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Optimization of Calcined Bentonite Clay Utilization in Cement Mortar using Response Surface Methodology

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ABSTRACT

Discovery of alternative to the pozzolanic materials generated from industrial wastes was needed because of its unavailability when the industries was shutdown permanently. This paper deals the optimization of calcined bentonite clay utilization in cement mortar using response surface methodology (RSM). The variables were taken as three levels of calcination temperature (room temperature, 700^{0}C and 800^{0}C) and seven levels of calcined bentonite (0%, 5%, 10%, 15%, 20%, 25% and 30%). The compressive strength, workability, strength activity index and sorpitivity were taken as responses. The fresh and hardened properties of all determined for all mixes. Design Expert 11.0 version was utilized to carried out modelling and optimization using RSM. Workability was decreased upon increasing the calcination temperature and bentonite content in cement mortar. This attributed to high water absorption capacity of bentonite. The peak compressive strength was displayed by 20% replaced bentonite calcined at 800^{0}C cement mortar after 28 days curing. Strength activity was improved upon increasing the percentage of bentonite calcined at 800^{0}C. The sorpitivity of cement mortar was improved by incorporation of bentonite calcined at 800^{0}C. The generated models from RSM were significance in all the factors considered. Optimum performance of the responses was observed at 15.25 % bentonite substitution calcined at 800^{0}C

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NOMENCLATURE

Degree Celcius	^{0}C	*Mega Pascal*	*MPa*
Kilo Newton	kN	Analysis of Variance	ANOVA

1. INTRODUCTION

Utilization of pozzolanic materials will enhance the fresh and hardened properties of cement mortar and concrete [1]. Presently, fly ash, GGBS, silica fume, metakaolin etc. are utilizing as a pozzolanic materials in concrete for better performance as well as reduction of CO_2 emissions to the environment [2]. Fly ash is one of the discarded materials of thermal power plants. It is one of the widely using pozzolanic materials in construction industry [3]. The performance of fresh and hardened properties of concrete will enhance by incorporation of fly ash in concrete [4]. Fly ash will not be generated once the thermal power plants is permanently shutdown. So, the discovery of alternative to the pozzolonic materials generated from industrial wastes was needed.

Bentonite is a natural pozzolonic material, which can be used instead of industrial wastes in concrete [5, 6]. A few research was available on the evaluation of the properties of bentonite incorporated cement mortar [6-8] as well as concrete [9, 10]. The fresh and hardened properties of concrete was increased by incorporation of bentonite in concrete [11, 12]. Few examinations were done to assess the combination of bentonite and other pozzolonic materials [13, 14]. The ambiguity in the effect of bentonite was observed based on it's source of collection and process of treatment [15, 16]. therefore, investigation on process of treatment of bentonite is needed to estimate the influence on bentonite.

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Calcination is a method of heating a material under regulated temperature and in a monitored environment. A few investigations were made to evaluate the effect of calcined bentonite on strength and durability of cement mortar and concrete as well as self-compacting concrete which is available in Pakistan [17-19].

Mirza et. Al. [20] initiated the research on the effect of calcined bentonite on cement mortar and concrete by considering 150^0C, 250^0C, 500^0C, 750^0C and 950^0C as calcination temperatures. Apart from the effect of bentonite calcined at 150^0C on cement mortar and concrete, no results or discussion was made on the effect of the remaining calcination temperatures. The research on the same topic was extended by Ahmad et. al. [21]. They considered the calcination temperatures as 500^0C and 900^0C, evaluated the fresh and hardened properties of cement mortar and concrete. It was observed almost the same strength activity for raw bentonite and bentonite calcined at 500^0C. The strength activity was drastically reduced by incorporation of bentonite calcined at 900^0C. Moreover, no optimal solution was provided in terms of bentonite substitution or calcination temperature of bentonite [21].

Optimization included the utilization of Response Surface Methodology (RSM) employing theories of mathematical and statistical analysis techniques between variables and responses [22]. RSM was utilized for the optimization of the wanted set of objectives, either independent variables or responses, to implement multi-objective optimization in different concrete materials [23, 24]. Mohammed et al. [25] performed multi-objective optimization to accomplish a connection between variables and responses of the properties of roller-compacted concrete by keeping fly ash content as constant, considering the combined effect of both crumb rubber and nano-silica [26]. This research aimed at the optimization of calcined bentonite usage in cement mortar by using RSM. The bentonite substitution (0%, 5%, 10%, 15%, 20%, 25% and 30%) and calcination temperatures (RT, 700^0C and 800^0C) were taken as variables. Compressive strength, workability, strength activity index and sorpitivity were considered as responses

2. EXPERIMENTAL PROGRAM.

2. 1. Materials OPC 53 grade cement was used in experimental work, the properties are within the permissible limits as per the standard procedure IS12269. Stand sand was used in the determination of compressive strength, the properties are within the permissible limits as per the standard procedure IS650. Bentonite was collected form Tandur (17°14′27″N and 77°35′14″E), southern region of India. The calcination of the bentonite was done with muffle furnace having maximum temperature of 1500^0C and working temperature of 1200^0C. The chemical composition of bentonite was displayed in Table 1. The XRD analysis was performed by using X-Pert X-ray Diffractometer of PANalytical, model number: PW 3040/00. Five mineral crystalline structures was observed upon analysis, Figure 1 exhibits the different phases. Scanning electron microscopy (SEM) images were obtained by using Nova Nano SEM/ FEI for further pursuit. It was observed that the crystalline structure by studying the images displayed in Figure 2.

2. 2. Testing of Specimens Normal consistency, initial setting time and final setting time tests for all mixes was conducted as per the standard procedure IS4031. The workability of mixes was tested as per the standard procedure IS 4031 Part-7.

TABLE 1. Chemical analysis of bentonite sample

S. No.	Component	% Mass
1	SiO_2	51.11
2	Al_2O_3	16.38
3	CaO	7.12
4	MgO	7.57
6	Fe_2O_3	7.65
7	K_2O	1.34
8	Na_2O	0.29
9	P_2O_5	0.29
10	MnO	0.14
11	V_2O_5	0.07
12	TiO_2	1.29
13	LoI*	6.75

* Loss on Ignition

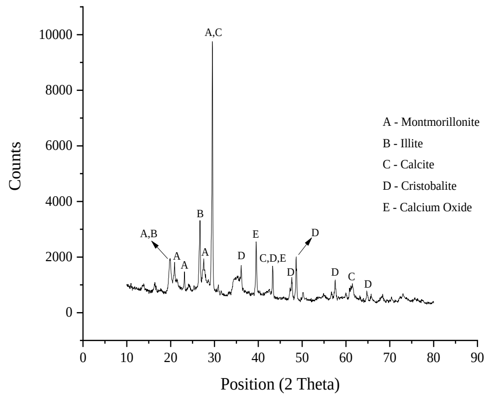

Figure 1. XRD Analysis of Bentonite

A total number of 189 cement mortar cubes were cast and tested to failure after 3,7 and 28 days curing as per the standard procedure IS 4031 Part-6. Testing of all specimens was done with compression testing machine of maximum capacity 3000 kN and rate of loading as1kN/s. The strength activity index test was conducted as per the standard procedure ASTM C311. The sorpitivity test was conducted as per the standard procedure ASTM C1585 – 20.

The sorpitivity test was conducted as per the standard procedure ASTM C1585 – 20. The proportions of all mixes were calculated as per the standard procedure IS 4031 Part-6, displayed in Table 2

3. RESULTS AND DISCUSSION

Analysis was performed for the values taken after testing the specimens of all mixes as per the standards.

3. 1. Normal Consistency, Initial & Final Setting The normal consistency of all mixes is shown in Figure 3. Rise in the normal consistency was observed upon increasing calcined bentonite addition. Initial and final setting times of all mixes was displayed in Figures 4. and 5. Increase in the initial and final setting times were observed by addition of bentonite. This attribute the high-water absorption capacity of bentonite and removal of moisture in bentonite upon calcination.

3. 2. Workability The workability of calcined bentonite mortar was summarized, displayed in Figure 6. The workability was decreased upon increasing the percentage of calcined bentonite addition. This may be the result of high-water demand of bentonite, it will increase by calcination process.

3. 3. Compressive Strength The compressive strength of all mixes was calculated, the results were summarized in Figures 7, 8 and 9 for 3, 7 and 28 days of curing, respectively. Less compressive strength was noticed at 3 days curing calcined bentonite blended cement mortar mixes. Compressive strength was improved at 7 days curing for calcined bentonite at 800^0C among all mixes. The cement mortar exhibits highest compressive strength for 20 % bentonite substitution and calcined at 800^0C temperature. This attribute the pore filling effect between cement particles by bentonite addition, the particle size of calcined bentonite was less than cement. The secondary C-S-H gel formation at lateral ages of cement mortar because bentonite is a pozzolanic material. Calcination at 800^0C eliminates the impurities of bentonite, leads to enhancement of compressive strength at lateral ages.

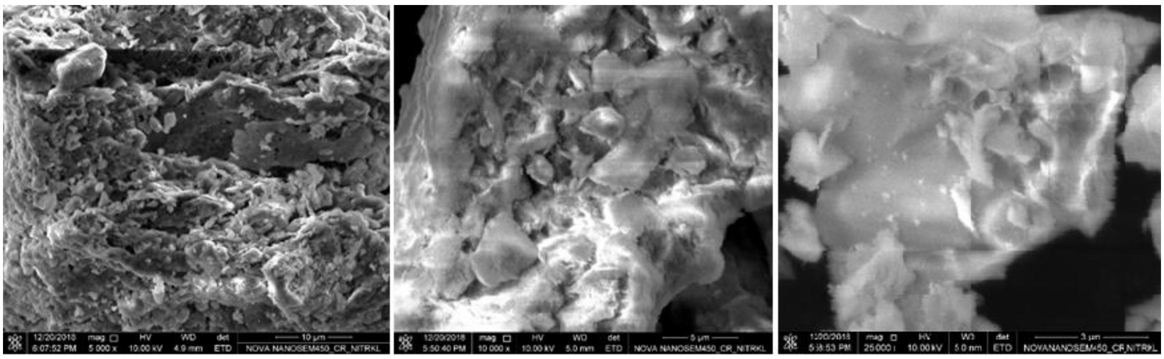

Figure 3. SEM Analysis of Bentonite

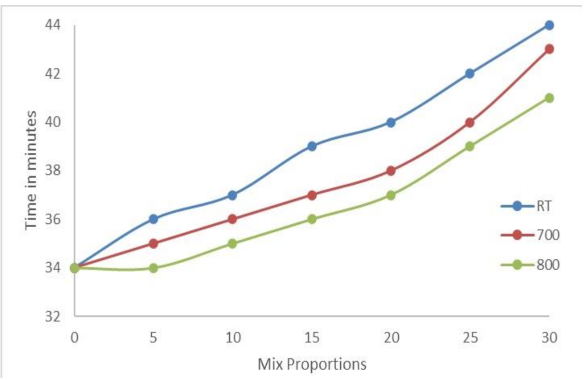

Figure 3. Normal Consistency setting time of all mixes

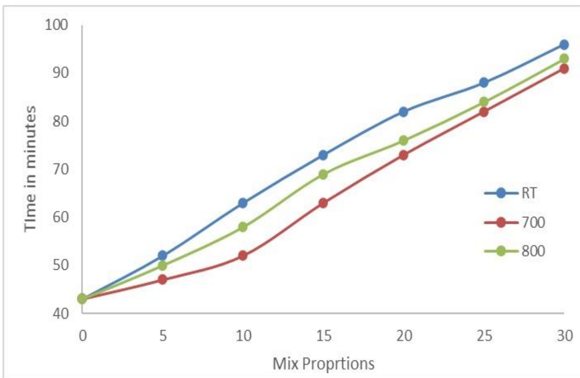

Figure 4. Initial setting time of all mixes

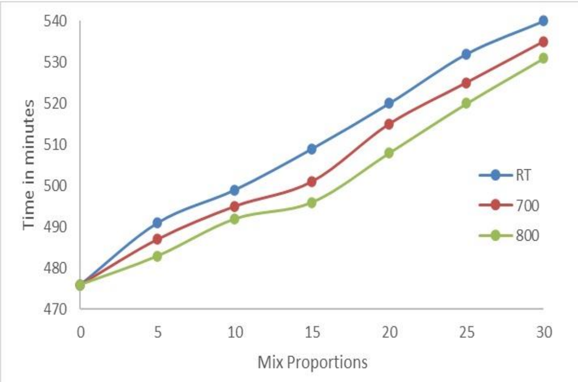

Figure 5. Final setting time of all mixes

3. 4. Strength Activity Index Strength activity index of all specimens was calculated at 28 days curing. The summary of results was displayed in Figure 11. More strength activity index was seen for the cement mortar replaced by 20 % bentonite calcined at 800^{0}C. This may be because of pozzolonic activity occurrence upon addition of calcined bentonite.

3. 4. Strength Activity Index Strength activity index of all specimens was calculated at 28 days curing. The summary of results was displayed in Figure 11. More strength activity index was seen for the cement mortar

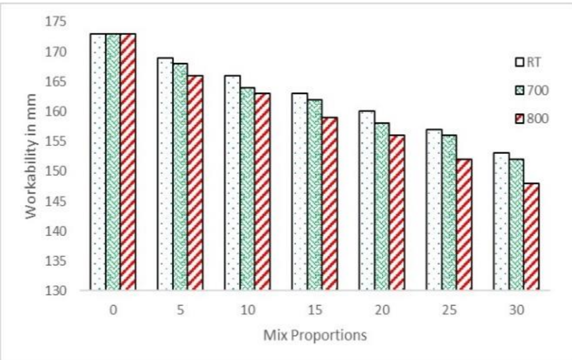

Figure 6. Workability of all mixes

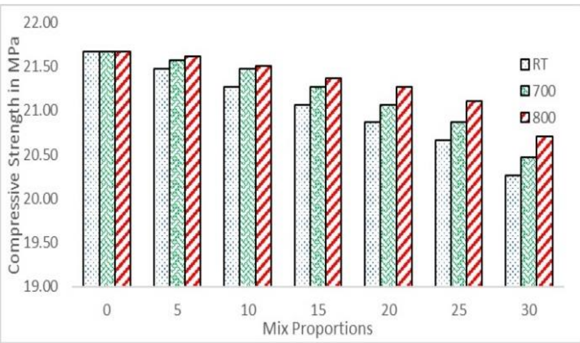

Figure 7. Compressive strength of calcined bentonite mixes after 3 days curing

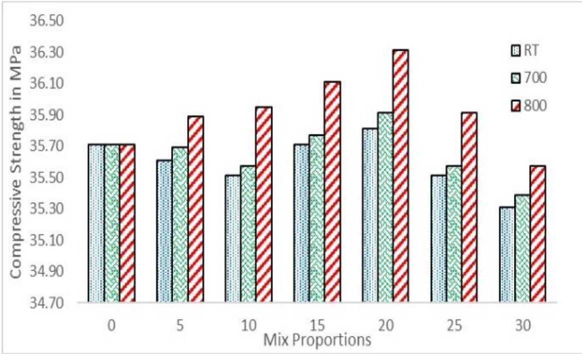

Figure 8. Compressive strength of calcined bentonite mixes after 7 days curing

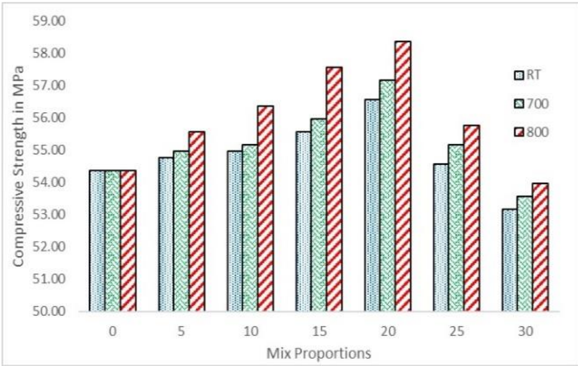

Figure 9. Compressive strength of calcined bentonite mixes after 28 days curing

replaced by 20 % bentonite calcined at 800^{0}C. This may be because of pozzolonic activity occurrence upon addition of calcined bentonite.

3. 5. Sorpitivity Figure 11 displays the sorpitivity for all mixes after 28 days curing. Drastic decrease in the sorpitivity was observed upon addition of bentonite as well as calcined bentonite. This may be as a result of high-water absorption capacity of bentonite and calcined bentonite.

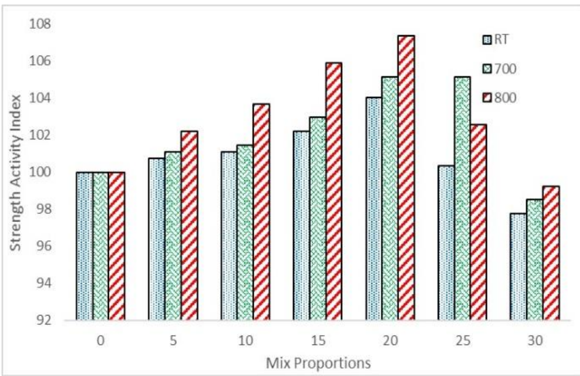

Figure 10. Strength Activity Index of calcined bentonite mixes after 28 days curing

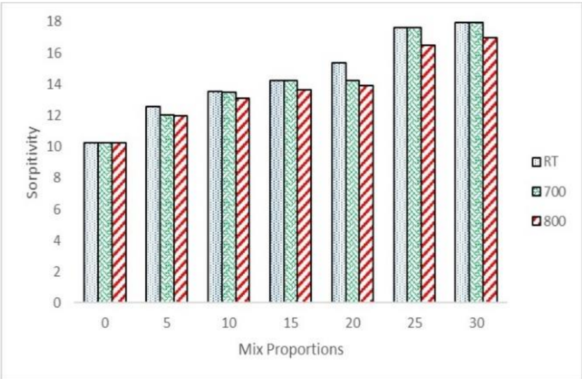

Figure 11. Sorpitivity of calcined bentonite mixes

4. RSM MODELLING

4. 1. Development of Model Various types of models are available like Central Composite, Box Behnken and optimal (custom) in a randomized design in RSM. Selecting a suitable model depends on the type of available data and levels for each factor. The modelling and optimization were done after performance of laboratory experiments using central composite design method of RSM. CCC models are the unique form of the central composite design. The star points are at some α distance from the center based on the properties required for the design and the number of factors in the design. To maintain rotatability, the value of α depends on the number of trial runs in the factorial portion of the CCC. However, the factorial portion can also be a fractional factorial design of resolution V. The value of α also varies on whether the design is orthogonally blocked.

4. 2. Mix Matrix Design In this investigation, Design Expert 11.0 version was used. The design of experiments was created by a composite design technique based on two variables (calcination temperature and bentonite replacement). Three levels of calcination temperature (Room Temperature (RT), 700^0C and 800^0C) and six levels % of bentonite replacement (0%, 5%, 10%, 15%, 20%, 25% and 30%) were used. 21 combinations of mixtures were developed in RSM. Table 3 represents the details of all combination of variables. The responses of calcined bentonite blended concrete (workability, compressive strength, strength activity index and sorpitivity) were determined for all mixtures, considered for RSM analysis and optimization.

4. 3. Analysis of Variance The summaries of ANOVA for the responses were analyzed, displayed in Tables 5-8. F-values are 9.60, 260.48, 7.10 and 155.56 for workability, compressive strength, strength activity index and sorpitivity respectively, pointing all models to be significant. The quadratic model was used for workability, compressive strength and strength activity index. For sorpitivity, linear model was used. In models of workability and sorpitivity, all terms are significant. For compressive strength and strength activity index, AB and A^2 and was the insignificant term and interaction in the quadratic model. The final models for workability, compressive strength, strength activity index and sorpitivity of all mixes consist of all the terms are given in Equations (1)-(4).

$$\text{Workability}=+172.50305+0.020037A-0.741675B-0.000129AB-0.000027A^2+0.003651B^2 \quad (1)$$

$$\text{Compressive strength}=+53.83245-0.006978A+0.331324B+0.000030AB+9.69751E\text{-}06A^2-0.011656B^2 \quad (2)$$

$$\text{Strength activity index}=+98.80342-0.006795A+0.612087B+0.000084AB+0.000010A^2-0.021419B^2 \quad (3)$$

$$\text{Sorpitivity}=+10.94914-0.000724A+0.238712B \quad (4)$$

3-dimensional (3D) response surface plot was used to illustrate the relationship between responses and independent variables. Figures 12-15 show the 3D response surface plots illustrating the relationship between responses (workability, compressive strength, strength activity index and sorpitivity) and independent variables (RT, 700^0C and 800^0C) for all mixes.

4. 4. Optimization Optimization was employed with the aim of getting an optimized variable of all bentonite mixes. Optimization was performed by using Design Expert Software. Table 8 shows optimization results of bentonite mixes. The optimized mix was achieved at 15.25 % of substitution of bentonite calcined at 800^0C. The optimum values of responses were 57.17 MPa compressive strength, 159.01 mm workability, 105.14 strength activity index and 14.03 sorpitivity. The optimized performance was proposed by considering different performance indexes.

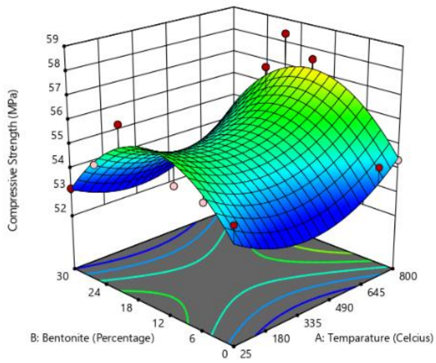

Figure 12. 3D surface model for compressive strength

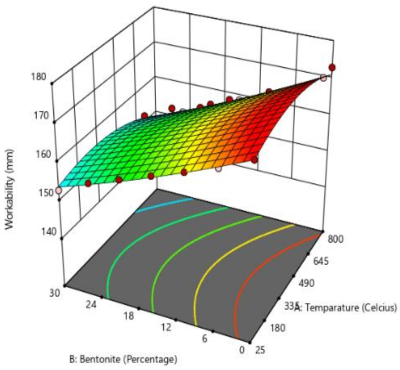

Figure 13. 3D surface model for workability

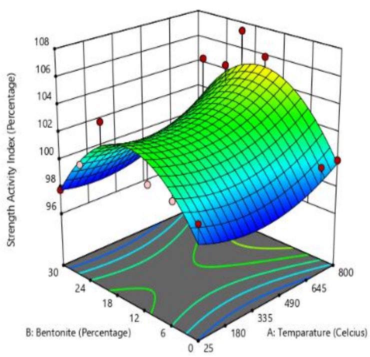

Figure 14. 3Dsurface model for Strength activity Index

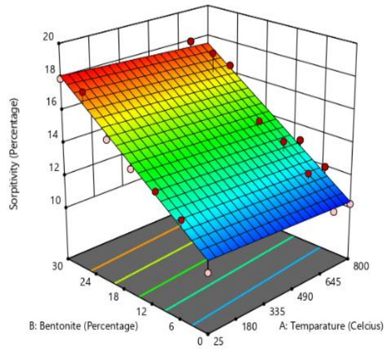

Figure 15. 3D surface model for Sorpitivity

TABLE 3. details of all combination of variables

	F:1	F:2	R:1	R:2	R:3	R:4
S. No	**Calcination Temparture**	**Bentonite Percentage**	**Compressive strength**	**Workability**	**Strength Activity Index**	**Sorpitivity**
1	25	0	54.37	173	100	10.23
2	25	5	54.77	169	100.73	12.54
3	25	10	54.97	166	101.1	13.523
4	25	15	55.57	163	102.2	14.208
5	25	20	56.58	160	104.06	15.345
6	25	25	54.57	157	100.36	17.619
7	25	30	53.17	153	97.79	17.92
8	700	0	54.37	173	100	10.23
9	700	5	54.97	168	101.1	12.01
10	700	10	55.17	164	101.47	13.45
11	700	15	55.98	162	102.96	14.208
12	700	20	57.18	158	105.16	14.208
13	700	25	55.17	156	105.15	17.619
14	700	30	53.57	152	98.52	17.965
15	800	0	54.37	173	100	10.23
16	800	5	55.57	166	102.2	11.98
17	800	10	56.38	163	103.69	13.12
18	800	15	57.58	159	105.9	13.64
19	800	20	58.38	156	107.37	13.894
20	800	25	55.77	152	102.57	16.482
21	800	30	53.97	148	99.26	16.98

TABLE 4. ANOVA for quadratic model, R1: Compressive strength

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	26.49	5	5.30	9.60	0.0003	significant
A-Temperature	4.60	1	4.60	8.33	0.0113	
B-Bentonite	0.0652	1	0.0652	0.1181	0.7359	
AB	0.2306	1	0.2306	0.4177	0.5279	
A^2	1.69	1	1.69	3.06	0.1006	
B^2	21.40	1	21.40	38.76	< 0.0001	
Residual	8.28	15	0.5520			
Cor Total	34.77	20				

TABLE 5. ANOVA for quadratic model, R2: Workability

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1070.90	5	214.18	260.48	< 0.0001	significant
A-Temperature	41.14	1	41.14	50.04	< 0.0001	
B-Bentonite	915.22	1	915.22	1113.07	< 0.0001	
AB	4.13	1	4.13	5.02	0.0406	
A^2	13.40	1	13.40	16.29	0.0011	
B^2	2.10	1	2.10	2.55	0.1309	
Residual	12.33	15	0.8222			
Cor Total	1083.24	20				

TABLE 6. ANOVA for quadratic model, R3: Strength Activity Index

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	89.92	5	17.98	7.10	0.0014	significant
A-Temperature	15.54	1	15.54	6.13	0.0256	
B-Bentonite	0.0341	1	0.0341	0.0135	0.9092	
AB	1.76	1	1.76	0.6937	0.4180	
A^2	1.80	1	1.80	0.7098	0.4127	
B^2	72.26	1	72.26	28.53	< 0.0001	
Residual	38.00	15	2.53			
Cor Total	127.92	20				

TABLE 7. ANOVA for linear model, R4: Compressive strength

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	120.97	2	60.48	155.56	< 0.0001	significant
A-Temperature	1.30	1	1.30	3.35	0.0838	
B-Bentonite	119.67	1	119.67	307.76	< 0.0001	
Residual	7.00	18	0.3888			
Cor Total	127.97	20				

TABLE 8. Optimized calcined bentonite mixes

Bentonite percentage	Calcination temperature	Compressive strength	Workability	Strength activity Index	Sorpitivity
15.26	800	57.17	159.01	105.14	14.03
21.34	538.78	55.00	159.72	102.32	15.65
19.37	792.08	56.90	156.26	104.80	15.00
17.91	310.96	54.96	163.26	102.21	15.00
29.11	800	54.93	149.55	101.39	17.31

5. CONCLUSIONS

The following conclusions were made based on experimental and statistical analysis,

- Initial and final setting times were increased upon addition of calcined bentonite.
- Workability was decreased by increasing the percentage of calcined bentonite addition. This attributed to the higher water absorption capacity of the bentonite
- The maximum compressive strength was observed for 20 % bentonite substitution and calcined at 800^{0}C temperature due to pore filling effect since the particle size of bentonite is lesser than cement.
- Strength activity was improved; best strength activity was observed for the mix made with 20 % bentonite substitution calcined at 800^{0}C because bentonite obeys pozzolanic properties.
- The sorpitivity of cement mortar decreased significantly upon increasing the addition of calcined bentonite due to the lesser particle size of bentonite comparatively with cement.
- The model was prepared, and optimization was done by using RSM. The optimized values of variables were determined as 15.25 % bentonite substitution and 800^{0}C calcination temperature.
- The optimum values of responses were 57.17 MPa compressive strength, 159.01 mm workability, 105.14 strength activity index and 14.03 sorpitivity.

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Persian Abstract

چکیده

کشف جایگزین برای مواد پوزولانی تولید شده از ضایعات صنعتی به دلیل در دسترس نبودن آن هنگام خاموشی دائمی صنایع مورد نیاز بود. در این مقاله بهینه سازی استفاده از رس بنتونیت کلسینه در ملات سیمان با استفاده از روش سطح پاسخ (RSM) پرداخته شده است. متغیرها به عنوان سه سطح دمای کلسینه (دمای اتاق ، ۷۰۰C˚ و C˚ ۸۰۰و هفت سطح بنتونیت کلسینه شده (۰٪ ، ۵٪ ، ۱۰٪ ، ۱۵٪ ، ۲۰٪ ، ۲۵٪ و ۳۰٪) گرفته شدند. مقاومت فشاری ، کارایی ، شاخص فعالیت قدرت و میزان جذب به عنوان پاسخ در نظر گرفته شد. خواص تازه و سخت شده همه برای همه مخلوط ها تعیین می شود. برای انجام مدل سازی و بهینه سازی با استفاده از RSM از نسخه Design Expert 11.0 استفاده شده است. با افزایش دمای کلسیم و مقدار بنتونیت در ملات سیمان ، میزان کارایی آن کاهش یافت. این امر به ظرفیت جذب بالای بنتونیت نسبت می دهد. حداکثر مقاومت فشاری پس از ۲۸ روز پخت با ۲۰٪ جایگزین شده بنتونیت کلسینه شده در ملات سیمان C˚ ۸۰۰ نشان داده شد. با افزایش درصد بنتونیت کلسینه شده در C˚ ۸۰۰ ، فعالیت قدرت بهبود یافت. میزان جذب ملات سیمان با ترکیب بنتونیت کلسینه شده در C˚ ۸۰۰ بهبود یافت. مدلهای تولید شده از RSM در کلیه فاکتورهای مورد بررسی اهمیت داشتند. عملکرد مطلوب پاسخها در ۱۵/۲۵ درصد تعویض بنتونیت که در ۸۰۰ درجه سانتیگراد کلسینه شده مشاهده گردید.