leverage for compressive strength and free lime responses.

Runs	Compressive strength (MPa)				Free lime (%)			
	Actual value	Predicted value	Residual	Leverage	Actual value	Predicted value	Residual	Leverage
1	46.35	46.59	−0.24	0.18	1.91	1.91	< −0.01	0.76
2	46.95	46.59	0.36	0.18	1.90	1.90	< −0.01	0.76
3	45.00	47.57	−2.57	0.22	1.95	1.94	<0.01	0.22
4	49.90	47.57	2.33	0.22	1.94	1.94	< −0.01	0.22
5	47.02	47.57	−0.55	0.22	1.95	1.94	<0.01	0.22
6	48.30	47.57	0.73	0.22	1.93	1.93	<0.01	0.76
7	51.80	51.13	0.67	0.62	1.93	1.93	<0.01	0.76
8	42.70	42.00	0.70	0.62	1.89	1.89	< −0.01	0.76
9	45.80	46.54	−0.74	0.62	1.93	1.94	< −0.01	0.76
10	41.70	42.41	−0.71	0.62	1.96	1.96	<0.01	0.76
11	45.20	44.48	0.72	0.62	1.92	1.92	< −0.01	0.76
12	43.70	44.46	−0.76	0.62	1.95	1.94	<0.01	0.22
13	48.25	48.07	0.18	0.76	1.94	1.94	< −0.01	0.18
14	38.05	37.89	0.16	0.76	1.92	1.92	< −0.01	0.62
15	37.84	38.01	−0.16	0.76	1.92	1.92	<0.01	0.62
16	47.83	47.97	−0.14	0.76	1.93	1.93	<0.01	0.62
17	48.65	48.78	−0.13	0.76	1.93	1.94	< −0.01	0.18
18	48.10	48.88	−0.78	0.76	1.92	1.92	−0.01	0.62
19	46.50	45.70	0.80	0.76	1.95	1.95	< −0.01	0.62
20	45.75	45.61	0.14	0.76	1.92	1.92	<0.01	0.62

3.2. Development of quadratic models

The experimental data from the Design Expert software in the CCD-RSM were obtained by partial substitution of bentonite in mortar suited to different models, such as cubic, quadratic, logarithmic, and linear models. Therefore, the statistical experiment performed various regression analyses to reach the best prediction of the response variables by the different models and suggested one of the most suitable models for the response variables. As shown in Table 4, the CCD-RSM used the response parameters Y_1 -compressive strength and Y_2 -free lime to obtain regression models based on the functions of all independent influencing factors, as indicated in Eq. (2) (Fode et al., 2024a).

$$Y = \mu_0 + \sum_{i=1}^n \mu_i X_i + \sum_{i=1}^n \mu_{ii} X_i^2 + \sum_{i=1}^{n-1} \sum_{j=i+1}^n \mu_{ij} X_i X_j + \varepsilon \quad (2)$$

where Y is the predicted response variable, μ_0 is the constant coefficient, μ_i is the linear coefficient, μ_{ii} is the quadratic coefficient, μ_{ij} is the interaction coefficient, X_i and X_j are the coded values of the variables, and ε is the error or unpredicted response variables in the experimental data.

As indicated in Tables 5 and 6, the quadratic models for compressive strength and free lime response were selected because of their statistically significant models with sequential p -values compared to the other regression models (cubic, 2FI, and linear). Both the compressive strength and free lime cubic model are aliased, indicating that the model is not appropriate (Myers and Montgomery, 2016). The adjusted R^2 of 0.85 and 0.92 for compressive strength and free lime, respectively, and the expected R^2 of 0.73 and 0.86, with a disparity of less than 0.2, indicate a strong correlation of the model's predicted values (Mohammed et al., 2019; Dahish and Almutairi, 2023).

Therefore, the experimental and predicted data were correlated according to the coefficient of the quadratic equation, to reflect a significant quadratic model. In addition, the lack of fit can also be used to assess the quality of the model: a smaller value of less than 0.05 indicates unworthy model. The p -values for compressive strength and free lime were 0.91 and 0.72, respectively, demonstrating that the lack of fit is not significant and shows the quadratic model's excellent fitness and robustness (Mohammed et al., 2019).

A sufficient degree of precision was used to measure the signal-to-noise ratio, which must be above 4.00. Thus, as Table 7 illustrates, the precision rates of the compressive strength and free lime statistical models were 12.79 and 13.98, respectively, demonstrating the

reliability and appropriateness of the anticipated outcome of the suggested quadratic model (Dahish and Almutairi, 2023). The coefficients of variation (CV%) for the compressive strength and free lime responses are 3.05 and 0.28, respectively, and are deemed credible and repeatable because they deviate very little from the mean (less than 5.00%).

3.3. Analysis of variance for the quadratic model

The ANOVA of the quadratic models revealed the components' statistical significance and interactive effects on the response. Therefore, using the F and P -values of the substitution of bentonite on the strength and free lime of cement composite materials, the ANOVA was able to detect the interaction of the model significance and suitability for the independent components. Hence, the CCD-RSM produced ANOVA for the suggested quadratic models for each response variable, as shown in Tables 8 and 9, which indicates the significance of the model suggests that all the factors have an impact on the response parameters (Dahish and Almutairi, 2023).

The model is statistically significant since the compressive strength and free lime have F -values of 12.71 and 23.69, respectively. This is because the F -value is quite high compared with the p -value (<0.0001). So, strong quadratic model adaptability is shown in Tables 6 and 7, which display the compressive strength and free lime of the LOF of P -values 0.91 and 0.84 with F -values of 0.28 and 0.38, respectively. These results show that compared with the pure error, the LOF is not significant (Mohammed et al., 2019).

In addition, Tables 8 and 9's response variables for the two created quadratic models indicate that the models are statistically significant, with p -values less than 0.05. Also, the compressive strength and free-lime efficiency were significantly influenced by the three interaction factors. However, the quadratic model's inconsequential terms may be a discrepancy between the adjusted correlation coefficient (R^2) and determination coefficient. Eqs. (3) and (4) are quadratic results generated by CCD-RSM, which show that compressive strengths A , BC , B^2 , and C^2 have negative signs that indicate a lessening effect, while B , C , AB , and AC have positive signs indicating a rising effect. Additionally, factors such as A , AB , A^2 , B^2 , and C^2 have negative signs, indicating a lessening effect, and AB , BC , C , and AC parameters have positive signs, which increases the occurrence of free lime in cement composite materials. Thus, it was observed that increasing factor A : the bentonite dose reduces the compressive strength; however, increasing the bentonite dose can consume free lime, which is due to the addition of pozzolana, which consumes free lime and produces CSH gel. This is responsible for

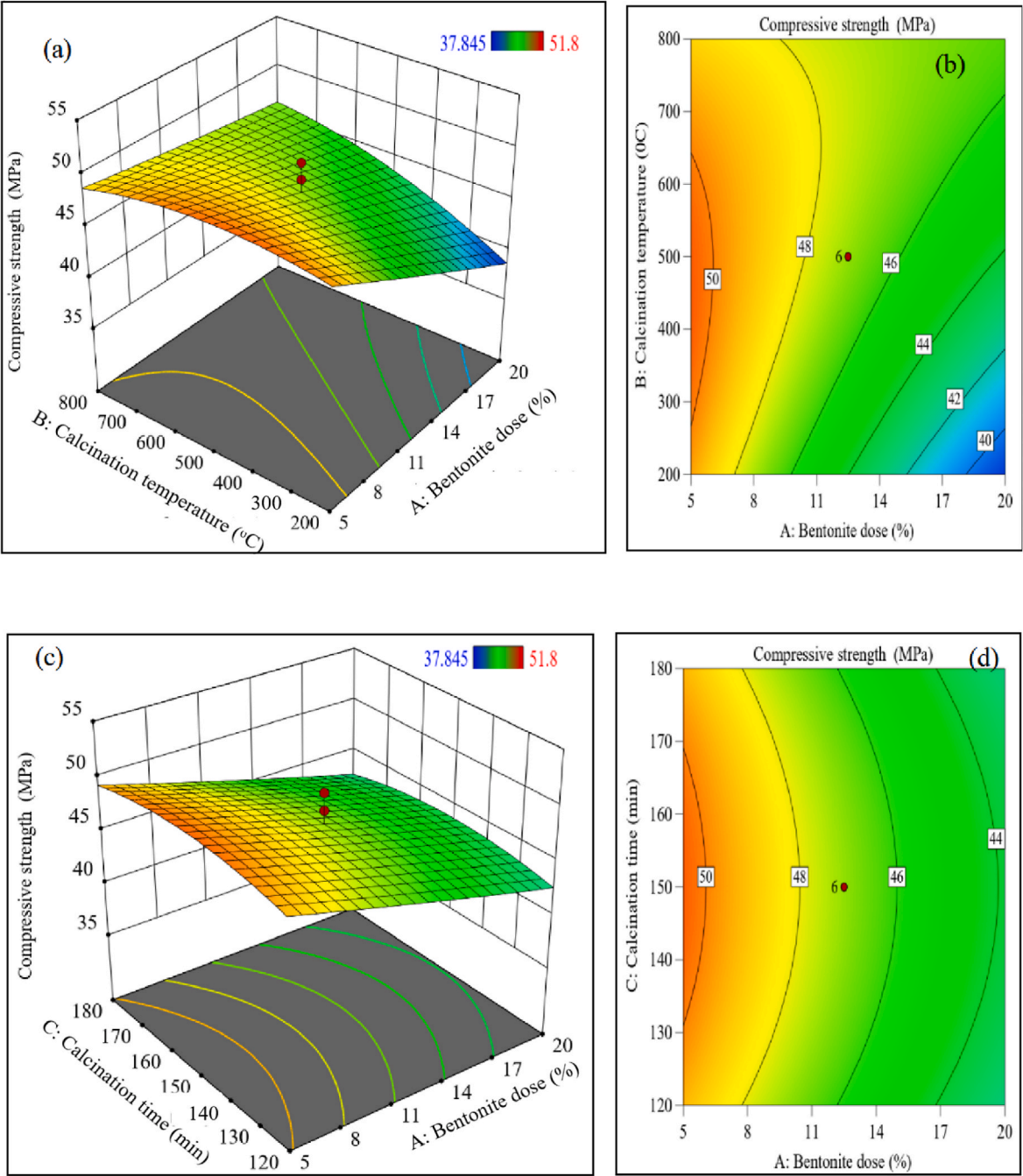


Fig. 6. Compressive strength response variable model interaction by 3D surface and contour plots.

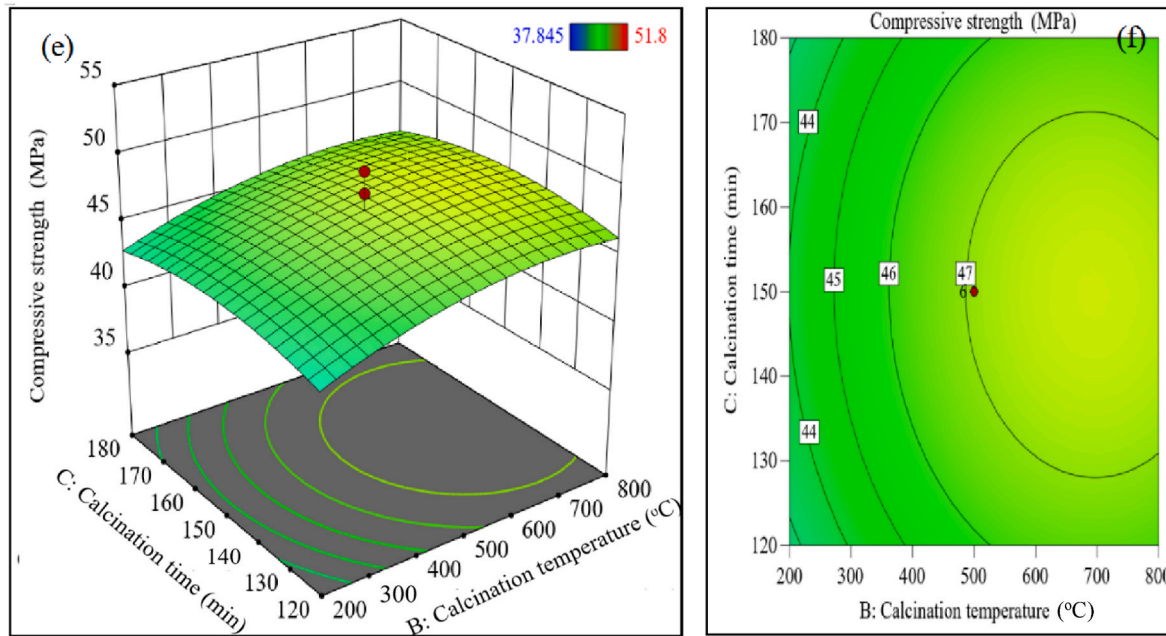


Fig. 6. (continued).

the improvement in strength, although a consistent increase in the pozzolanic material dose cannot provide a consistent improvement in strength. Therefore, the optimization of the two parameters is required to obtain the optimum bentonite dose that is responsible for the acceptable compressive strength and low free lime.

$$Y_1 \text{ (Compressive strength)} = +47.08 - 3.31A + 1.72B + 0.0043C + 2.13AB + 0.0019AC - 0.0519BC + 0.1037A^2 - 1.34B^2 - 1.22C^2 \quad (3)$$

$$Y_2 \text{ (Free lime)} = +1.94 - 0.0124A - 0.0004B + 0.0023C + 0.0033AB + 0.0117AC + 0.0083BC - 0.0030A^2 - 0.0116B^2 - 0.0078C^2 \quad (4)$$

where A: bentonite dose B: bentonite calcination temperature, and C: bentonite calcination time all are independent factors.

In addition, the ANOVA results in Tables 8 and 9 show $p < 0.01$ and F -values of 64.53 and 69.81, for bentonite replacement, respectively, to the response of strength and free lime, which indicates that the bentonite doses have a major influence among the three independent factors studied, because the lower the p -values and the higher the F -values, which shows that the higher the influence of the model terms indicating the model effectiveness (Gour et al., 2022; Dahish and Almutairi, 2023).

3.4. Adequacy and suitability test for quadratic models

As presented in Figs. 3–5, the diagnostic graphs can be used to evaluate the appropriateness and suitability of the suggested quadratic models for the effect of bentonite replacement on the strength and free lime of cementing material. Hence, the normal % probability plot of studentized residuals is the most acceptable graphical tool for evaluating the residual normality and validating multivariate regression models. The normal % probability vs. externally and internally studentized residual plots for the compressive strength and free lime by the addition of calcined bentonite are illustrated in Fig. 3(a–d). From the results, it is observed that the residual distribution on both sides of the normal line for calcined bentonite replacement on the compressive strength and free lime shows a very small distribution of normal error to the predicted and actual values of the response variables (Mohammed et al., 2019). In addition to this, the random normal distributions of residuals between -3 and $+3$, -4 and $+4$, and -2 and $+2$ sigma values are within the limits that indicate the suitability, reliability, and soundness of the quadratic model used (Cai et al., 2013). Also, the DFFITS versus run

number presented in Fig. 4 indicates that all the run points are distributed between the limits of $+2$ and -2 for both the compressive strength and free lime, which shows that the experimental data can affect the anticipated data. Other detailed diagnostic tests of the CCD-RSM are shown in Figures S1–6, which indicate the accuracy and reliability of the quadratic model used for both response factors.

As shown in Eqs. (5) and (6), the actual and predicted data can be suited to the proposed quadratic models of compressive strength and free lime content, respectively. Hence, Fig. 5(a and b) indicates the variation between the actual and predicted values of compressive strength and free lime content, which indicates that the graphs have a significant relationship between the predicted quadratic models' and experimental data for both response variables. In addition, because the regression model best fits the data, the predicted and actual response variations must not be high. Therefore, as presented in Table 10, there is a very small variation between the actual and predicted data for both response variables, indicating the accuracy and valid predictions of the suggested response surface quadratic models (Dahish and Almutairi, 2023). The numerical values of the actual and predicted data for the compressive strength and content of free lime owing to the independent factors of bentonite dose, calcination temperature, and calcination time are summarized in Table 10.

3.5. Response surface modeling by factors interactions

As illustrated in Figs. 6 and 7 contour and 3D response surfaces graph of the response variables represent quadratic model regression, which can evaluate the relationship between the independent variables and responses.

3.5.1. Compressive strength

As shown in Fig. 6(a) and (b), increasing the bentonite dose significantly reduced the free lime content at low calcination temperatures. In addition, the strength increased at low doses of bentonite replacement and decreased at higher replacement levels at a fixed low calcination temperature of 200°C ; however, increasing the calcination temperature at high bentonite replacement can enhance the strength. This is mainly because bentonite appears in a consolidated form that requires activation by heating to increase the reactivity. The same finding was reported by Keke et al. (2022), who found that the use of uncalcined bentonite

reduces the compressive strength of mortar and reported that 750 °C is the best pozzolanic activation temperature for raw bentonite. In contrast, the study found that the activation of bentonite at 900 °C was not conducive to improve the pozzolanic reactivity.

Additionally, as shown in Fig. 6(c and d), the activation of bentonite between the lower and upper limits of the calcination time at the low replacement dose can increase the compressive strength of the mortar. Also, the calcination time between the middle of the lower and upper limits increases the strength of bentonite at higher calcination temperatures, as shown in Fig. 6(e and f). This is mainly because the reactivity of bentonite increases with increasing calcination temperature and time (Keke et al., 2022). Similar observations were made by Wahab et al. (Wahab, 2008), where calcination at 750 °C enhanced the strength and pozzolanic reactivity, and increased the concentration of the alkali solution and the ion exchange capacities of bentonite. However, from 850 °C onward, the reddish bentonite color changed to reddish-black,

which indicates particles of bentonite started burning; hence, prolonged exposure of pozzolanic materials to temperatures above the temperature of dehydroxylation that enhances re-crystallization and, hence, loses its pozzolanic reactivity.

3.5.2. Free lime

The 3D plot of the interaction of bentonite calcination temperature and replacement dose on free lime is shown in Fig. 7. As shown in Fig. 7 (a and b), the free lime content decreases with increasing bentonite replacement doses and calcination temperature. The free lime in cement composite materials has a great chance of being consumed by the higher doses of bentonite replacement at higher bentonite calcination temperatures. Hence, the calcination of bentonite promises the enhancement of physico-chemical properties, mainly to produce an extra C-S-H gel in mortar or concrete mixtures (Mesboui and Benyounes, 2021). Similarly, as shown in the same graph, at the lower point of calcination

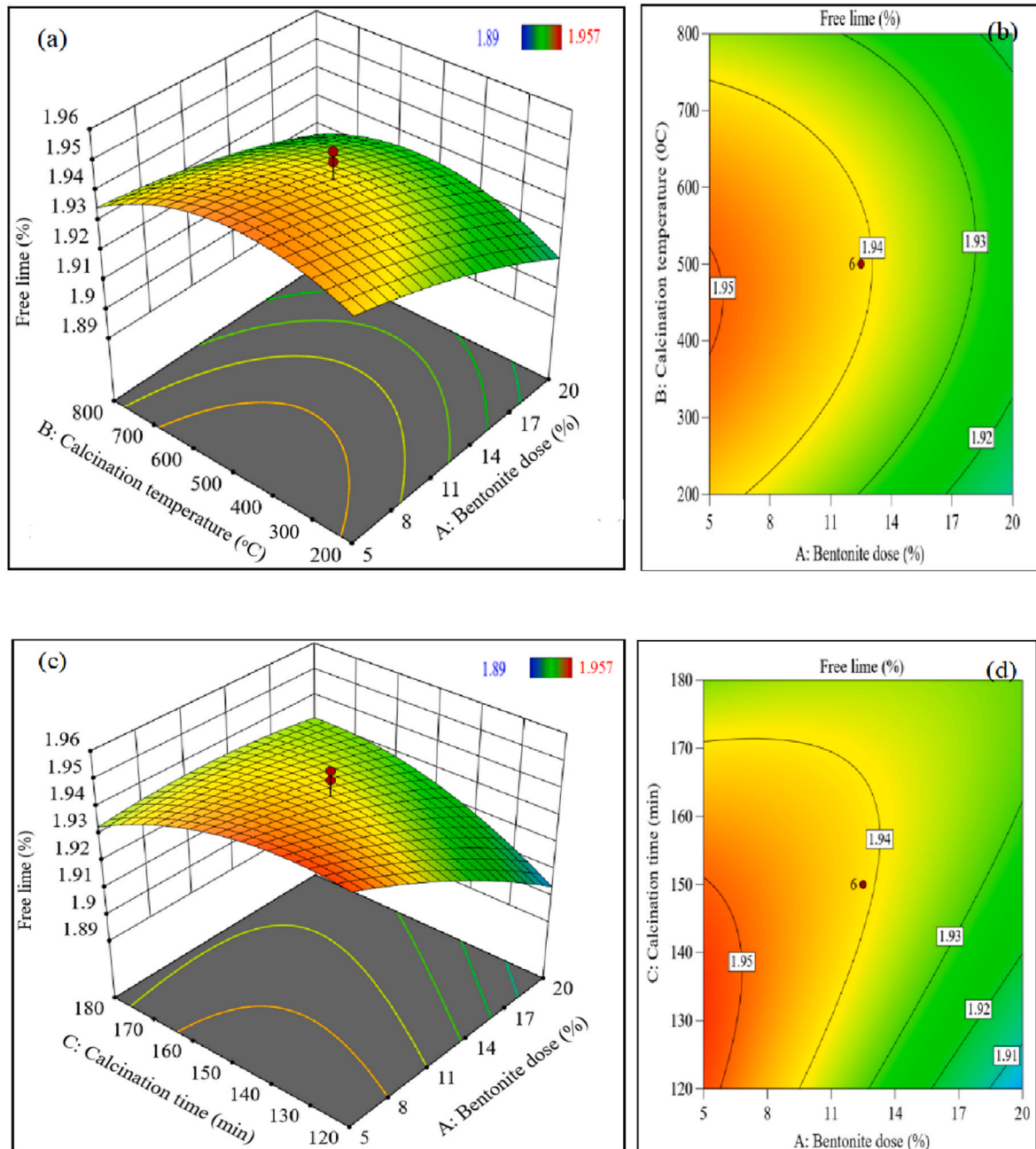


Fig. 7. Free lime content response variable model interaction by 3D surface and contour plots.

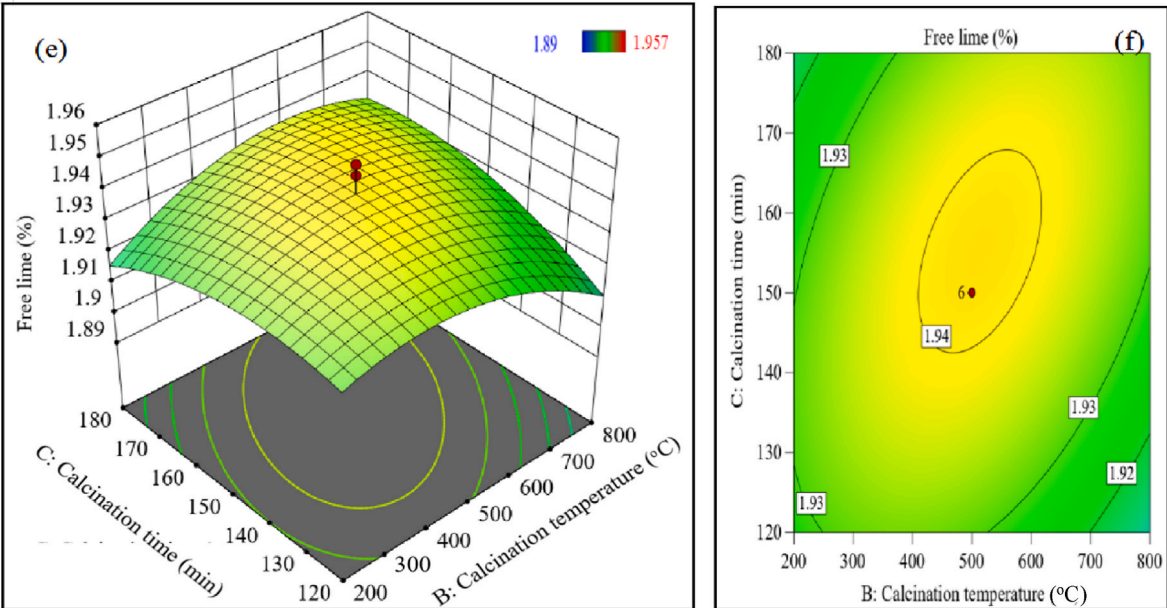


Fig. 7. (continued).

Table 11
For all factors and response variables, established optimization goals.

Name	Goal	Lower limit	Upper limit	Lower weight	Upper weight	Importance
A: Bentonite dose	Is in range	5.00	20.00	1.00	1.00	3.00
B: Calcination temperature	Is in range	200.00	800.00	1.00	1.00	3.00
C: Calcination time	Is in range	120.00	180.00	1.00	1.00	3.00
Compressive strength	Maximize	37.84	51.80	1.00	1.00	3.00
Free lime	Minimize	1.89	1.96	1.00	1.00	3.00

temperature and higher bentonite replacement dose, there is the occurrence of the highest amount of free lime; hence, it was discovered that the high substitution of raw bentonite cannot increase the consumption of free lime. This is mainly because bentonite needs activation to react more with the free lime in cementitious materials, which can enhance the strength (Rehman et al., 2019a).

As illustrated in Fig. 7(c and d), reducing the calcination time by increasing the bentonite substitution dose can significantly reduce the occurrence of free lime. However, substituting the smaller bentonite at a lower calcination temperature results in higher free lime, and in contrast, increasing the calcination time at a lower substitution of bentonite slightly reduces the free lime. In addition, as shown in Fig. 7(e and f), at a low calcination time and high calcination temperature, or at a high calcination time and low calcination temperature, the presence of free lime was significantly reduced. This indicates besides the bentonite dose, the consumption of free lime highly affected both calcination temperature and time for activation of bentonite in the mortar. This is due to the reason that bentonite mostly exists in a consolidated form; which indicates need a suitable treatment method. Hence, it cannot be directly used as a supplementary material to improve the performance of cementing materials (Rehman et al., 2019a), and many raw clays have crystal structures that require calcination for active pozzolanic reactivity (Tironi et al., 2014).

Besides these, Hakamy et al. (2015) studied the effect of employing calcined nano-bentonite clay and raw nano-bentonite clay in cement composite materials and the study found adding calcined nano-bentonite clay offered a higher amount of C_3S and C_2S with a lower amount of free lime compared to using of raw nano-bentonite clay. Thus, this study confirmed the high pozzolanic reaction found by the employment of calcined bentonite rather than raw bentonite replacement in cement composite materials that can consume more free lime.

3.6. CCD-RSM numerical optimization

The numerical optimization can be carried out by the CCD-RSM through the specified goals to obtain the best performance from the desired output. So, as presented in Table 11, the criteria used to get the maximum strength and minimum free lime of the investigated calcined bentonite replacement variables are “in range.” Based on the established criteria, 50 solutions containing the optimum experimental operating conditions of bentonite employment-independent factors were utilized. Under this, selected optimum experimental conditions are 19.99% of bentonite replacement dose, 799.99 °C calcination temperature, and 135.04 min calcination time as presented in Fig. 8, the higher strength at minimum free lime was 46.10 MPa and 1.904% respectively at the desirability of 0.68. Therefore, the optimum results of the maximized compressive strength and reduced free lime were compared with those of the control mixture, which were higher than 24.94% and 3.08% for compressive strength and free lime, respectively. Similarly, according to Reddy & Reddy (Reddy and Reddy, 2021), with 20% bentonite substitution at a calcination temperature of 800 °C, a higher compressive strength was recorded and it was found that the improvement in strength was mainly due to the micro-filling ability of bentonite because the particle size of bentonite is much smaller than that of cement and the higher reactivity of calcined bentonite at 800 °C, which forms a secondary CSH gel by consuming free lime.

3.6.1. Optimization and desirability

Furthermore, Fig. 9(a–i) verifies that the obtained optimization solution on the 29th is more acceptable than the other 50th solutions. For instance, desirability in the multiple response method indicates a desirable solution using an objective function ranging from 0 to 1. Hence, the possible best combinations of various optimization goals

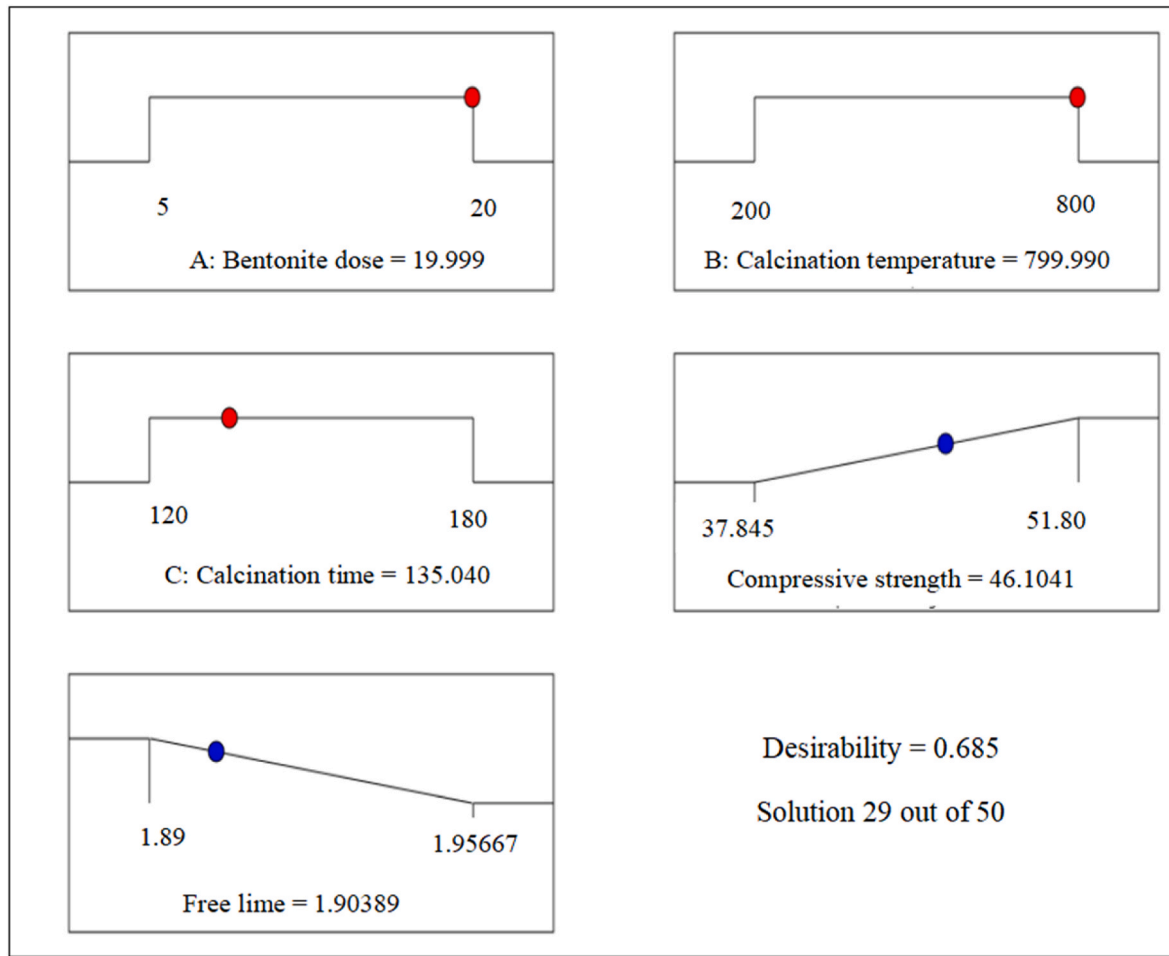


Fig. 8. Optimal CCD-RSM conditions by optimization ramps for factor variables and maximized response variables.

must reach an objective function near 1, resulting in anticipated values that match expectations (Myers and Montgomery, 2016). Therefore, from Fig. 9(a–c), it can obtain a higher strength than the obtained optimum value at a very low bentonite replacement; however, it has the lowest desirability that cannot indicate the best combination value. In addition, the lowest bentonite replacement dose cannot reduce the occurrence of free lime, energy consumption, or environmental pollution problem, and is not more cost-effective compared to the higher replacement of bentonite in mortar. Similarly, as illustrated in Fig. 9 (d–f), increasing the bentonite dose substitution significantly reduces the occurrence of free lime, which can lessen the expansion of construction materials. In addition, from Fig. 9(g–i) for the chosen criteria presented in Table 11, the maximum strength and, at the same time, lower free lime were obtained at 800 °C compared to the other calcination temperatures. Generally, from these statistical experimental regression values, the chosen factor (independent) variable had a significant influence on the response (dependent) variables.

Generally, observed the optimized bentonite calcination temperature is much lower than cement clinker which mostly requires 1450 °C (Taylor-Lange et al., 2015), and grinding of major raw materials of cement, especially, limestone requires much energy, besides the cost of OPC is higher which is reported that one kg of cement unit price is 85.7% more than the same bentonite content (Ali et al., 2012; Aygörmec, 2021). Hence, replacing bentonite in mortar or concrete is crucial to reduce CO₂ emissions and energy consumption, and is cost-effective in addition to improving the strength and reducing concrete expansion due to free lime reduction.

3.7. Experimental validation for CCD-RSM optimized results

For both strength and free lime the optimized results were experimentally validated to assess their practical applicability, bentonite dose 19.99%, calcination temperature 799.99 °C, and calcination time 135.04 min as it can give experimentally 46.10 MPa and 1.90 or near up to ±5% error. The difference between the validated experimental result and model-predicted optimized value was determined as the error using Eq. 10 as (Adamu et al., 2022).

$$\varepsilon = \frac{(a - b)}{a} \times 100 \quad (5)$$

where ε is the percentage of error, a and b are respectively the experimental and model response values.

The same method was used to determine the compressive strength and free-lime content. As presented in Table 12, the experimental validation has a 95% confidence level to the optimized results, found 46.45 MPa and 1.89% for compressive strength and free lime, respectively, which is very close to the optimum predicted by CCD-RSM, confirming the validity and accuracy of the quadratic model.

3.8. Experimental assessment on durability of optimized results in a different adverse environments

The optimized bentonite replacement parameters were evaluated for mortar strength and mass loss at 56 days of curing age in 5% HCl and HNO₃ and compared with the control mixture, as shown in Fig. 10(a and b). The results indicated that the strength and mass loss of optimized

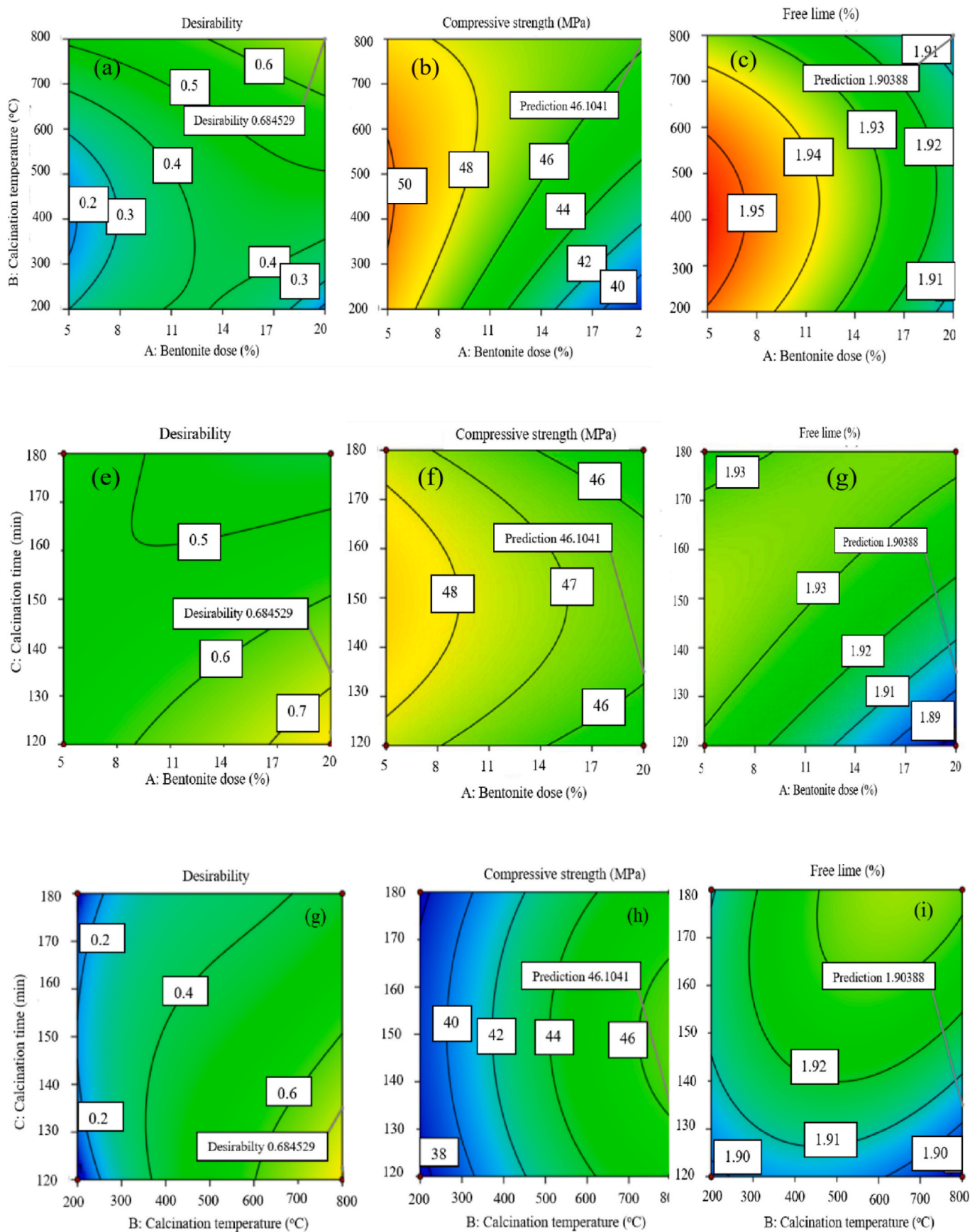


Fig. 9. 2-D contour plot of optimization containing values of optimal factor variables and maximized response variables.

Table 12

Experimental validation of optimized CCR-RSM values.

Response variables	CCD-RSM Optimum predicted value	95% lower bound to predicted value	Experimental value	95% upper bound to predicted value
Compressive strength (MPa)	46.10	43.80	46.45	48.41
Free lime (%)	1.90	1.81	1.89	1.99

bentonite in mortar is less than that of the control mixture; hence, it can reduce the effects of 5% HCl and HNO₃, respectively strength loss by 82% and 33.4%, and mass loss by 22.72% and 59.5%, respectively, compared to the control mixture. This is basically due to the employment of optimized calcined bentonite with higher reactivity, which can consume more free lime and produce extra entringite that can interlock the mortar matrix and protect the penetration of acids; hence, it can resist deterioration in acidic environments.

The effects of 10% sodium chloride and an elevated temperature of 200 °C for 2 hours on the control mixture and optimized bentonite substitution in the mortar are presented in Fig. 11(a). The result indicates that the replacement of the optimized bentonite reduces mass loss by 33.08% owing to immersion in 10% sodium chloride solution compared to the control mixture at the age of 56 days. In addition, the

optimized bentonite replacement also improved the mass loss at an elevated temperature of 200 °C for 2 hours by 69.56% compared to the control mixture. This is mostly due to the high reactivity of bentonite at 800 °C, which can form pozzolanic reactions, interlock the matrix, and create a dense mortar structure that cannot easily dehydrate water at high temperatures, and consequently, cannot easily lose mass in the salt environment (Rehman et al., 2019a). Therefore, it is crucial for concrete-reinforced steel bars to prevent corrosion. In addition, as shown in Fig. 11(b), the water absorption of the optimized bentonite replacement is significantly less than that of the control mixture, which is due to calcined bentonite having less specific gravity and very fine grains compared to Portland cement, which fills the pores and reduces the penetration of water (Al-Hammood et al., 2021b).

3.9. Comparison of present results with different literature

In the present study, the percentage improvement in strength at 28 days and the lower occurrence of free lime compared to the previous studies are shown in Table 13, through the novel treatment of bentonite to different influencing factors for both responses. This is mostly because bentonite treatment provides a beneficial approach to be employed in mortar by highly improving the strength of conventional cementing materials and indirectly reducing the energy consumption and environmental pollution problems facing the world due to the production of cement.

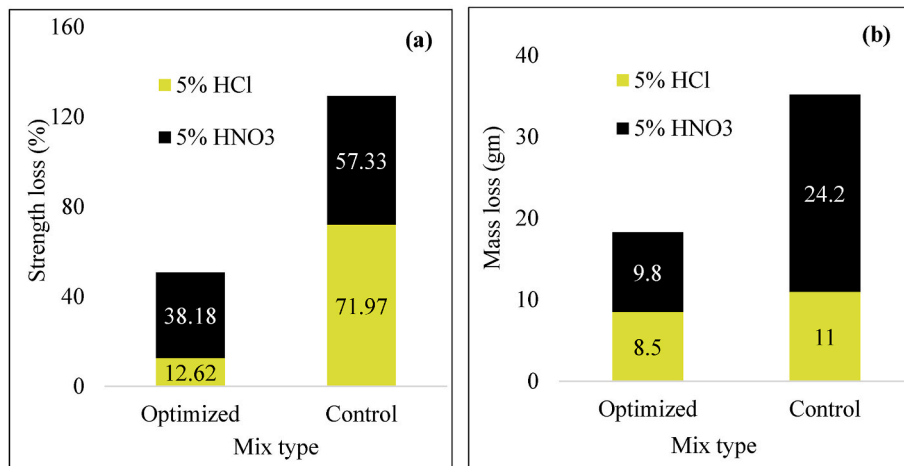


Fig. 10. The effect of curing in 5% of HCl and HNO₃ for optimized bentonite replacement and control mixture (a) strength loss and (b) mass loss.

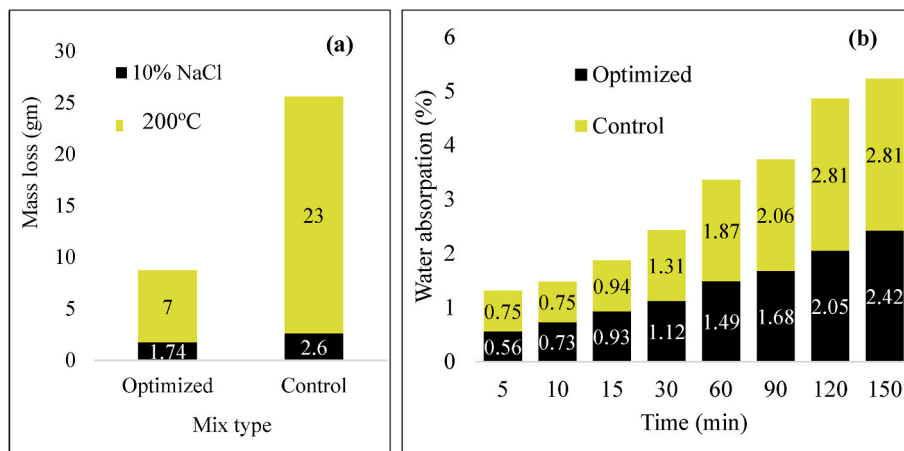


Fig. 11. (a) mass loss due to curing in 10% of NaCl and elevated temperature at 200 °C and (b) percentage of water absorption for mortar employed optimized bentonite and control mixture.

Table 13

Comparison of the optimized compressive strength and free lime values improved due to bentonite employment in mortar or concrete with the previous studies.

Type of cement composite material	W/C	Bentonite dose (%)	Calcination		Using 20% bentonite increased compressive strength (%)	Reduced free lime (%)	Reference
			Range of temperature (°C)	Optimum temperature (°C)			
Mortar	0.485	5–20	200–800	800	24.94	3.08	Present study
Mortar	0.34	5, 10, 15, 20	Not calcined	–	5.74	–	Kadhim et al. (2022)
Mortar	–	10, 20, 30	200, 400, 600	400	2.96	–	Vijay and Achyutha Kumar Reddy (2021)
Mortar	0.55	20, 25, 30, 40, 50, 100	150, 250, 500, 750, 950	150	4.00	–	Mirza et al. (2009)
Mortar	0.50	5, 10, 15, 20, 25, 30	600, 800, 1000	600	8.37	–	Laidani et al. (2022)
Concrete	0.45	5, 10, 15, 20	Not calcined	–	7.14	–	Ahmad et al. (2021)
Concrete	0.40	5, 10, 15, 20, 25, 30	Not calcined	–	4.48	–	Laidani et al. (2020)
Mortar	0.484	5	750	–	–	2.37	Alani et al. (2020)
Mortar	–	5, 10, 15, 20, 25, 30	700 & 800	800	7.34	–	Reddy and Reddy (2021)

4. Conclusions

The present study investigated the modeling and optimization of different bentonite doses, calcination temperatures, and calcination times on the strength and free lime of mortar using the statistical method CCD-RSM, and experimentally validated the optimized statistical values. In addition, different durability tests were conducted on the optimized bentonite replacement in various adverse environments, and the following conclusions were drawn.

1. The model for both compressive strength and free lime response variables was found to be significant, accurate, reliable, and have excellent fitness to the experimental work because the ANOVA of the model showed a p -value of less than 0.05, and all the diagnostic tests were within the limit.
2. Increasing the calcination temperature and bentonite replacement can significantly enhance the compressive strength and reduce the free lime. However, at lower calcination temperatures and higher bentonite replacements of 200 °C and 20%, respectively, there is the occurrence of high free lime; hence, it was discovered that the high substitution of bentonite doses cannot grant to the consumption of free lime at lower calcination temperatures of bentonite.
3. From different 50n solutions obtained by CCD-RSM, the optimum was found at independent factors A: Bentonite dose 19.99%, B: Calcination temperature 799.99 °C and C: Calcination time 135.04 min that gives the response variables Y_1 : Compressive strength 46.10 MPa and Y_2 : Free lime 1.90% at the criteria of maximizing the strength and minimizing the free lime which is also, experimentally validated found within $\pm 95\%$ confidence level. Thus, the optimized response results increased the strength by 24.94% and reduced the free lime by 3.08% compared to the control mixture.
4. Furthermore, the optimized replacement of bentonite at A: Bentonite dose 19.99%, B: Calcination temperature 799.99 °C, and C: Calcination time 135.04 min, have significantly improved durability in the different adverse environments and reduced the effect of 5% HCl and HNO₃, corresponding to a strength loss of 82% and 33.4 %, respectively, while mass loss by 22.72% and 59.5%, respectively, compared to the control mixture. Furthermore, the optimized bentonite replacement reduced the mass loss by 33.08% due to immersion in 10% sodium chloride solution and decreased the mass loss at an elevated temperature of 200 °C for 2 hours by 69.56% compared to the control mixture at a mortar age of 56 days.

Generally, the optimized bentonite replacement in mortar is highly beneficial for improving strength, reducing concrete expansion due to lessening free lime, and can improve the durability of mortar in different adverse environments, in addition to highly reducing environmental

pollution, especially CO₂ emissions, energy consumption, and cost-effectiveness compared to conventional mortar/concrete production.

5. Recommendation

The optimum factors and response found in this study belong to bentonite with the same or very similar chemical composition as that in the present study. Therefore, studies should be conducted at different sites of bentonite extraction. In addition, the authors suggest that future studies conduct more detailed tests such as R3, flexural and splitting tensile strength, fatigue strength, and all other tests needed for construction materials that are not included in the present study related to the employment of calcined bentonite in either mortar or concrete.

CRedit authorship contribution statement

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

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.clet.2024.100844>.

Data availability

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

References

- A. C618, 2012. Standard Specification for Coal Fly Ash and Raw or Calcined Natural Pozzolana for Use in Concrete. ASTM International, West Conshohocken, PA, pp. 3–6. <https://doi.org/10.1520/C0618>. ASTM Stand., 2010.
- Adamu, M., Marouf, M.L., Ibrahim, Y.E., Ahmed, O.S., Alanazi, H., Marouf, A.L., 2022. Modeling and optimization of the mechanical properties of date fiber reinforced concrete containing silica fume using response surface methodology. *Case Stud. Constr. Mater.* 17, e01633. <https://doi.org/10.1016/j.cscm.2022.e01633>.
- Adjei, S., Elkhatny, S., Sarmah, P., Mohsen, A., 2021. Evaluation of calcined Saudi calcium bentonite as cement replacement in low-density oil-well cement system. *J. Pet. Sci. Eng.* 205, 108901. <https://doi.org/10.1016/j.petrol.2021.108901>.
- Ahad, M.Z., Ashraf, M., Kumar, R., 2018. Thermal, physico-chemical, and mechanical behaviour of mass concrete with hybrid blends of bentonite and fly ash. *Materials-MDPI*. <https://doi.org/10.3390/ma12010060>.
- Ahmad, J., Tufail, R.F., Aslam, F., Mosavi, A., Alyousef, R., Javed, M.F., Zaid, O., Khan Niazi, M.S., 2021. A step towards sustainable self-compacting concrete by using partial substitution of wheat straw ash and bentonite clay instead of cement. *Sustain. Times* 13, 1–17. <https://doi.org/10.3390/su13020824>.
- Ahmed, M.A., 2017. Structural performance of reinforced concrete beams with NanoMeta-kaolin in shear. *IOSR J. Mech. Civ. Eng.* 14, 88–96. <https://doi.org/10.9790/1684-1402048896>.
- Akbar, J., Alam, B., Ashraf, M., Afzal, S., Ahmad, A., Shahzade, K., 2012. Evaluating the effect of bentonite on strength and durability of high performance concrete. *Int. J. Adv. Struct. Geotech. Eng.* 2.
- Al-Hamoud, A.A., Frayeh, Q.J., Abbas, W.A., 2021a. Raw bentonite as supplementary cementitious material - a review. *J. Phys. Conf. Ser.* 1795. <https://doi.org/10.1088/1742-6596/1795/1/012018>.
- Al-Hamoud, A.A., Frayeh, Q.J., Abbas, W.A., 2021b. Thermally activated bentonite as a supplementary cementitious material - a Review. *Eng. Technol. J.* 39, 206–213. <https://doi.org/10.30684/etj.v39i2A.1733>.
- Alani, S., Hassan, M.S., Jaber, A.A., Ali, I.M., 2020. Effects of elevated temperatures on strength and microstructure of mortar containing nano-calcined montmorillonite clay. *Construct. Build. Mater.* 263, 120895. <https://doi.org/10.1016/j.conbuildmat.2020.120895>.
- Ali, M.S., Arsalan, R., Khan, S., Lo, T.Y., 2012. Utilization of Pakistani bentonite as partial replacement of cement in concrete. *Construct. Build. Mater.* 30, 237–242. <https://doi.org/10.1016/j.conbuildmat.2011.11.021>.
- Anand, A., Kumari, S., 2022. A study on strength and durability of concrete by partial replacement of cement with bentonite. *Int. Res. J. Mod. Eng. Technol. Sci.* 4, 3964–3967.
- Andrade, C., Martínez-serrano, A., Sanjuán, M.A., Tenorio Ríos, J.A., 2021. Reduced carbonation, sulfate and chloride ingress due to the substitution of cement by 10% non-precalcined bentonite. *Materials* 14, 1–18. <https://doi.org/10.3390/ma14051300>.
- Arrigoni, A., Panesar, D.K., Duhamel, M., Opher, T., Saxe, S., Posen, I.D., MacLean, H.L., 2020. Life cycle greenhouse gas emissions of concrete containing supplementary cementitious materials: cut-off vs. substitution. *J. Clean. Prod.* 263, 121465. <https://doi.org/10.1016/j.jclepro.2020.121465>.
- ASTM C109/C109M-02, 2002. Standard test method for compressive strength of hydraulic cement mortars. *Annu. B. ASTM Stand.*
- Aygörmüş, Y., 2021. Assessment of performance of metabentonite and metazeolite-based geopolymers with fly ash sand replacement. *Construct. Build. Mater.* 302. <https://doi.org/10.1016/j.conbuildmat.2021.124423>.
- Cai, L., Wang, H., Fu, Y., 2013. Freeze-thaw resistance of alkali-slag concrete based on response surface methodology. *Construct. Build. Mater.* 49, 70–76. <https://doi.org/10.1016/j.conbuildmat.2013.07.045>.
- Chamundeswari, J., 2012. Experimental study on partial replacement of cement by bentonite in paverblock. *Int. J. Eng. Trends Technol.* 3, 41–47. <http://www.ijettjournal.org>.
- Channa, S.H., Mangi, S.A., Bheel, N., Soomro, F.A., Khahro, S.H., 2022. Short-term analysis on the combined use of sugarcane bagasse ash and rice husk ash as supplementary cementitious material in concrete production. *Environ. Sci. Pollut. Res.* 3555–3564. <https://doi.org/10.1007/s11356-021-15877-0>.
- Cheng, S., Shui, Z., Sun, T., Yu, R., Zhang, G., 2018. Durability and microstructure of coral sand concrete incorporating supplementary cementitious materials. *Construct. Build. Mater.* 171, 44–53. <https://doi.org/10.1016/j.conbuildmat.2018.03.082>.
- Coruh, S., Elevli, S., 2015. Optimization study of dye removal by cement kiln dust using the central composite design of experiments. *Glob. NEST J.* 17, 93–102. Copyright© 2015 Global NEST.
- Costafreda, J.L., Mart, D.A., Presa, L., 2021. Effects of a natural mordenite as pozzolan material in the evolution of mortar settings. *Mater. MDPI* 1–22, 10.3390/ma14185343.
- Dahish, H.A., Almutairi, A.D., 2023. Effect of elevated temperatures on the compressive strength of nano-silica and nano-clay modified concretes using response surface methodology. *Case Stud. Constr. Mater.* 18, e02032. <https://doi.org/10.1016/j.cscm.2023.e02032>.
- Deboucha, W., Oudjit, M.N., Bouzid, A., Belagraa, L., 2015. Effect of incorporating blast furnace slag and natural pozzolana on compressive strength and capillary water absorption of concrete. *Procedia Eng.* 108, 254–261. <https://doi.org/10.1016/j.proeng.2015.06.145>.
- Dunuweera, S.P., Rajapakse, R.M.G., 2018. Cement types, composition, uses and advantages of nanocement, environmental impact on cement production, and possible solutions. *Adv. Mater. Sci. Eng.* 2018. <https://doi.org/10.1155/2018/4158682>.
- Elbar, M., Senhadji, Y., Benosman, A.S., Khelafi, H., Mouli, M., 2018. Effect of thermo-activation on mechanical strengths and chlorides permeability in pozzolanic materials. *Case Stud. Constr. Mater.* 8, 459–468. <https://doi.org/10.1016/j.cscm.2018.04.001>.
- Fernandez, R., Martirena, F., Scrivener, K.L., 2011. Cement and Concrete Research the origin of the pozzolanic activity of calcined clay minerals : a comparison between kaolinite, illite and montmorillonite. *Cement Concr. Res.* 41, 113–122. <https://doi.org/10.1016/j.cemconres.2010.09.013>.
- Fezzioui, N., Amrane, B., Souici, K., Hani, B., Kennouche, S., Safi, B., Nadir, M., 2021. Effect of metakaolin as partially cement replacement on the compressive strength of standard mortars. *Rev. Rom. Ing. Civila/Romanian J. Civ. Eng.* 12, 268–280. <https://doi.org/10.37789/rjce.2021.12.2.6>.
- Firdous, M., Singh, B., 2021. Supplementary cementitious materials in concrete and associated structural and environmental benefits : a review Supplementary cementitious materials in concrete and associated structural and environmental benefits : a review. In: *IOP Conf. Ser. Earth Environ. Sci.*. <https://doi.org/10.1088/1755-1315/889/1/012077>.
- Fode, T.A., Jande, Y.A.C., Kivevele, T., 2023. Effects of different supplementary cementitious materials on durability and mechanical properties of cement composite – comprehensive review. *Heliyon* 9, e17924. <https://doi.org/10.1016/j.heliyon.2023.e17924>.
- Fode, T.A., Jande, Y., Abeid, Chande, Kivevele, T., 2024a. Modelling and optimization of multiple replacement of supplementary cementitious materials for cement composite by response surface method. *Clean. Eng. Technol.* 19, 1–18. <https://doi.org/10.1016/j.clet.2024.100735>.
- Fode, T.A., Abeid, Y., Jande, C., Kivevele, T., 2024b. Effects of raw and different calcined bentonite on durability and mechanical properties of cement composite material. *Case Stud. Constr. Mater.* 20, 1–17. <https://doi.org/10.1016/j.cscm.2024.e03012>.
- Fode, T.A., Abeid, Y., Jande, C., Kivevele, T., 2024c. Effect of natural pozzolana on physical and mechanical properties of concrete. *Adv. Civ. Eng.* 2024, 1–17. <https://doi.org/10.1155/2024/3356641>.
- Gour, C.P., Dhurvey, P., Shaik, N., 2022. Optimization and prediction of concrete with recycled coarse aggregate and bone china fine aggregate using response surface methodology. *J. Nanomater.* 2022. <https://doi.org/10.1155/2022/2264457>.
- Hakamy, A., Shaikh, F.U.A., Low, I.M., 2015. Characteristics of nanoclay and calcined nanoclay-cement nanocomposites. *Composites, Part B* 78, 174–184. <https://doi.org/10.1016/j.compositesb.2015.03.074>.
- Hamada, H.M., Skariah, B., Yahaya, F.M., Muthusamy, K., Yang, J., Abdalla, J.A., Hawileh, R.A., Pascal, M., 2021. Sustainable use of palm oil fuel ash as a supplementary cementitious material : a comprehensive review. *J. Build. Eng.* 40, 102286. <https://doi.org/10.1016/j.jobte.2021.102286>.
- Haq, I.U., 2022. P OLYPROPYLENE concrete containing bentonite & silica fume. In: *3rd Conf. Sustain. Civ. Eng.*.
- Hossain, M.U., Poon, C.S., Dong, Y.H., Xuan, D., 2018. Evaluation of environmental impact distribution methods for supplementary cementitious materials. *Renew. Sustain. Energy Rev.* 82, 597–608. <https://doi.org/10.1016/j.rser.2017.09.048>.
- Hu, L., He, Z., Zhang, S., 2020. Sustainable use of rice husk ash in cement-based materials: environmental evaluation and performance improvement. *J. Clean. Prod.* 264, 121744. <https://doi.org/10.1016/j.jclepro.2020.121744>.
- Jaskulski, R., Daria, J., 2020. Calcined clay as supplementary cementitious material. *Mater. MDPI*. <https://doi.org/10.3390/ma13214734>.
- Kadhim, M.J., Kamal, H.M., Hasan, L.M., 2022. Hydro-mechanical properties of cement mortar by usage of bentonite as partial cement replacement in press , accepted manuscript – note to user. *Int. J. Nanoelectron. Mater.*
- Karthikeyan, M., Ramachandran, P.R., Nandhini, A., Vinodha, R., 2015. Application on partial substitute of cement by bentonite in concrete. *Int. J. ChemTech Res.* 8, 384–388.
- Keke, L., Yong, L., Liuliu, X., Junjie, Z., Kangning, L., Dingqiang, F., 2022. Rheological characteristics of Ultra-High performance concrete (UHPC) incorporating bentonite. *Construct. Build. Mater.* 349, 128793. <https://doi.org/10.1016/j.conbuildmat.2022.128793>.
- Khan, M.N.N., Jamil, M., Karim, M.R., Zain, M.F.M., Kaish, A.B.M.A., 2017. Filler effect of pozzolanic materials on the strength and microstructure development of mortar. *KSCE J. Civ. Eng.* 21, 274–284. <https://doi.org/10.1007/s12205-016-0737-5>.
- Khan, K., Amin, M.N., Usman, M., Imran, M., Al-faiad, M.A., Shalabi, F.I., 2022. Effect of fineness and heat treatment on the pozzolanic activity of natural volcanic ash for its utilization as supplementary cementitious materials. *Crystals*. <https://doi.org/10.3390/cryst12020302>.
- Küçükylıdır, E., Uzal, B., 2014. Characteristics of calcined natural zeolites for use in high-performance pozzolan blended cements. *Construct. Build. Mater.* 73, 229–234. <https://doi.org/10.1016/j.conbuildmat.2014.09.081>.
- Laidani, Z.E., Benabed, B., Abousnina, R., Gueddouda, M.K., Kadri, E., 2020. Experimental investigation on effects of calcined bentonite on fresh , strength and durability properties of sustainable self-compacting concrete. *Construct. Build. Mater.* 230, 117062. <https://doi.org/10.1016/j.conbuildmat.2019.117062>.
- Laidani, Z.E.A., Benabed, B., Abousnina, R., Gueddouda, M.K., Khatib, M.J., 2022. Potential pozzolanicity of Algerian calcined bentonite used as cement replacement optimisation of calcination temperature and effect on strength of self-compacting mortars. *Eur. J. Environ. Civ. Eng.* 26, 1379–1401. <https://doi.org/10.1080/19648189.2020.1713898>.
- Lima-Guerra, D.J., Mello, I., Resende, R., Silva, R., 2014. Use of bentonite and organobentonite as alternatives of partial substitution of cement in concrete manufacturing. *Int. J. Concr. Struct. Mater.* 8, 15–26. <https://doi.org/10.1007/s40069-013-0066-8>.

- Mesbouda, N., Benyounes, K., 2021. Calcinated bentonite as supplementary cementitious materials in cement-based mortar. *J. Appl. Eng. Sci.* 11, 23–32. <https://doi.org/10.2478/jaes-2021-0004>.
- Milović, T., Šupić, S., Malešev, M., Radonjanin, V., 2022. The effects of natural zeolite as fly ash alternative on frost resistance and shrinkage of blended cement mortars. *Sustain. Times* 14. <https://doi.org/10.3390/su14052736>.
- Mirza, J., Riaz, M., Naseer, A., Rehman, F., Khan, A.N., Ali, Q., 2009. Pakistani bentonite in mortars and concrete as low cost construction material. *Appl. Clay Sci.* 45, 220–226. <https://doi.org/10.1016/j.clay.2009.06.011>.
- Mohammed, B.S., Haruna, S., Mubarak bn Abdul Wahab, M., Liew, M.S., 2019. Optimization and characterization of cast in-situ alkali-activated pastes by response surface methodology. *Construct. Build. Mater.* 225, 776–787. <https://doi.org/10.1016/j.conbuildmat.2019.07.267>.
- Mushtaq, S.F., Ali, A., Khushnood, R.A., Tufail, R.F., Majidi, A., Nawaz, A., Durdyev, S., Doru, D., Nergis, B., Ahmad, J., 2022. Effect of bentonite as partial replacement of cement on residual properties of concrete exposed to elevated temperatures. *Sustain. Times*.
- Myers, C.M.A.R.H., Montgomery, D.C., 2016. *Response Surface Methodology: Process and Product Optimization Using Designed Experiments*.
- Nikhil K, K., Ajay A, H., 2015. Evaluation of strength of plain cement concrete with partial replacement of cement by meta kaolin & fly ash. *Int. J. Eng. Res.* 4. <https://doi.org/10.17577/ijertv4is050860>.
- Pacewska, B., Wilińska, I., 2020. Usage of supplementary cementitious materials : advantages and limitations. *J. Therm. Anal. Calorim.* 142, 371–393. <https://doi.org/10.1007/s10973-020-09907-1>.
- Park, S., Wu, S., Liu, Z., Pyo, S., Tarantino, M., 2021. Materials the role of supplementary cementitious materials (SCMs) in ultra. High Perform. Concr. (UHPC): A Review. <https://doi.org/10.3390/ma14061472>.
- Raghav, M., Park, T., Yang, H., Lee, S., Karthick, S., Lee, H., 2021. Review of the effects of supplementary cementitious materials and chemical additives on the physical , mechanical and durability properties of hydraulic concrete. *MDPI- Mater.* <https://doi.org/10.3390/ma14237270%0A>.
- Reddy, S.S., Reddy, M.A.K., 2021. Optimization of calcined bentonite caly utilization in cement mortar using response surface methodology. *Int. J. Eng. Trans. A Basics.* 34, 1623–1631. <https://doi.org/10.5829/IJE.2021.34.07A.07>.
- Rehman, S.U., Yaqub, M., Noman, M., Ali, B., Nasir, M., Khan, A., Fahad, M., Abid, M.M., Gul, A., 2019a. The influence of thermo-mechanical activation of bentonite on the mechanical and durability performance of concrete. *Appl. Sci. MDPI.* <https://doi.org/10.3390/app9245549>.
- Rehman, S.U., Yaqub, M., Ali, T., Shahzada, K., Khan, S.W., Noman, M., 2019b. Durability of mortars modified with calcined montmorillonite clay. *Civ. Eng. J.* 5, 1490–1505. <https://doi.org/10.28991/cej-2019-03091347>.
- Ruan, S., Unluer, C., 2017. Influence of supplementary cementitious materials on the performance and environmental impacts of reactive magnesia cement concrete. *J. Clean. Prod.* 159, 62–73. <https://doi.org/10.1016/j.jclepro.2017.05.044>.
- Salamatpoor, S., Jafarian, Y., Hajiannia, A., 2018. Physical and mechanical properties of sand stabilized by cement and natural zeolite. *Eur. Phys. J. Plus.* 133. <https://doi.org/10.1140/epjp/i2018-12016-0>.
- Scrivener, K.L., John, V.M., Gartner, E.M., 2018. Eco-efficient cements: potential economically viable solutions for a low-CO2 cement-based materials industry. *Cement Concr. Res.* 114, 2–26. <https://doi.org/10.1016/j.cemconres.2018.03.015>.
- Shafiq, N., Nuruddin, M.F., Khan, S.U., Ayub, T., 2015. Calcined kaolin as cement replacing material and its use in high strength concrete. *Construct. Build. Mater.* 81, 313–323. <https://doi.org/10.1016/j.conbuildmat.2015.02.050>.
- Sriwattanapong Photisan, M., 2018. Influence of the calcination thickness of kaolin on strength development of mortars. In: *Knowl. Innov. Eng. Sci. Technol.*, pp. 1–11. <https://doi.org/10.33422/4kiconf.2018.12.22>.
- Taklymi, S.M.Q., Rezaifar, O., Gholhaki, M., 2020. Investigating the properties of bentonite and kaolin modified concrete as a partial substitute to cement. *SN Appl. Sci.* 2, 1–14. <https://doi.org/10.1007/s42452-020-03380-z>.
- Tayeh, B.A., 2018. Effects of marble , timber , and glass powder as partial replacements for cement. *J. Civ. Eng. Constr.* 2, 63–71.
- Taylor-Lange, S.C., Lamon, E.L., Riding, K.A., Juenger, M.C.G., 2015. Calcined kaolinite-bentonite clay blends as supplementary cementitious materials. *Appl. Clay Sci.* 108, 84–93. <https://doi.org/10.1016/j.clay.2015.01.025>.
- Tebbal, N., El Abidine Rahmouni, Z., 2019. Rheological and mechanical behavior of mortars with metakaolin formulation. *Procedia Comput. Sci.* 158, 45–50. <https://doi.org/10.1016/j.procs.2019.09.026>.
- Tironi, A., Castellano, C.C., Bonavetti, V.L., Trezza, M.A., Scian, A.N., Irassar, E.F., 2014. Kaolinitic calcined clays - Portland cement system: hydration and properties. *Construct. Build. Mater.* 64, 215–221. <https://doi.org/10.1016/j.conbuildmat.2014.04.065>.
- Trümer, A., Ludwig, H.M., Schellhorn, M., Diedel, R., 2019. Effect of a calcined Westerwald bentonite as supplementary cementitious material on the long-term performance of concrete. *Appl. Clay Sci.* 168, 36–42. <https://doi.org/10.1016/j.clay.2018.10.015>.
- Vijay, C., Achyutha Kumar Reddy, M., 2021. Optimization of bentonite modified cement mortar parameters at elevated temperatures using RSM. *IOP Conf. Ser. Mater. Sci. Eng.* 1197, 012040. <https://doi.org/10.1088/1757-899x/1197/1/012040>.
- Ibrahim, A.M., Wahab, K.A.A., 2008. Effect of temperature on the pozzolanic properties of Metakaolin produced from Iraqi kaolin clay. *AL- Fatih J. Diyala Univ. Iraq.* 4, 268–285. <https://www.iasj.net/iasj?func=article&aId=17256>.
- Wei, J., Gencturk, B., 2019. Cement and Concrete Research Hydration of ternary Portland cement blends containing metakaolin and sodium bentonite. *Cement Concr. Res.* 123, 105772. <https://doi.org/10.1016/j.cemconres.2019.05.017>.
- Xie, Y., Li, J., Lu, Z., Jiang, J., Niu, Y., 2019. Preparation and properties of ultra-lightweight EPS concrete based on pre-saturated bentonite. *Construct. Build. Mater.* 195, 505–514. <https://doi.org/10.1016/j.conbuildmat.2018.11.091>.
- Yu, C., Li, G., Gao, J., Lan, B., Wei, Q., Xu, D., 2013. Effect of bentonite on the performance of the limestone manufactured-sand mortar. *Appl. Mech. Mater.* 360, 1374–1378. <https://doi.org/10.4028/www.scientific.net/AMM.357-360.1374>.
- Zhang, Y., Zhang, J., Luo, W., Wang, J., Shi, J., Zhuang, H., Wang, Y., 2019. Effect of compressive strength and chloride diffusion on life cycle CO 2 assessment of concrete containing supplementary cementitious materials. *J. Clean. Prod.* 218, 450–458. <https://doi.org/10.1016/j.jclepro.2019.01.335>.