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* **Corresponding author**.

shalini.me19@gmail.com

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Experimental studies on the utilization of kaolinite amalgamated in cement mortar

Logesh Kumar M[1], Baskar Prabu[2], Annadurai Shalini[2]*

1 Associate Professor, Department of Civil Engineering, Sona College of Technology, 636005, India
2 Assistant Professor, Department of Civil Engineering, Sona College of Technology, 636005, India

Abstract

Objective: To investigate the physical and strength properties of mortar using locally available mineral admixture. **Methodology:** The compressive strength of the mortar at different temperatures was found. Setting time, consistency, and porosity were also studied by blending natural kaolinite and metakaolinite (MK) with cement in different proportions. An experimental work was carried out using control mix and adding kaolinite from 5% to 20% in ordinary Portland cement (OPC) at 5% equal intervals. **Findings:** The test results revealed that the use of natural admixtures such as Natural kaolinite (NK) and MK in OPC enhances the performance of the binder in mortar. The compressive strength enhancement of natural kaolinite cement–blended binder was comparatively lower than that of control mortar for all percentages whereas the mortar added to MK had increased strength. MK mortar with a 15% mix proportion was the optimum one to yield maximum compressive strength. **Novelty:** The present research work is focused on the partial replacement of metakaoline with temperature variant and the percentage of dosage used as a double variant parameter.

Keywords: Natural kaolinite; Metakaolinite; Calcination; Apparent porosity; Compressive strength

1 Introduction

There is a huge demand for ordinary Portland cement (OPC) in the construction industry and it cannot meet all the needs of the country for construction of pavements, bridges, and canal works. Cement is used as binder material in concrete for various construction purposes. It leads modern construction materials without any rival. On the one hand, the source materials used in the cement production are uneconomical and create environment pollution. On the other hand, there is rapid industrialization, which leads to various pollution issues posing a threat to the environment. Identifying a suitable alternative material to replace cement either partially or fully to produce environment-friendly concrete at a lower cost is urgently required. An enormous amount of clay has been left as unused and it has valuable minerals. The use of these minerals helps in avoiding environmental degradation with respect to natural resources. Mineral admixtures such as rice husk ash, ground-granulated blast-furnace slag (GGBS), silica fume, metakaolinite (MK) and fly ash (FA) can be utilized as substitute for the cement to produce concrete with same pozzolanic material. Kaolinite is a mineralogical term used for kaolin. It is very effective in strength and durability aspects against sulfates and alkalis reaction.

When MK was partially replaced, the relative strength of concrete with MK increased, which lasted up to 14 days before it started declining[1]**.** Using cement with 15% replacement of MK resulted in the enhanced compressive strength[2,3]. MK reduces early age autogenous shrinkage from initial setting time and changes in reduction was higher at increase in level of replacement[4,5]. The presence of MK helps mortar in maintaining its resistance at elevated temperatures[6]. Partial replacement of OPC with calcined kaolin in mortar improves the compressive strength and durability. Using 15%–20% calcined kaolin in mortar, it was noticed from the observation, at 28 days of curing mortar strength was increased. The mortar strength increased due to blending of different materials[7]. The concrete properties like shrinkage and creep resulted in reduced early age autogenous shrinkage which containing 0%–15% calcined clay, which was measured from the time of initial set[8]. The enhancement in the relative strength of concrete with variation in the curing time and amount of Metakaoline added was because of the filler effect and pozzolanic reaction[9,10]. The strength and other properties were enhanced at the optimum replacement level of 15%[1]. The assessment of hydration process was performed in MK-blended cement. It was noticed that, the rate of CH consumption and pozzolanic reaction was comparatively higher than at early ages when compared to FA-blended cement[11].

The compressive strength increases till 200 °C and starts falling beyond it. Thermal shock resistance was reduced when OPC was replaced by 15% MK and 5% SF. Some researchers compared the pozzolanic reaction of MK on the hydration of cement with other pozzolanic material and found that MK mortar had a slight increase in heat resistance compared to 100% PC mortar due to high pozzolanic content[7]. A desired qualitied mortar can be prepared with the characteristics of pozzolans[8]. An extensive research work has been carried out on the manufacturing of mortar with different types of suitable clay as a pozzolanic material. From the past literatures, it is evident that the role of pozzolanic material's chemical and physical properties plays an important criterion in strength properties identification. But only a minimal amount of research work has been done on the production of mortar with natural clay as pozzolanic material. The previous research work was performed only on the utilization of metakaoline for partial replacement for cement content under single temperature variant. The novelty of the present research work is focused on the partial replacement of metakaoline with temperature variant and the percentage of dosage used as a double variant parameter.

Therefore, in this research, an attempt has been made to manufacture the mortar with locally available kaolinite and to study the physical and strength properties when the mortar is exposed to different temperatures. The OPC was replaced with different proportions of blended kaolinite to prepare the mortar in order to evaluate its properties.

2 Materials used

In this research, OPC 43 grade conforming to IS 8112: 2013[12] and tested as per BIS standards[13–17] was purchased and used as a binder. River sand was used as a fine aggregate conforming to IS 383-1970 (reaffirmed 2011)[18]. Potable water was added to attain the desired workability as per specifications confirming to IS 456:2000[19]. The natural admixtures such as kaolinite and MK were purchased from the Ceramic Industry (Virudhachalam, Tamil Nadu, India). The physical and compressive strength of cement were tested as per IS 8112: 2013[12]. The properties of river sand were determined as per the guidelines mentioned in BIS 2386-1963 (reaffirmed 2002)[20]. For the sieve analysis test, fine aggregate was dropped on Zone III grading in accordance to BIS 383-1970 (reaffirmed 2011)[18]. Natural and calcined kaolinite (400, 600, 800 °C) were used for this study.

The physical properties such as specific gravity and specific surface area were calculated as per guidelines in IS 1727-1967[21] for natural and calcined kaolinite. Also for kaolinite blended cement, consistency and setting times of the final and initial stages were measured as per guidelines[13–17]. Scanning electron microscopy was used to study the physical properties. Apparent porosity, and compressive strength at different temperatures were studied and detailed explanations are given. The chemical composition of natural and calcined kaolinite was tested as per IS 4032-1985[22].

3 Mix proportions

Different mixes for 70.6 mm mortar were prepared in a ratio of 1:3 (binder/sand) by weight. Different mix proportions were made by replacing cement with kaolinite and Metakaoline at 5%, 10%, 15% and 20%. Required amount of water was mixed in all the ratios and adjusted to obtain the requirements given in BIS 1727-1967[21]. Table 1 shows the details of mortar mix with different percentages of kaolinite and Metakaoline.

Table 1. Different percentage of mix details for mortar

Materials	Kaolinite				
	0%	5%	10%	15%	20%
Cement (kg)	200	190	180	170	160
Kaolinite (kg)	0	10	20	30	40
Sand (kg)	600	600	600	600	600
W/c ratio for natural blending	0.42	0.45	0.46	0.49	0.5
W/c ratio for 400°C	0.42	0.39	0.41	0.43	0.45
W/c ratio for 600°C	0.42	0.38	0.41	0.42	0.43
W/c ratio for 800°C	0.42	0.41	0.43	0.44	0.49
Flow, at 25 drops of flow table (%)	105	105	105	105	105

4 Preparation, curing and Testing of specimen

The ingredients were mixed thoroughly in a Hobart mixer for about 3–5 min to get a homogeneous mix. The mortar cubes were kept at room temperature in an atmosphere of 90% relative humidity for 24 h. The prepared fresh mortar mix was placed in a mold and consolidated by a vibrator machine. Next day, all hardened mortar was demolded and named for further reference. The curing tank was filled with clean fresh water and the mortar was submerged in the tank for the desired number of days. Mortar cubical size of 70.6 mm were prepared to study the compressive strength at ages of 7, 28, 56, and 90 days in accordance to BIS: 516-1959 (reaffirmed 2004)[(23)].

The physical property and porosity of mortar were studied at 28, 56, and 90 days. Porosity is considered as the ratio of weight difference before and after soaking in water and divided by mortar volume. The compressive strength of the mortar was calculated for the curing periods of 28, 56, and 90 days. Compressive strength was taken as the average of three cubes for each period.

5 Test Results and discussion

5.1. Specific gravity of natural and calcined kaolinite

Table 2 shows the particle size, specific gravity, and specific surface area of natural and calcined kaolinite at different temperatures. Compared to the specific gravity of cement, natural kaolinite possesses very low specific gravity of 2.70. The increase in temperature during calcination owing to continuous reduction in specific gravity of calcined kaolinite and kaolinite calcined at 800 °C was as low as 2.54.

Table 2. Physical properties of natural and calcined kaolinite at different temperatures

S. No	Characteristics	NK	Metakaoline (MK)		
			400°C	600°C	800°C
1	Specific gravity (no unit)	2.70	2.67	2.62	2.54
2	Specific surface area (cm^2/g)	7280	9556	10010	10180
3	Particle Size (average) (μm)	3.2	-	-	2.4

5.2 Specific surface area and Particle Size

The specific surface area (SSA) was measured using the Brunauer–Emmett–Teller method as per IS 11578-1986[(24)] and the value was 7280 cm^2/g for natural kaolinite. At 800 °C, specific surface area significantly increased to 10180 cm^2/g in increased calcination temperature. The SSA of kaolinite calcined at 800°C showed drastic improvement than that of cement. It was measured as 3080 cm^2/g, which is double time lower than natural kaolinite. Also three time lower than the calcined kaolinite. Higher specific surface area might adversely affect the consistency of the binder and flow rate and workability of mortar. However, higher specific surface area accelerates hydration and pozzolanic reactions of calcined kaolinites with CH and improves filler effect in concrete.

Particle size of the natural as well as calcined kaolinite was measured using a scanning electron microscope (SEM) and illustrated in Figure 1. The SEM micrograph of natural kaolinite shows that the particles are not of uniform size. Particle size varies between 2.8 and 3.4 μm with a median size of 3.2 μm. Average particle size of kaolinite was reduced to 2.4 μm during calcinations at 800 °C[7]. As the particle size of calcined kaolinite is less than that of cement, the properties of mortar a well as concrete might improve significantly due to filler effect and increased pozzolanic activity. Further, higher specific surface area will be resulted in the particle size distribution and the blending property also get improved. Owing to this, particles with higher specific surface area resulted in the higher compressive strength.

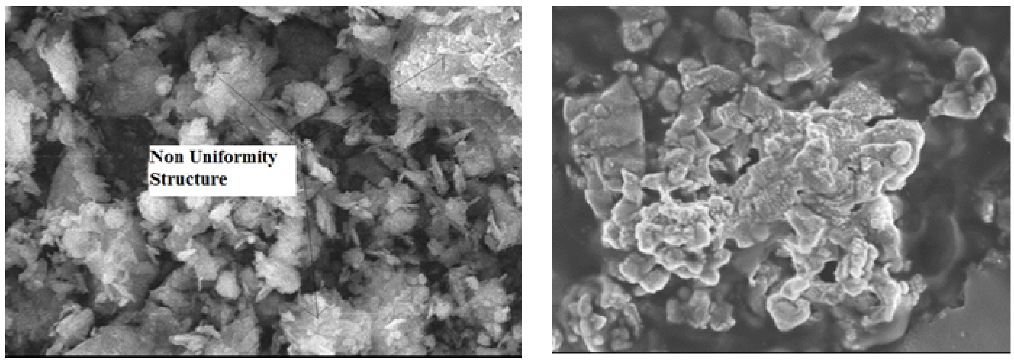

Fig 1. SEM image of natural kaolinite and calcined kaolinite (800°C)

5.3. Consistency of binder at different percentage levels

Consistency levels of OPC and cement replaced at 5% interval from 5% to 20% of natural kaolinite were measured. Test results confirm improvement in in consistency of the blended cement compared to the controlled binder. The results showed that the level of is consistency improved with addition to the percentage of NK in cement. In another point of OPC added to the MK illustrated that consistency progressively increased with the increase in MK content. The enhanced consistency was happening because of higher SSA and finer particle size MK. The consistency level of kaolinite calcination was marginally reduced because of modification of internal structure during calcination of NK[25].

5.4. Setting time

It is clear from Table 3, compared to control binder the setting times of cement added with NK are higher. There is appreciable increase in setting time by increasing the amount of NK. The amount of kaolinite and degree of calcination influences the setting times of cement added to the MK. It was noticed that increases in temperature of calcination resulted in reduced setting times gradually. Also, increase in kaolinite content gradually decreased both initial and final setting times. Kaolinite-blended cement mortar calcined at 800 °C had an large reduction in setting times when compared to control specimen. Reduction in setting time could be due to the presence of amorphous SiO_2 and Al_2O_3. The formation of calcium sulfo aluminate and C–S–H gel happens due to reaction of constituent materials with cement hydration, during early hydration which creates the cement paste framework.

Table 3. Setting times of natural and calcined kaolinite added with cement in different percentage

Blended mix ratio	Setting time of Control Mix (mins)		Setting time of NK (mins)		Setting time of MK (400°C), mins		Setting time of MK(600°C), mins		Setting time, mins (800°C)	
	Initial	Final	Initial	Final	Initial	Final	Initial	Final	Initial	Final
Control mix	140	215	-	-	-	-	-	-	-	-
5% ka+ 95% C	-	-	210	270	203	285	180	252	140	211
10% ka+ 90% C	-	-	225	285	198	273	167	229	125	182
15% ka+ 85% C	-	-	207	260	195	262	140	193	120	171
20% ka+ 80% C	-	-	210	270	176	236	130	192	128	176

5.5. Apparent porosity

Table 4 presents the results of apparent porosity at different temperatures of mortar produced withNK and cement varies from the control mix (0%) and in relation to the curing period. From the test results, it is clear that, porosity of control mortar reduced in when the amount of NK increased up to 20% in binder. It is also noted that there is a minor reduction in apparent porosity and extended up to 90 days of curing. Moreover, the inclusion of calcined kaolinite revealed a disparity in the degree of reduction in the porosity value with respect to the curing period[26]. In blended mortar, there is a reduction is apparent porosity on increase in the content of calcined kaolinite. At 28 days, it was noticed that there will be a considerable reduction in apparent porosity and subsequently there was no more changes in the reduction. It is clear from the test results; the kaolinite calcination temperature influences the properties of apparent porosity. The maximum changes in reduction of apparent porosity was observed at calcined at 600 °C in mortar. Moreover, the enhancement of calcination temperature had no appreciable impact on the mortar porosity[27].

Table 4. Apparent porosity of natural kaolinite mortar and incorporated with metakaolinite in different temperature

Mix Code	At Room Temperature			With Metakaoline At 400°C			With Metakaoline at 600°C			With Metakaoline at 800°C		
	28d	56d	90d	28d	56d	90d	28d	56d	90d	28d	56d	90d
0% NK	8.11	6.30	4.16	8.11	6.30	4.16	8.11	6.30	4.16	8.11	6.30	4.16
5% NK	11.27	9.47	6.40	3.64	3.38	3.42	3.88	3.29	3.31	3.59	3.11	3.05
10% NK	9.27	6.25	4.27	3.53	3.18	3.22	3.19	3.07	3.09	4.05	2.95	2.95
15% NK	8.94	6.75	4.37	3.56	3.21	3.05	2.55	2.83	2.55	2.73	2.85	2.55
20% NK	8.22	6.43	3.11	3.51	2.95	2.85	2.49	2.55	2.48	2.45	2.44	2.28

5.6. Compressive strength for mortar

5.6.1 Compressive strength development of natural kaolinite

Natural kaolinite was replaced in cement up to 20% at a regular interval of 5%. Mortar strength was measured by a compressive testing machine in the Concrete Laboratory. Compressive value of 0% replacement showed a promising result from 28 days. Further, when natural kaolinite was incorporated at different percentages, compressive strength value decreased significantly. This might be due to less specific surface area compared with the calcined specimen which had not been thermally exposed to different temperatures. The strength development of natural kaolinite and metakaolinite incinerated at 400° C, 600° C & 800° C are shown in Table 5.

Table 5. Compressive strength of natural kaolinite mortar and incorporated with metakaolinite in different temperature (N/mm^2)

Mix code	At Room Temperature			With Metakaoline At 400°C			With Metakaoline at 600°C			With Metakaoline at 800°C		
	28d	56d	90d	28d	56d	90d	28d	56d	90d	28d	56d	90d
0% NK	50.21	55.21	61.87	50.21	55.21	61.87	50.21	55.21	61.87	50.21	55.21	61.87
5% NK	34.07	36.0	37.76	50.46	59.11	64.4	50.54	59.33	64.40	50.46	60.78	68.10
10% NK	28.56	33.88	35.47	49.13	57.74	63.09	49.13	57.74	63.09	51.21	63.30	70.03
15% NK	43.71	47.35	51.4	52.43	62.56	69.32	52.43	62.56	69.32	54.55	70.12	75.54
20% NK	41.29	42.29	31.46	43.24	52.26	60.03	43.52	52.38	60.04	47.04	61.29	69.31

5.6.2 Compressive strength development of metakaolinite incinerated at 400 °C, 600 °C & 800 ° C

Compressive strength test results of cement replaced with up to 20% MK treated at 400 °C were analyzed. Compared to the control mix, up to 10% replacement showed strength increment at later ages and almost the same strength value at 28 days. Further, 15% and 20% replacement showed promising strength at 28 days with gradual increases at later stages. Strength increment was mainly because of fineness of the material, specific surface area, and thermal treatment[28,29]. Compared to different replacement levels of natural kaolinite, Metakaoline showed a promising strength development at the age of 28 days, which was due to the calcination and pozzolanic activity between the binders.

When the compressive strength of MK treated at 600 °C was compared to that of material treated at 400°C, it was found to be almost the same. Also, compared to different percentage levels, 15% replacement showed promising strength value. The consistency level was marginally reduced due to the change in the structure of minerals, which affects the dihydroxylation temperature (600° C) and influences the pozzolanic activity and water demand of the mortar.

The compressive strength of MK shows promising strength values at all levels of replacement. Further, 28-day results at all replacement levels are twice higher than the conventional mix. Moreover, when the number of days is higher, strength value is saturated and yields better compressive strength than conventional mortar at the age of 90 days [30]. From the test results it has been observed that when compared with NK, metakaoline exhibit promising results in terms of strength properties. It has been noted that natural kaolinite replaced with cement up to 20% does not shows any significant strength when compared with control mix. Further in case of metakaoline, replacement level up to 15% shows good strength but beyond that there is no appreciable increase in strength [31] due to the effect of clinker dilution in partial replacement of cement and not allowed to further participation of cementitious materials in the reaction [32,33].

Summary

The compressive of mortar made by blending cement and natural kaolinite was less than that of the control specimen at all replacement levels. However, the mortar with 20% MK showed lesser strength compared to mortar with 15% MK. The strength of mortar with MK increased with increase in temperature of kaolinite calcination [34]. The reduction in the strength of natural kaolinite mortar was due to the formation of crystallites and calcination of kaolinite up to the replacement level of 15%. Degradation of strength beyond 15% replacement level occurred due to heat hydration and filler effect, which shows considerable strength enhancement in short and long term [7]**.** The mortar incorporated with MK and calcined at 800 °C showed considerably higher strength than that calcined at 400 and 600 °C. It was happening because of the quartz and crystalline kaolinite changes into amorphous form. The reaction of amorphous silica with Ca $(OH)_2$ at the time of hydration process, which resulted in formation of C–S–H (Calcium Silicate Hydrate) [35]. Also, the reaction of Al_2O_3 presence of in kaolinite with lime and enhances the strength properties due to formation of tetra calcium aluminate hydrate [36,37].

6 Conclusions

Based on the experimental results, owing to higher specific surface area of kaolinite, it showed that consistency gradually increases with increase in the percentage of kaolinite replacement. It proved that the level of consistency of the binder gradually decreases in kaolinite, particularly at higher temperatures. Natural kaolinite was added in excess to the binder, it marginally shortened the initial and final setting times to some extent. Incorporation of metakaolinite in OPC considerably changed setting times. It was due to the reaction of SiO_2 and AlO_2 present in kaolinite with the hydrate of cement that formed C–S–H gel and calcium sulfoaluminite, which formed a framework for cement paste during early hydration. Both the natural and calcined kaolinite showed that while ageing was prolonged, the t value decreased gradually compared to initial aging. Also, the degree of calcination did not affect the porosity values. The mortar strength varied with the content and the calcination temperature of kaolinite. Strength gradually increased on increasing MK content from 5% to 15% and started reducing thereafter. Also, increasing the calcination temperature of MK from 400 to 800 °C significantly improved the mortar strength. Lastly, improvement in the compressive strength of mortar incorporated with MK at different ages could be due to the arrest of voids by the filler and increase in the hydration of Portland cement and its reaction with pozzolanic materials. The present study is limited to calcination temperature from 400°C to 800°C, the research may further proceed with addition of other pozzolanic materials with NK.

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