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Development of Pozzolanic material from clay

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Abstract. The following paper concentrates on the objective of studying the influences of extent of duration and temperature on the Pozzolanic properties as well as reactivity of locally existing natural clay of Nai Gaj, district Dadu, Sindh Pakistan. The activation of the clay only occurs through heating when temperature in a furnace chamber reaches 600, 700 and 800oC for 1, 2 and 3 hours and at 900 and 1000°C for 1 and 2 hours. Furthermore, the strength activity index (SAI) of advanced pozzolanic material happens to be identified through 20% cement replacement for different samples of calcined clay as per ASTM C-618. The compressive strength test of samples had been operated for 7 and 28-days curing afterwards. The maximum compressive strength had been seen in mix E in which cement was replaced with clay calcined at 700°C for 1 hour that is 27.05 MPa that is 24.31% more than that of control mix. The results gathered from the SAI verdicts the optimal activation temperature is 700°C within a one-hour time period. The SAI at a temperature of 700°C with a one-hour duration at 28 days is 124.31% which happens to satisfy the requirements of the new Pozzolanic material, in order to be applied in mortar/concrete (i.e., 75%). The Energy- dispersive spectrometry (EDS) along with the X-ray diffraction (XRD) have been carried out in means of verifying whether there is silica content or amorphous silica present in metakaolin that has been developed. The findings gathered from the SAI were validated, as the analysis of XRD verified that there is in fact Pozzolanic activity of developed metakaolin. Additionally, based on observation, the activated metakaolin holds a significant influence on the increase in mortar's compressive strength.

Keywords: natural clay; Pozzolanic; Metakaolin; partial replacement; mortar; concrete

1. Introduction

Presently, with the advancement in various fields of Science, energy crises as well as global warming are amongst some of the largest issues that need to be combated globally. For starters, manufacturing one ton of cement emits about 0.8 tons of CO_2 into the air. This percentage considered around 5-8% of CO_2 emissions globally. Although, it is not just CO_2 emission but also SO_3 and NOx that are being freed to the surrounding atmosphere due to the production of cement which harms the environment and causes acid rains. Supplementary Cementing Materials (SCMs) are being extensively utilized all over the globe within the precast concrete category because of their economic as well as environmental benefits. Ground granulated blast-furnace slag, phosphogypsum fly ash lime stone powder, groundnut shell ash, waste paper pulp, Sidoarjo mud, hypo sludge, coconut shell ash, saw dust ash, grits, marble dust powder, metakaolin, baggas, silica fume and paper mill sludge ash etc. get utilized as SCMs (Owaid *et al.* 2012). Utilizing these SCMs with concrete may partially decrease cement consumption and construction cost, but increase mechanical properties and improve the workability and durability. Supplementary Cementing Material may possess latent hydraulic reactivity, Pozzolanic properties or even a mixture of both of these materials (Owaid *et al.* 2012). There are several factors that affect the reactivity of treated clay, namely quantity and the character of impurities, the form and quantity of clay minerals, thermal action adapted for Pozzolanic activity as well as the specific surface area (SSA) that is reached prior to calcinations (Tironi *et al.* 2013). Three different types of kaolin had been analyzed, specifically their chemical and mineralogical compositions, disregarding their physical properties, like its grain size distribution along with SSA. The SSA along with grain size seemingly comprise a power against the Pozzolanic activity from the metakaolins, hence why the metakaolin had been categorized to authenticate kaolinite's transformation to metakaolinite as well as confirming the modification on grain size and SSA. Evidently, the grain size and Pozzolanic reactivity of the metakaolins are in fact associated with one another (Fabbri *et al.* 2013). In order to test natural Pozzolanic material, Kaolinitic clay that was gathered from Tabarka in Tunisia

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Table 1 Mortar's mix proportion

Mix ID	CT (°C)	HD (Hours)	Cement (g)	Calcined clay (g)	W/b	Sand (g)
A	----	------	800	---	0.49	2200
B	600	1	640	160	0.49	2200
C	600	2	640	160	0.49	2200
D	600	3	640	160	0.49	2200
E	700	1	640	160	0.49	2200
F	700	2	640	160	0.49	2200
G	700	3	640	160	0.49	2200
H	800	1	640	160	0.49	2200
I	800	2	640	160	0.49	2200
J	800	3	640	160	0.49	2200
K	900	1	640	160	0.49	2200
L	900	2	640	160	0.49	2200
M	1000	1	640	160	0.49	2200
N	1000	2	640	160	0.49	2200

T=Calcination Temperature: HD=Heating Duration.

Table 2 Mineralogical compositions of raw material clay and developed Pozzolanic material

Mineral	Natural material; clay (%)	Developed Pozzolanic material (%)
Quartz	53.5	39.1
Illite	19.4	29.7
Stevensite	7.1	11.5
Calcite Magnesium	2.5	7.3
Kalonite	17.5	12.4

was gathered. It had been established how an increase in the specific surface area on the treated clay is in fact accredited on increases in the intensity of cement replacement (Samet *et al.* 2007). K1 and K3 are examples of Iranian mineral raw material along with K2 which was one by product from the domestic kaolin beneficiation process, and happened to get activated through heat temperatures 650–900°C for 1 and 2 hours in a furnace chamber (Moodi *et al.* 2011). With the intention of improving Pozzolanic activity, there was ZnO applied in the metakaolin-cement mixtures. As well as for this, by establishing a system for acquiring better Pozzolanic reactivity, it is possible to make use of less pure treated clays as a form of supplementary cementing components which emphasizes on the results obtained from this study. By implementing such a system, it is possible to alter the capacity of zinc-contaminated materials in concrete (Taylor-Lange *et al.* 2012). Temperature and time are two variables that the production of Metakaolin depends on. Metakaolin is typically created by several different researches at different durations and temperature points. For example, the metakaolin has been arranged from thermally treated kaolin at 800°C for 1-hour duration (Saikia *et al.* 2006, Ramezanianpour *et al.* 2012). In order to prepare the metakaolin, the kaolin has been calcined at 800°C for 2-hours duration (Ambroise *et al.* 1994, Vu *et al.* 2001, Morsy 2005, Xin-tao *et al.* 2008, Lin *et al.* 2009, Liew *et al.* 2012, Zhao *et al.* 2019, Alyousef *et al.* 2020, Cao *et al.* 2020b, Cao *et al.* 2020c). A thermal treatment of kaolin at 800°C for 3-hours duration prepared the metakaolin (He *et al.* 2010, Wang *et al.* 2012). A thermal treatment of kaolin at 800°C for 6-hours duration prepared the metakaolin (Zhang *et al.* 2012). A thermal treatment of kaolin at 700°C for 12-hours duration had produced the metakaolin.(Zhang *et al.* 2005).

It should be mentioned that concrete is cast in different types and shapes including lightweight, high strength, green, porous and self-consolidating concrete. Moreover, Fresh and hardened properties are two types of concrete characteristics while the fresh properties are referred to the important features of concrete such as slump and workability. On the other hand, hardened properties include flexural strength, compressive strength, shear strength, and corrosion resistance. There are various methods applied to enhance these properties such as cementitious replacement powders the inclusion of the fibres, and surface protection (Shariati *et al.* 2010, Hamidian *et al.* 2011, Shariati *et al.* 2011a, Shariati *et al.* 2011b, Shariati *et al.* 2011d, Sinaei *et al.* 2011, Mohammadhassani *et al.* 2014a, Mohammadhassani *et al.* 2014c, Shariati *et al.* 2014, Arabnejad Khanouki *et al.* 2016, Shariati *et al.* 2016, Toghroli *et al.* 2017, Heydari *et al.* 2018, Nosrati *et al.* 2018, Shariat *et al.* 2018, Toghroli *et al.* 2018b, Ziaei-Nia *et al.* 2018, Li *et al.* 2019, Luo *et al.* 2019, Safa *et al.* 2019, Sajedi *et al.* 2019, Shariati *et al.* 2019c, Shariati *et al.* 2019f, Suhatril *et al.* 2019, Trung *et al.* 2019b, Xie *et al.* 2019, Afshar *et al.* 2020, Cao *et al.* 2020a, Naghipour *et al.* 2020a, Qi 2020, Toghroli *et al.* 2020).

There are different types of formed steel used in construction applications such as hot and cold framed sections. Cold framed steel connections are employed in industrial and storage applications. Hence, the performance of rack connections has been analyzed in different conditions to improve their performance (Shah *et al.* 2015, Shah *et al.* 2016a, Shah *et al.* 2016b, Shah *et al.* 2016c, Chen *et al.* 2019, Naghipour *et al.* 2020b, Zhao *et al.* 2020a).

The shear strength and load transmitting potential of the composite structures can be improved by shear connectors, which are one of the most fundamentals parts of composite systems. Since the shear connectors performance can be affected by high temperatures, several investigations have been carried out to address the strength loss of these elements (Shariati *et al.* 2011c, Shariati *et al.* 2012b, Shariati *et al.* 2012a, Shariati *et al.* 2012e, Shariati 2013, Shariati 2014, Khorramian *et al.* 2015, Shariati *et al.* 2015, Khorramian *et al.* 2016, Shahabi *et al.* 2016a, Shahabi *et al.* 2016b, Tahmasbi *et al.* 2016, Khorramian *et al.* 2017, Hosseinpour *et al.* 2018, Ismail *et al.* 2018, Nasrollahi *et al.* 2018, Paknahad *et al.* 2018, Wei *et al.* 2018, Davoodnabi *et al.* 2019, Cao *et al.* 2020d, Liu *et al.* 2020, Luo *et al.* 2020, Razavian *et al.* 2020).

Artificial intelligence methods and classic numerical techniques have been combined together which created the new hybrid algorithms in order to solve the sophisticated problems. In this regard, experimental data can be employed for optimization and prediction process. It is worthwhile to mention that several approaches have been applied in recent papers such as extreme learning machine,

Table 3 Chemical composition of OPC, Clay and developed Pozzolanic material

Constituents	Percentage by the weight of Cement	Percentage by the weight of Clay	Percentage by weight of produced Pozzolanic material	Sum of SiO2+ Al2O3+ Fe2O3 Produced Pozzolanic material
SiO_2	20.75	49.89	56.01%	
Al2O3	5.14	21.51	20.37%	
CaO	60.79	1.51	2.91%	
MgO	3.1	1.53	1.39%	92.67 %
Fe_2O_3	3.15	18.35	16.29%	
K_2O	-----	4.89	1.05%	
Na_2O_3	-----	1.23	1.09%	
TiO_2	-----	1.09	0.89%	
LOI (%)	1.70	6.3	0.5	
Specific Gravity	3.14	2.65	2.62	

neural network, and genetic programming (Shariati *et al.*, Arabnejad Khanouki *et al.* 2011, Daie *et al.* 2011, Sinaei *et al.* 2012, Mohammadhassani *et al.* 2013, Mohammadhassani *et al.* 2014b, Toghroli *et al.* 2014, Mohammadhassani *et al.* 2015, Toghroli 2015, Mansouri *et al.* 2016, Safa *et al.* 2016, Toghroli *et al.* 2016, Sadeghipour Chahnasir *et al.* 2018, Sari *et al.* 2018, Sedghi *et al.* 2018, Toghroli *et al.* 2018a, Katebi *et al.* 2019, Mansouri *et al.* 2019, Shariati *et al.* 2019b, Shariati *et al.* 2019d, Shariati *et al.* 2019e, Shariati *et al.* 2019g, Trung *et al.* 2019a, Xu *et al.* 2019, Alaskar *et al.* 2020a, Armaghani *et al.* 2020, Safa *et al.* 2020, Shariati *et al.* 2020a, Shariati *et al.* 2020c, Shariati *et al.* 2020d, Shariati *et al.* 2020e, Shariati *et al.* 2020f, Zhao *et al.* 2020a).

Engineers should consider seismic events, which are devastating for building in the construction process. Therefore, in order to improve the dynamic performance of the structures, different elements are designed by researchers. Dampers and reduced section connections (RBS) are the two most applicable elements that can be useful in this regard. In addition, the structural behavior of shear connectors and rack systems has been investigated through different experiments such as monotonic and full cyclic, half-cyclic, and reversed cyclic tests (Arabnejad Khanouki *et al.* 2010, Jalali *et al.* 2012, Shariati *et al.* 2012c, Shariati *et al.* 2012d, Shariati *et al.* 2013, Khorami *et al.* 2017a, Khorami *et al.* 2017b, Shariati *et al.* 2017, Shariati *et al.* 2018, Zandi *et al.* 2018, Milovancevic *et al.* 2019, Shariati *et al.* 2019a, Alaskar *et al.* 2020a, Alaskar *et al.* 2020b, Shariati 2020, Shariati *et al.* 2020b, Shariati *et al.* 2020g, Zhao *et al.* 2020b).

The production and manufacturing of just one ton of cement results in 5-8% of the global CO_2 emission, as it emits about 0.8 tons of CO_2 into the air (Janotka *et al.* 2010). Additionally, it is notable to mention how concrete is the most common construction material that is applied and because of that, cement is heavily manufactured and applied. In Pakistan for example, the price of cement increases daily. There is a great need for supplementary material to produce cement in an environmentally friendly manner, to reduce CO_2 emissions as well as the cost of construction. Hence, the purpose of this research is to create Pozzolanic material that can be applied in concrete through the naturally attainable clay from Sindh, Pakistan. This is because it has yet to be examined and used a supplementary cementing material.

2. Experimental work

2.1 Materials

The type of cement utilized throughout this investigation were typical Portland cement (OPC), Lucky brand, following ASTM C150 type I guideline. Meanwhile, the clay that is naturally available in billions of tonnes in the region of Dadu in Sindh, Pakistan had been applied in this study. The natural clay locally available at Nai Gaj Dam site, district Dadu, Sindh Pakistan is used as raw material to develop the Pozzolanic material. In the discussion and results section of this study, the mineralogical and chemical composition along with the physical properties of ordinary Portland cement, developed Pozzolanic material along with raw material clay. The sand that was adapted inside the mortar was in fact clean, natural hill sand that retained on sieve no.60 and went through sieve no. 30.

2.2 Mortar specimens preparation

The mortar specimens were readied with replacing 20% of the cement with activated materials through weight. Due to ASTM C 311, the mixes of mortar were constructed through applying a sand-to-cementitious material ratio (S/C) that was 2.75 as well as a W/C at 0.49.

Table 1 provides the ratio and portion of each ingredient used in details. In correlation with ASTM C305 (ASTM 2014) as well as ASTM C109 (ASTM 2013), mixing, compaction along with molding had been carried out. Specimens had been cast and then removed from the molds 24 h later and also preserved for 7 and 28-days curing, fully in a wet environment.

3. Results and discussion

3.1 Mineralogical composition of natural clay and developed pozzolanic material

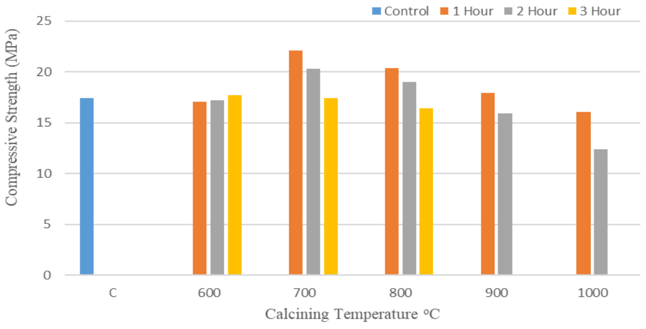

Fig. 1 Comparison of compressive strength of mortar at 7 days of control mix and mortar with inclusion of local clay activated at different temperatures and duration

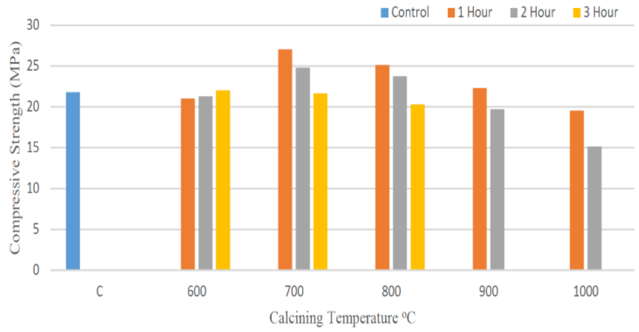

Fig. 2 Comparing the compressive strength of mortar at 28 days of control and mortar with inclusion of Calcined clay activated at different temperatures and duration

In Table 2, the findings about the X-ray diffraction tests regarding mineralogical composition of developed Pozzolanic material along with raw clay material.

Noticeably, Table 2 presents data proving how after the calcination of natural Clay, the quartz's quantity (raw impurities) decreased from 53.5 to 39.1%. Research by (Tironi *et al.* 2013) examined how compressive strength is able to be kept up and potentially increased through substituting 30% of cement in mortars through heated clays containing a soaring amount of impurities (higher than 40% from quartz).

3.2 Chemical and physical properties of natural clay and developed pozzolanic material

The findings on Natural Clay's chemical and physical properties and developed Pozzolanic material are presented in Table 3.

As per ASTM C 618, the base prerequisites for a Pozzolanic material is $SiO_2+Al_2O_3+Fe_2O_3 \geq 70\%$. Meanwhile, for metakaolin, it is $SiO_2+Al_2O_3+Fe_2O_3 \geq 85\%$ (Rashad 2013). Table 3 shows how $SiO_2+Al_2O_3+Fe_2O_3$, is in fact equivalent to 92.67%. What this means is that the EDS analysis displayed how the metakaolin changed from natural clay material. Thus, the chemical composition of Clay that has been treated (developed Pozzolanic material) meets the requirements for that of natural Pozzolanic material along with metakaolin, and rationalizes its usage in concrete in line with ASTM C-618 (ASTM 2001).

3.3 Compressive strength

Mortars that had 20% calcined clay were examined through compressive strength tests, with the intention of finding out the reactivity of activated natural clay. In line with ASTM C109 (Standard 2010), sample tests were conducted at the ages of 7 and 28 days, consequently prior to wet curing.

3.3.1 Compressive strength at 7 days

Fig. 1 shows the contrast of 7 days of the compressive strength of mortar created with inclusion as well as replacement of cement along with natural clay material that has been calcined at differential durations as well as temperatures.

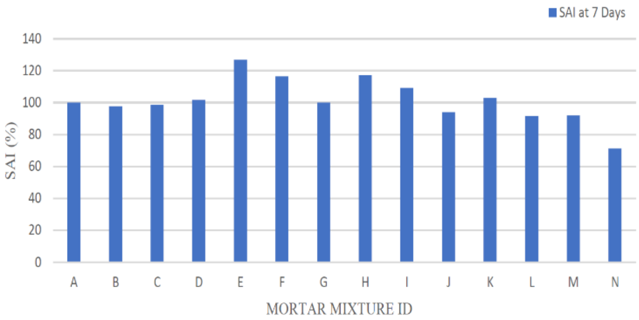

Fig. 3 SAI at 7 days of mortar with inclusion of Calcined clay activated at different temperature points as well as durations

The findings gathered in Fig. 3 shows how the substitution of cement by thermally activated clay calcined at 700°C at 1-hour duration showed a maximum compressive strength of 22.12 MPa, which is 27.05% more than that of control mortar. The reaction ability of treated Calcined clay increases when the temperature increases has been discovered from Fig. 1. Although, as the temperature approaches 800ºC, reactivity decreases as recrystallization commences.

Additionally, contrasting the diagrams in Fig. 1, what is observed is how increasing the heating duration from 1 hour in all temperatures of treated calcined clay, compressive strength decreases except the temperature of 600°C. F (Ambroise *et al.* 1992) had studied that at high temperatures, since dehydroxylation has fully undergone, further increasing the temperature can cause further development of crystals. The implications would be a decrease in the particular surface of metakaolin as well as a decrease in the compressive strength as well as reactivity of mortar samples.

3.3.2 Compressive strength of mortar at 28 days

Results regarding the mortar's compressive strength i.e. control mortar and mortar prepared with 20% cement replacement with Calcined clay tested after twenty-eight days are given below in Fig. 2.

The findings provided in Fig. 2 shows how the replacement cement through clay that has been thermally activated, Calcined at 700°C at 1-hour duration gave maximum compressive strength 27.05 MPa which is

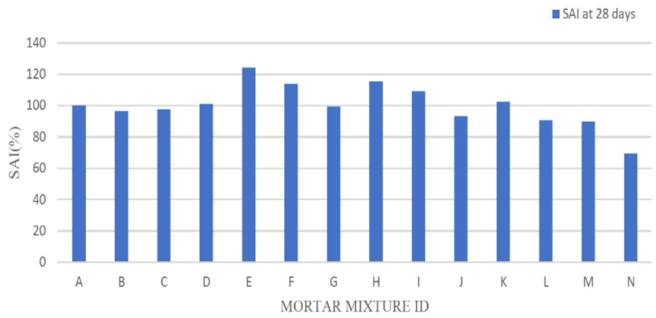

Fig. 4 SAI at 28 days of mortar including Calcined clay activated at different temperatures and durations

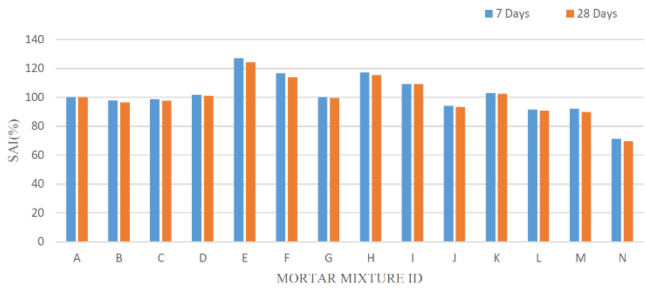

Fig. 5 Comparison of SAI at 7 and 28 days of mortar containing different samples of Clay, activated at different temperature points as well as duration

24.31% more than that of control mortar. From Fig. 2, the trend shows how Calcined clay's ability of reaction does in fact increase when temperature is increased as well. Although, when the temperature is approaching 800°C, the recrystallization starts and meanwhile, the reactivity decrease.

Research by (Ambroise *et al.* 1992) portrayed how calcination that occurs below a point of 700° Cleads to less reactive metakaolinite along with more residual kaolinite. At points above 800°C, crystallization happens while reactivity declines. Research by (Sabir *et al.* 2001) stated how the thermal activation temperature of kaolin clay is typically ranging between 700-800°C which is highly important as it relies on the base mineral that is applied.

3.4 Strength Activity Index (SAI)

Research by (Tironi *et al.* 2013) discovered how the strength activity index (SAI) indeed provides the most accurate as well as reliable approach for analyzing Pozzolanic activity throughout time. It also dismisses the contribution of the Pozzolanic effect in order to densify microstructures.

3.4.1 Strength Activity Index (SAI) at 7 days

The outcomes from SAI of control mortar and mortar prepared with 20% cement replacement with Calcined clay tested after twenty-eight days are given below Fig. 3.

Fig. 3 demonstrates The strength activity index results conducted for 7 days, clearly indicates how SAI (%) for every mortar mixture that has activated samples of calcined clay are much higher than the controlled mixture except 600°C at 1hr and 2hr, 800°C at 3hr, 900ºC at 1 hr and 1000°C at 1hr and 2hr. Although, the maximum SAI (%) can be seen in E mix, where cement has been replaced with calcined clay treated at 700°C for 1-hour duration, which is 127.05%, which satisfy the requirements for new Pozzolanic material in order to be applied inside mortar/concrete (i.e., 75%) as per the ASTM C 618.

3.4.2 Strength Activity Index (SAI) At 28 days

The strength activity index of mortar for twenty-eight days is given below in Fig. 4.

Maximum SAI is achieved at E mix, at E mix SAI is 124.31% is achieved and E mix is prepared with 20% replacement with Calcined clay at 700°C for 1hr.

3.4.3 Comparison SAI Results of 7 And 28 days

The comparison of the strength activity index of mortar prepared with 20% cement replacement with Calcined clay at different temperature points as well as different time durations for 7 days and 28 days is shown in Fig. 5

Fig. 5 demonstrates the findings of the strength activity index. The outcomes display the SAI (%) of six mortar mixes that have Calcined clay are seemingly much higher than the control mix. It is also noticeable how From Fig. 5, it is also clear how the SAI for every modified mix prepared with Calcined clay at 7 days is more than that of SAI at 28 days. Although, the greatest SAI (%) is seen in the E-mix, which has actually been made with cement replacement as well as activated clay at 700°C for a 1-hour duration. The highest SAI (%) achieved is over 120%, which is significantly higher than the needed SAI (%) which is 75% in order to be applied as Pozzolanic material in concrete, as following ASTM C 618.

4. Conclusions

The conclusions below were drawn from the outcomes of EDS<XRD and SAI (compressive strength of mortar):

- Local clay material is able to be transformed into Pozzolanic material (metakaolin) when heated at a temperature of 700°C for 1 hour.
- Because of the Pozzolanic reaction (treated Clay), the strength of mortars of most of the modified samples at 7 days as well as 28 days that consist of Calcined clay has improved when contrasted with the OPC control sample.
- Within an optimal activation temperature 700°C with 1 h being the duration of heating, maximum improvement could be seen in the mortar's compressive strength i.e., 24% more as compared to control mortar.
- The chemical composition, strength activity index and loss on ignition of the developed Pozzolanic material (Metakaolin) satisfy the provision for natural Pozzolanic material to be applied through ASTM C-618 in concrete.
- Based on results of strength activity index and chemical composition as per ASTM C-618, the treated Clay at 700°C with heating duration of 1h is recommended as Natural pozzolanic material to be applied in concrete/mortar.

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