

Optimization of Calcined Bentonite Utilization In Cement Concrete Using Response Surface Methodology

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Abstract – Finding an alternative to the pozzolanic materials developed from wastes from industries was needed as of its unattainability when the industries were shut down forever. This paper aims at optimization of calcined bentonite utilization in concrete using Response Surface Methodology (RSM). The microstructure of calcined bentonite was analyzed through SEM and XRD. The calcined bentonite replacement made with cement varied from 0% to 30% at an interval of 5% in concrete. Compressive strength split tensile strength, flexural strength, and rapid chloride ion penetration of tests were carried out to determine the strength and durability of calcined bentonite incorporated concrete. Specimens were cast and tested to failure for all compositions of mixes for curing periods of 7, 28, and 90 days. The modeling and optimization were carried out using Design-Expert software. The comparison was made for the performance of bentonite blended concrete with conventional concrete in terms of strength and durability. 5 percentage of bentonite incorporated concrete shown better performance among all mixes for both strength and durability of concrete. The models created from RSM are significant in all the factors considered. The optimum solution was obtained by 2.09 % of calcined bentonite substitution after 63.91 days of curing with 1.00 desirability.

Keywords - Calcined Bentonite, Compressive Strength, Rapid chloride penetration, Response Surface Methodology, Optimization.

I. INTRODUCTION

The production of cement generates an enormous amount of CO₂ into the environment. Presently, Industrial wastes are using as pozzolonic materials in concretes like fly ash, GGBS and silica fume, etc., to get better strength and durability properties [1,2]. So much research is being done with different pozzolonic materials replacing cement [3,4]. There are two types of pozzolonic materials, in a broader sense, that can be utilized as cement replacement [5–7]. One is natural pozzolonic materials like bentonite, and others are waste by-products from industries like flyash[8,9]. Nowadays, fly ash utilization in concrete became a very common practice [3]. Coal-based thermal power plants generate most of the fly ash. The unit cost of electricity generation is very high through thermal power

plants comparatively than other sources [10,11]. Thermal power plants produce a huge amount of CO₂ emissions into the environment. The production of fly ash causes damage to the environment [12,13]. The pozzolonic materials will not be generated once those industries got shut down permanently.

Bentonite is a lump of clay and had plenty of applications in various fields. The major applications that include the utilization of bentonite are drilling fluids, foundry bonds, pelletizing iron ore, cat litter, and absorbents [14,15]. It contains silica in rich amounts, obeys the pozzolanic properties, which can be used instead of industrial wastes [16,17]. CO₂ emissions into the environment can be reduced by incorporating bentonite in concrete production [18]. Investigations were done to investigate the effect of bentonite on the mechanical properties and durability of concrete [19,20]. The physicochemical properties of bentonite were changing based on the source of collection. This leads to ambiguity in the performance of bentonite incorporated concrete [8,21]. Few attempts were made to evaluate the effect of Pakistani bentonite heated up to 950°C. A decrease in strength and durability for cement mortar and concrete was observed [5,22].

Response Surface Methodology (RSM) was used for the generation of modeling. RSM has also been applied to carry out multi-objective optimization in different concrete materials [23]. Bashar et al. has done multi-objective optimization to find the relation between variables and responses of the properties of roller-compacted concrete [24]. Long et al. has done multi-objective optimization by restraining action of corrosion, fatigue & fiber content as independent variables and compressive strength, flexural strength & dynamic elastic modulus as responses [25,26]. This paper aims to find the optimum calcined bentonite content available at 17°14'27"N and 77°35'14"E in the southern region of India can be used in concrete.

II. EXPERIMENTAL PROGRAM

A. Materials

In this experimental work, ordinary Portland cement of 53-grade cement was used, which was conforming as per IS:12269-1987. Fine aggregate was used, which was conforming as per IS:383-2016. Coarse aggregate of sizes



10 to 12mm and 20 mm was used. The physical properties of coarse aggregate are tested as per IS2386-1963. The calcination of bentonite (at 850°C) was done with a muffle furnace of 1400°C as maximum temperature and 1200°C working temperature, displayed in Figure 1. The chemical analysis of calcined bentonite was mentioned in Table1. The color of calcined bentonite was brownish, and the specific gravity of the bentonite is 2.44. The microstructure of bentonite was analyzed by using an X-Pert X-ray Diffractometer of PANalytical, model number: PW 3040/00. The XRD analysis was performed. The analysis was conducted at a 2theta range of 10-80 with a step size of 0.5 and a scan rate of 5 degrees/min. Figure 2. shows the presence of different phases, which can be further identified as 5 different mineral crystalline structures. Scanning electron microscopy (SEM) images were obtained by using Nova Nano SEM / FEI for further pursuit. Figure 3. have also shown the presence of crystalline structures in the bentonite sample.

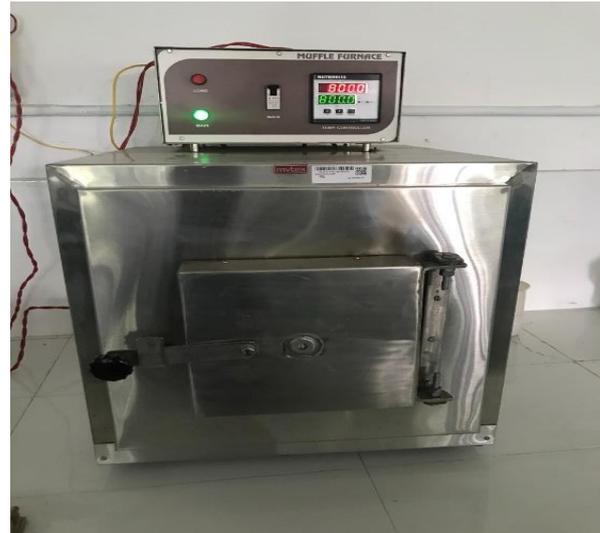


Fig. 1 Muffle furnace

Table 1. Chemical analysis of calcined bentonite sample

S. No.	Component	% Mass
1	SiO ₂	51.11
2	Al ₂ O ₃	16.38
3	CaO	7.12
4	MgO	7.57
6	Fe ₂ O ₃	7.65
7	K ₂ O	1.34
8	Na ₂ O	0.29
9	P ₂ O ₅	0.29
10	MnO	0.14
11	V ₂ O ₅	0.07
12	TiO ₂	1.29
13	LoI*	6.75

* Loss on Ignition

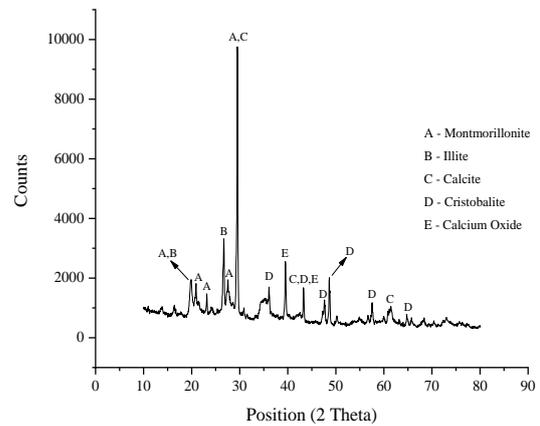


Fig. 2 XRD analysis of calcined bentonite

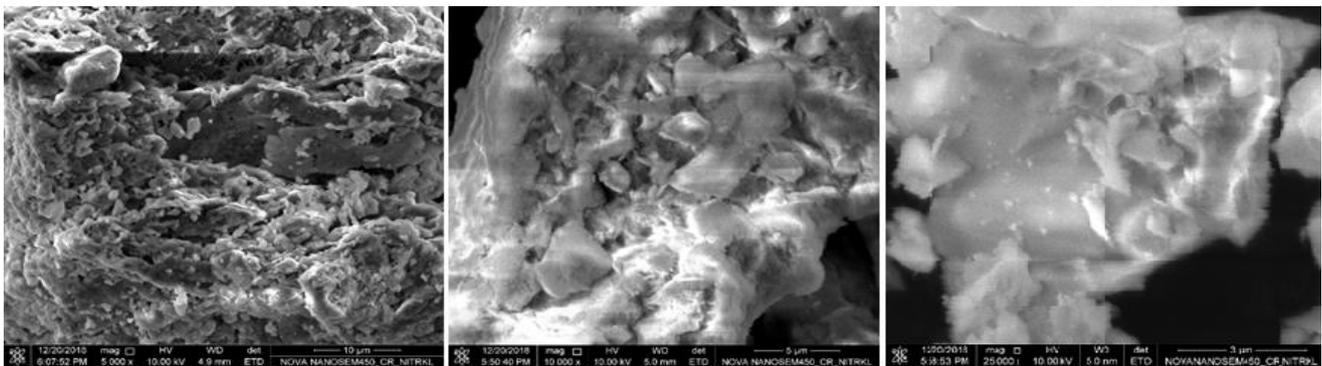


Fig. 3 SEM analysis of calcined Bentonite

Table 2. Details of all mixes

S. No	Mix Id	Cement	Calcined Bentonite	Fine Aggregate	Coarse Aggregate	W/C Ratio
1	CC	380	0	890.36	1045.20	0.45
2	M1	361	19	888.76	1042.96	0.45
3	M2	342	38	886.50	1040.78	0.45
4	M3	323	57	884.08	1037.80	0.45
5	M4	304	76	881.97	1034.76	0.45
6	M5	285	95	879.34	1032.52	0.45
7	M6	266	114	877.12	1030.12	0.45

B. Testing of specimens

The concrete mix design was prepared as per the standard procedure IS 10262, the details of all mixes were displayed in Table2. A total number of 63 concrete cubes (150x150x150mm), cylinders (150x300mm), and beam (150x150x700mm) specimens were cast and tested to failure at curing ages of concrete (7, 28, and 56 days) to determine the compressive strength, split tensile strength and flexural strength as per the standard producers IS516, IS5816, and IS516 respectively. A rapid chloride ion penetration test was performed to determine chloride ion penetration of concrete as per standard code ASTM C 1202-97. A total number of 84 specimens were cast, tested against chloride ion permeability after 7, 28, and 56 curings, respectively. The test setups of Compressive strength split tensile strength, flexural strength, and RCPT were displayed in Figures 4-7, respectively.



Figure 4 Setup for compression strength test



Figure 5 Test setup of split tensile strength



Figure 6 Test setup of flexural strength



Figure 7 Setup for a rapid chloride penetration test.

III. RESULTS AND DISCUSSIONS

The rate of loading was used as 2 kN/S for the compressive testing machine and flexural testing machine. Results were taken after testing each specimen at 7 days, 28 days, and 56 days of curing.

A. Compressive strength

The compressive strength of all specimens was calculated. Figure 8. displays the compressive strengths of mixes after 7 days, 28 days, and 56 days curing. CC exhibits more compressive strength at 7days curing, M1 shows better compressive strength, whereas remaining mixes show poor compressive strength comparatively with CC after 28days curing. M1 & M2 exhibits the highest compressive strength than CC after 90 curings. This attributes the enhancement of pozzolanic activity and improvement of C-S-H formation upon appropriate substitution of bentonite in concrete.

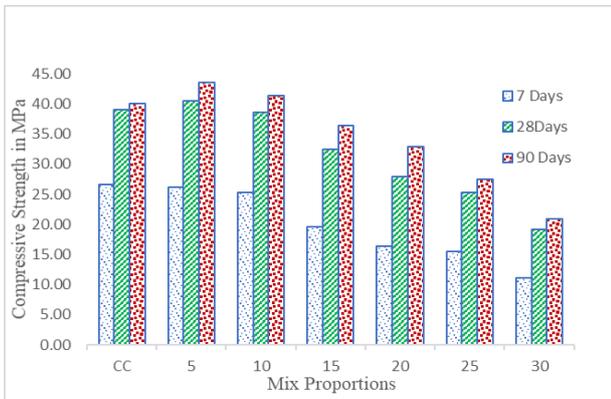


Fig. 8 Compression strength test results

B. Split tensile strength

The split tensile strength for all specimens was calculated; Fig. 9 shows the split tensile strengths of all mixes. The behavior of concrete was not changed drastically compared with compressive strength. A slight improvement was observed for split tensile strength upon 5% addition of calcined bentonite to concrete. Lower split tensile strength was observed for all calcined bentonite blended mixes for 7 days and 28 days of curing compared with control concrete. M1 exhibits more split tensile strength than control concrete after 90 days of curing. The reason behind this may be the occurrence of pozzolanic reaction since rich SiO₂ present in calcined bentonite.

C. Flexural strength

The flexural strength of all specimens was calculated in Figure 10. displays the flexural strengths of mixes after 7 days, 28 days, and 56 days curing. No difference in the behavior was of concrete was observed. CC exhibits more flexural strength among all mixes after 7 days of curing. Same flexural strength was observed for CC & M1 after 28 days of curing; lower flexural strengths were observed for remaining mixes. M1 exhibits a bit more flexural than among all mixes. This attribute less effect of pozzolanic reaction occurrence in flexural strength of concrete.

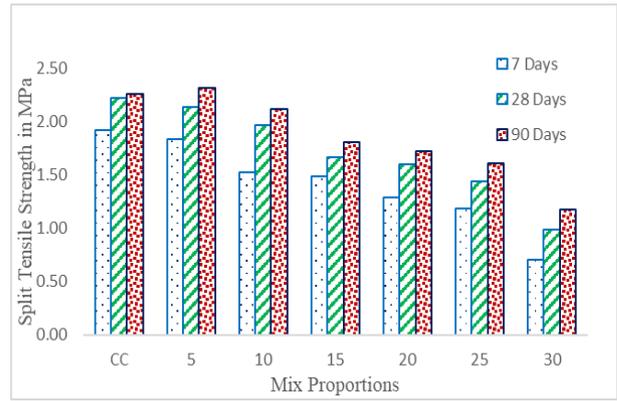


Fig. 9 Split tensile strength test results

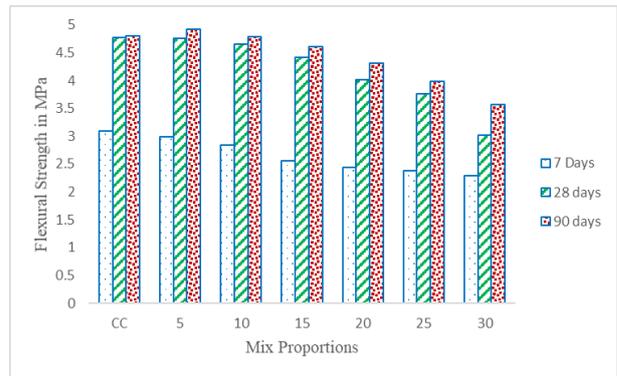


Fig. 10 Flexural strength test results

D. Rapid chloride penetration test

The chloride ion penetration was assessed by taking the average of 4 channels for all mixes; Fig. 9 shows the charge passed through the concrete of all mixes. Lower resistance was observed for all mixes after 7 days of curing, and CC exhibits the better resistance against chloride ion among all mixes. Concrete resistance against chloride ion was improved for calcined bentonite blended mixes after 28 days and 90 days curing; M1 & M2 shown better resistance among all mixes. The reason behind this may be the occurrence of pozzolanic reacting and effective formation of C-S-H gel upon the addition of calcined bentonite. The pore-filling was developed by the addition of calcined bentonite since it was finer than cement, and it fills the voids between the cement particles.

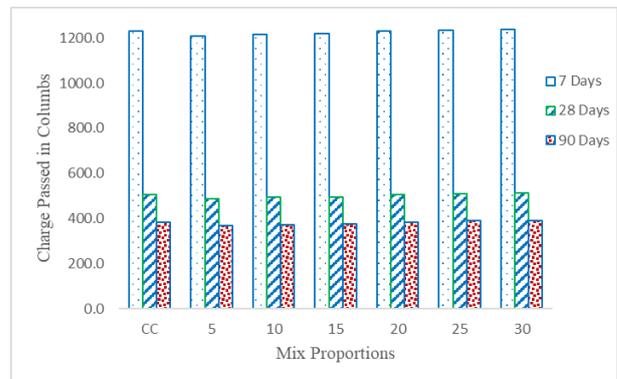


Fig. 11 Chloride ion penetration test results

IV. RSM modeling and optimization

A. Development of model

In RSM, different kinds of models are obtainable, like Box Behnken, Central Composite, and Optimal in a randomized design. The selection of the model will depend on the nature of the variable and responses. In this investigation, the central composite type of model was employed for the model development and optimization.

B. Mix matrix design

Design-Expert software was used for preparing the model and optimization. The design of experiments was done by considering two variables (bentonite replacement and age of curing). Six levels of bentonite replacement (0%, 5%, 10%, 15%, 20%, 25% and 30%) and three levels of age of curing (7,28, and 90 days) were used. A 21 combinations of mixes were formed in RSM, and Table 3 displays the details of all mixes. The responses for calcined bentonite blended concrete (compressive strength, split tensile strength, flexural strength, and charge passed) were determined for all mixtures, considered for RSM analysis and optimization

C. Analysis of variance

The summaries of ANOVA for the responses studied, F-values are 104.32, 92.34, 101.47, and 18422.58 for compressive strength, split tensile strength, flexural strength, and charge passed, respectively pointing all models to be significant, displayed in Table 4-7. The quadratic model was used for the determination of all variables. In all models, all terms are significant apart from AB. The final models for all responses were given in Eqs. 1-4, respectively. A 3-dimensional (3D) response surface plot was used to illustrate the relationship between responses and independent variables. Figure 12-15 shows the 3D response surface plots illustrating the relationship between responses and independent variables for all mixes.

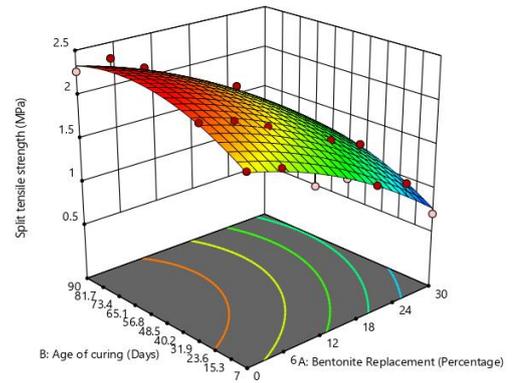


Figure 13. Split tensile strength

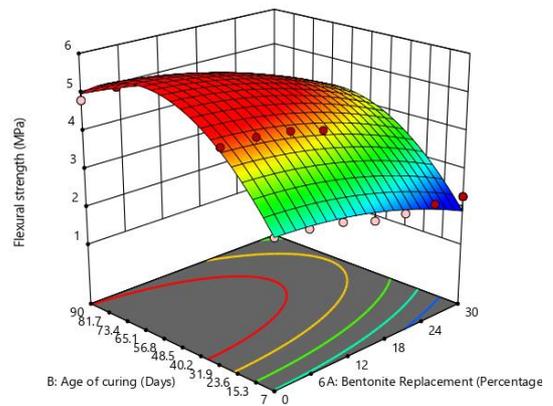


Figure 14. flexural strength

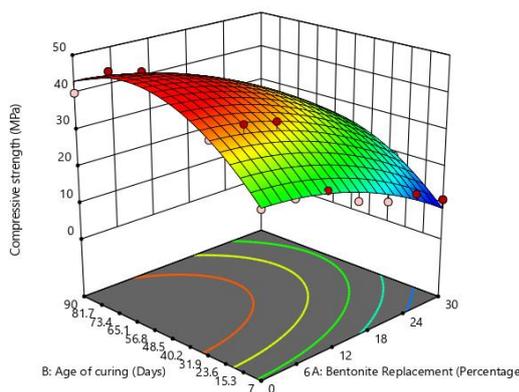


Figure 12. Compressive strength

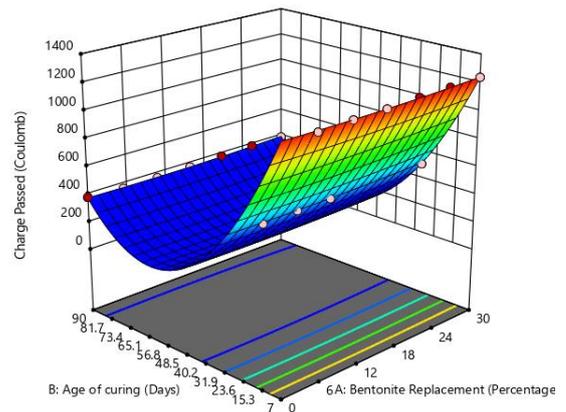


Figure 15. Charge passed

Compressive strength = +21.69345 - 0.042889*A + 0.795806*B - 0.001307*AB - 0.018624*A² - 0.006196*B² Eq.1

Split tensile strength = +1.78810 - 0.018434*A + 0.018709*B + 0.000017*AB - 0.000658*A² - 0.000141*B² Eq.2

Flexural strength = +2.41724 - 0.002931*A + 0.104145*B - 0.000084*AB - 0.001216*A² - 0.000840*B² Eq.3

Charge Passed = +1541.39637 - 1.06952*A - 48.15630*B - 0.000774*AB + 0.054802*A² + 0.391629*B² Eq.4

Table 3. Details of all combinations of variables.

	F1	F2	R1	R2	R3	R4
Run	Bentonite Percentage	Days of Curing	Compressive Strength	Split tensile strength	Flexural strength	Charge Passed
1	0	7	26.67	1.92	3.1	1229.5
2	5	7	26.22	1.84	2.99	1209.3
3	10	7	25.33	1.53	2.84	1216.5
4	15	7	19.56	1.49	2.56	1219.5
5	20	7	16.44	1.29	2.44	1229.5
6	25	7	15.56	1.19	2.38	1233.3
7	30	7	11.11	0.71	2.29	1238.8
8	0	28	39.11	2.22	4.78	507.0
9	5	28	40.44	2.14	4.76	488.3
10	10	28	38.67	1.97	4.65	494.3
11	15	28	32.44	1.67	4.42	495.0
12	20	28	28.00	1.60	4.01	506.5
13	25	28	25.33	1.44	3.76	508.0
14	30	28	19.11	0.99	3.02	511.5
15	0	90	40.00	2.26	4.81	383.5
16	5	90	43.56	2.32	4.92	369.8
17	10	90	41.33	2.12	4.79	370.3
18	15	90	36.44	1.81	4.61	374.8
19	20	90	32.89	1.73	4.32	383.5
20	25	90	27.56	1.61	3.99	389.5
21	30	90	20.89	1.17	3.57	390.5

Table 4. ANOVA for compressive strength

ANOVA for Quadratic model, R1: Compressive strength						
Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1796.44	5	359.29	104.32	< 0.0001	significant
A-Bentonite Replacement	895.06	1	895.06	259.88	< 0.0001	
B-Age of curing	739.91	1	739.91	214.83	< 0.0001	
AB	4.46	1	4.46	1.29	0.2732	
A ²	54.63	1	54.63	15.86	0.0012	
B ²	280.87	1	280.87	81.55	< 0.0001	
Residual	51.66	15	3.44			
Cor Total	1848.10	20				

Table 5. ANOVA for split tensile strength

ANOVA for Quadratic model, R2: Split tensile strength						
Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	3.72	5	0.7432	92.34	< 0.0001	significant
A-Bentonite Replacement	2.82	1	2.82	350.62	< 0.0001	
B-Age of curing	0.6739	1	0.6739	83.73	< 0.0001	
AB	0.0008	1	0.0008	0.0977	0.7589	
A ²	0.0683	1	0.0683	8.48	0.0107	
B ²	0.1456	1	0.1456	18.09	0.0007	
Residual	0.1207	15	0.0080			
Cor Total	3.84	20				

Table 6. ANOVA for flexural strength

ANOVA for Quadratic model, R3: Flexural strength						
Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	17.13	5	3.43	101.47	< 0.0001	significant
A-Bentonite Replacement	3.83	1	3.83	113.32	< 0.0001	
B-Age of curing	11.00	1	11.00	325.84	< 0.0001	
AB	0.0184	1	0.0184	0.5443	0.4720	
A ²	0.2328	1	0.2328	6.90	0.0191	
B ²	5.17	1	5.17	153.08	< 0.0001	
Residual	0.5064	15	0.0338			
Cor Total	17.63	20				

Table 7. ANOVA for charge passed

ANOVA for Quadratic model, R4: Charge passed						
Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	2.923E+06	5	5.846E+05	18422.58	< 0.0001	significant
A-Bentonite Replacement	583.56	1	583.56	18.39	0.0006	
B-Age of curing	2.499E+06	1	2.499E+06	78736.36	< 0.0001	
AB	1.56	1	1.56	0.0493	0.8273	
A ²	473.01	1	473.01	14.91	0.0015	
B ²	1.122E+06	1	1.122E+06	35357.81	< 0.0001	
Residual	476.02	15	31.73			
Cor Total	2.924E+06	20				

Table 8. Criteria setting for optimization

Variables/Responses	Goal	Lower Limit	Upper Limit
Bentonite replacement	In range	0	30
Age of curing	In range	7	28
Compressive strength	Maximize	11.11	43.55
Split tensile strength	Maximize	0.70	2.32
Flexural strength	Maximize	2.29	4.92
Charge passed	Minimize	369.75	1238.75

Table 9. Optimized calcined mixes

Bentonite replacement	Age of curing	Compressive strength	Split tensile strength	Flexural strength	Charge passed
2.09	63.91	46.89	2.36	5.61	61.23

D. Optimization

Optimization was performed by using Design Expert Software. Table 8 shows the upper and lower limits that were set to perform the optimization. Table 5 displays the optimization results of calcined bentonite mixes. The optimized mix was achieved at 2.09 % of substitution of bentonite after 63.91 days of curing. The optimum values of responses were 46.89 MPa compressive strength, 2.36 split tensile strength, 5.61 flexural strength, and 61,23 charge passed.

V. CONCLUSIONS

The following conclusions were made based on the experimental work,

- Compressive strength was increased by the incorporation of 5% and 10% calcined bentonite in concrete upon 90 days of curing.
- Improvement in the split tensile strength was observed for 5% calcined bentonite substitution in concrete and 90 days curing.
- Flexural strength was increased by the addition of 5 % calcined bentonite to the concrete after 90 days of curing
- Resistance against chloride ion of concrete was improved upon the incorporation of 5% and 10% calcined bentonite in concrete after both 28 days and 90 days of curing.
- The models created from RSM are significant in all the factors considered. The optimum solution was obtained by 2.09 % of calcined bentonite substitution after 63.91 days of curing with 1.00 desirability.

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