

# Effect Assessment of Calcination on Pozzolanic Activity of Natural Clay of District Dadu, Sindh

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**Abstract**— Nowadays calcined clays, particularly kaolin clays, are reevaluated as SCMs in order to reduce the CO<sub>2</sub> releases emitted into atmosphere from cement fabrication and energy consumption needed for the cement production. Calcined clays are potential source of SCMs to produce green cements. This study aims to introduce new pozzolanic material and to assess the effect of extent of calcination with different heating durations on the Pozzolanic reactivity of the natural clay of Nai Gaj Dam Project site, district Dadu, Sindh Pakistan. The clay was calcined in a furnace chamber at 600, 700 and 800°C for 1, 2 and 3 hours and at 900 and 1000°C for 1 and 2 hours. The strength activity index (SAI) was calculated with 20% substitution of cement with different samples of thermally activated clay at 7 days as per ASTM C-618. The SAI results outcome that the optimal activation temperature is 700°C with one hour duration. The SAI at 700°C with one-hour duration at 7 days is 127.05 % which satisfy the requirement of new pozzolanic material to be used in mortar and concrete (i.e.75%). The X-Ray diffraction (XRD) and Energy-dispersive spectrometry (EDS) tests were performed to confirm the existence of Silica contents in produced metakaolin. The XRD analysis verified pozzolanic reactivity of produced metakaolin and authenticated SAI results. In addition, it is also found that the compressive strength of mortars increased significantly.

**Index Terms**— Calcination, calcined clays, Compressive strength, Metakaolin, Pozzolanic material, Strength activity index, Kaolin clay.

## 1 INTRODUCTION

The cement is being extensively used as a construction material worldwide [1]. The consumption of cement has been increasing rapidly day by day[2]. In fact, approximately 0.8 tonne of CO<sub>2</sub> is emitted in the atmosphere due to the fabrication of one tonne of cement.[3], which is approximately 5–8% of total worldwide CO<sub>2</sub> releases [4]. Besides CO<sub>2</sub>, SO<sub>3</sub> and NO<sub>x</sub> is also produced due to fabrication of cement. Nowadays calcined clays, particularly kaolin clays, are reevaluated as SCMs in order to decrease the CO<sub>2</sub> releases and energy consumption instigated from cement fabrication [1][4].

Calcined clays are potential source of SCMs to produce green cements [5]. Kaolin clays, calcined at ambient temperatures, gives equal or higher value of pozzolanic activity than the eminent artificial pozzolans like fly ash and silica fume [6]. Thermally activated clays, calcined at high temperatures or with long heating durations, bears lower pozzolanic activity due to possible recrystallization [7].

The Kaolinitic clays are abundantly available in natural, and utilization of Kaolinitic calcined clays as substitute pozzolanic material has presently enhanced significantly in places where there is high transportation cost of industrial by-products or difficult accessibility of other industrial wastes [8][9].

If the Kaolin clays are heated, it can achieve encouraging pozzolanic reactivity and may be used as cement substitute material to reduce the emissions of CO<sub>2</sub>. The pozzolanic properties of calcined clays depend upon amount & type of clayey minerals, thermal activation, the amount and nature of impurities and specific surface obtained after calcinations.

## 2 EXPERIMENTAL WORK

### 2.1 Materials

Ordinary Portland cement (OPC), conforming to ASTM C150 type I, was used. The natural clay, available in huge quantity

in Taulka Johi of Dadu district, Sindh, Pakistan, was used in this research. The natural clay locally available at Nai Gaj Dam site, district Dadu, Sindh Pakistan was used as raw material to develop the pozzolanic material.

### 2.2 Calcination of natural clay

The heating temperatures for conversion of Clay into reactive metakaolin were kept 600 °C, 700 °C and 800 °C with heating durations of 1, 2, 3 hours and 900 °C and 1000 °C with heating duration of 1 and 2 hours. The different calcined clay samples are shown in fig 1. The electronic furnace was used for the calcinations process of raw clay. The maximum capacity of used furnace was 15 liters and highest temperature range was 1200°C. The grinding machine and loss angles machine was used for grinding of calcined clay. The samples of ground calcined clay as shown in fig 2 were passed through sieve No. 325 before using in mortar.



Fig 1: Calcined clay at different temperatures



Fig 2: Ground Calcined clay

### 2.3 Preparation of Mortar Specimens

The mortar cubes of 2"x2"x2" were casted by substituting the cement with 20 % calcined clay by weight. The mortar specimens were prepared using a sand-to-cementitious material ratio of 2.75 and W/b of 0.49, in accordance with ASTM C 311 [10]. The detail of mix proportion is given as under in Table 1.

TABLE 1.  
MIX PROPORTION OF MORTAR SPECIMENS

Mix ID	CT (°C)	HD (Hours)	Cement (g)	Calcined clay (g)	Sand(g)
A	----	-----	800	---	2200
B	600	1	640	160	2200
C	600	2	640	160	2200
D	600	3	640	160	2200
E	700	1	640	160	2200
F	700	2	640	160	2200
G	700	3	640	160	2200
H	800	1	640	160	2200
I	800	2	640	160	2200
J	800	3	640	160	2200
K	900	1	640	160	2200
L	900	2	640	160	2200
M	1000	1	640	160	2200
N	1000	2	640	160	2200

T = Calcination Temperature: HD = Heating Duration:

Mixing and compaction of mixes were done as per ASTM C305 [11], and molding were done in accordance with ASTM C109 [12]. The curing was performed for 7 days on specimen cubes.

## 3 RESULTS AND DISCUSSION

### 3.1 Mineralogical Composition of Natural clay and produced pozzolanic material

The X-ray diffraction analysis for mineralogical composition of natural clay and obtained pozzolanic material are given in

figure 3.

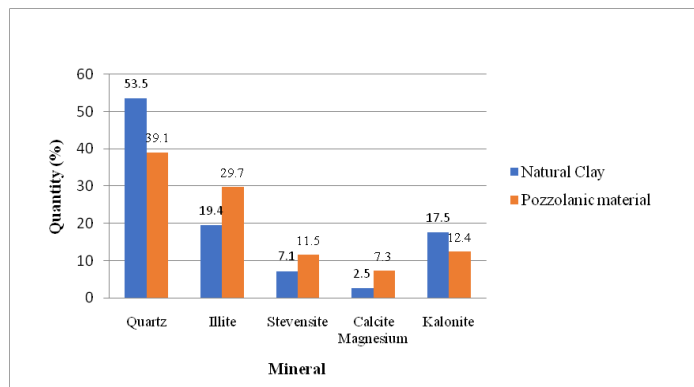


Figure 3: Mineralogical compositions of naturalclay and produced Pozzolanic material

It is obvious from the results presented in figure 3, the quantity of raw impurities (Quartz) has been reduced after the calcination of natural Clay from 53.5 to 39.1%. Tironi et al. [13] revealed that better results of compressive strength can be obtained by replacing the cement in mortars upto 30% calcined clays containing high value of impurities.

### 3.2 Chemical and Physical Properties of Natural Clay and Developed pozzolanic material

The chemical and physical properties of Natural Clay and developed pozzolanic material are presented in Table 2.

TABLE 2:  
CHEMICAL COMPOSITION

Constituents	Cement (%)	Natural Clay (%)	Pozzolanic material (%)
SiO <sub>2</sub>	20.75	49.89	56.01
Al <sub>2</sub> O <sub>3</sub>	5.14	21.51	20.37
CaO	60.79	1.51	2.91
MgO	3.1	1.53	1.39
Fe <sub>2</sub> O <sub>3</sub>	3.15	18.35	16.29
K <sub>2</sub> O	-----	4.89	1.05
Na <sub>2</sub> O <sub>3</sub>	-----	1.23	1.09
TiO <sub>2</sub>	-----	1.09	0.89
LOI (%)	1.70	6.3	0.5
Specific Gravity	3.14	2.65	2.62
<b>Pozzolanic material , SiO<sub>2</sub>+ Al<sub>2</sub>O<sub>3</sub>+ Fe<sub>2</sub>O<sub>3</sub> =92.67 %</b>			

As per ASTM C 618, SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub> ≥ 70 % for pozzolanic material, however the SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub> ≥ 85% for metakaolin [14]. The EDS results shows that the metakaolin produced from natural clay contains (SiO<sub>2</sub> + Al<sub>2</sub>O<sub>3</sub> + Fe<sub>2</sub>O<sub>3</sub>) equal to 92.67%, as presented in Table 2, and chemical composition of thermally activated Clay (pozzolanic material) satisfies the requirement of natural pozzolanic material and metakaolins and justifies its use in concrete/mortar in accordance with

ASTM C-618.

### 3.3 Strength Activity Index (SAI)

Algenra Tironi et al. [13] observed that SAI is the more precise method to evaluate the pozzolanic properties and to differentiate the real contribution of pozzolanic effect.

#### 3.3.1 SAI at 7 days

The results of SAI of control mortar and mortar prepared with 20% cement substitution with calcined clay tested after seven days are given below Figure 3.

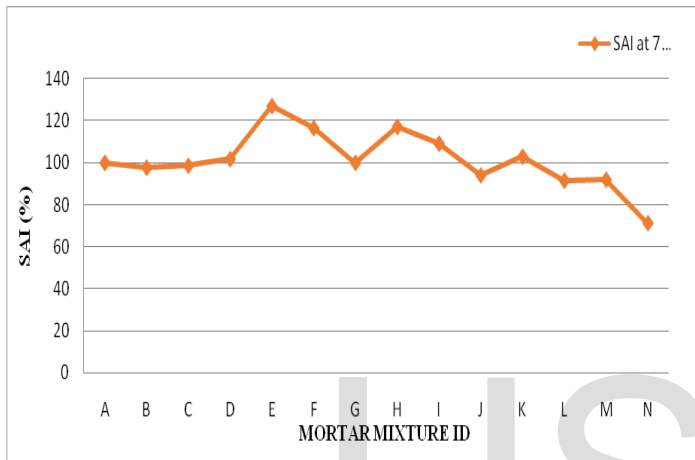


Figure 4- SAI at 7 days of mortar with inclusion of calcined clay activated at different temperatures and durations

The SAI results of 7 days in Figure 4, clearly indicates that SAI (%) of all mortar mixtures having thermally activated samples of calcined clay are more than that of controlled mixture except 600°C at 1hr and 2hr, 800°C at 3hr, 900°C at 1 hr and 1000°C at 1hr and 2hr. Thus, maximum SAI (%) is observed in E mix, and in E mix cement is replaced with calcined clay treated at 700°C for 1-hour duration, which is 127.05%, which satisfy the specification of new pozzolanic material to be used in mortar/concrete (i.e.75%) as per ASTM C 618. The results indicate that the reactivity of thermally activated Calcined clay is enhanced with increase in temperature. However, recrystallization starts as temperature reaches to 800°C, and thus the pozzolanic activity decreases.

Furthermore, by comparing the results in Fig 4, it can be observed that an increase in heating duration from 1 hour in all temperatures of treated calcined clay, SAI decreases except the temperature of 600°C. F Moodi et al [15] observed that after the completion of dehydroxilation process, further increase in heating duration at high temperatures results in recrystallization, which decreases the specific surface of metakaolin and resultantly pozzolanic reactivity and compressive strength is started to decrease.

## 4. CONCLUSIONS

Following conclusions can be made on the basis of obtained results of SAI, EDS and XRD;

- ❖ The natural clay material can be converted into pozzolanic

material, when calcined at a temperature of 700°C for 1 hour and The SAI at 700°C with one-hour duration at 7 days is 127.05 %

- ❖ The results of SAI, chemical composition and loss on ignition of the produced pozzolanic material satisfy the requirement of pozzolanic material as per ASTM C-618; thus, it can be used in concrete and mortar.
- ❖ Based on the results of SAI and chemical composition as per ASTM C-618, the natural Clay calcined at 700°C with heating duration of 1h is recommended as Pozzolanic material to be used in concrete/mortar.

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