Exposure of calcined clay and low calcium flyash-based mortar on moderate acid environment

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Abstract. The resource management is one of the crucial issues in managing the various natural resources available. Presently, the application of cement in construction industry is very frequent in producing different structural elements. Such use of cement in preparing structural formworks emits a reasonable amount of carbon dioxide and other hazardous gases into the atmosphere during the manufacturing of cement. As a result, these mechanisms pollute the environment and degrade the thickness of the protective layer of ozone. Several concerns and measures have been taken to lower the footprint of carbon emission. One of them is the adoption of pozzolana. In the present study, Class F fly ash and calcined clay is taken as the substitute for ordinary Portland cement in 4%, 8%, 12%, 16%, and 20% for making mortar cubes cured up to 90 days. The cubes were subjected to the analysis of consistency, initial and final setting times, compressive strength, and acid attacks. The study revealed the interaction of pozzolans with the properties of the mortars and dependencies of the present proportions in forming the strength of the mortar cubes.

Keywords: Class F Fly Ash; Calcined Clay; Ordinary Portland Cement; Compressive Strength; and Acid attack.

1. Introduction
Now a days, the production of structures is cement-based confirming to the wide adaptively of cement as a second largest consumable product in construction history. The structures made from cement ensure relative serviceability and durability under feasible climatic condition.

Cement is frequent in construction practices for manufacturing the cement-based structures [1]. It is widely acceptable material utilized in producing various major construction infrastructures like dams, connecting bridges, roadways and the high-rise structures. The increased population and growing demand of the residential, industrial and the commercial platform will cause cement application to grow faster [2], [3]. In a report of IBEF, it is indicated that the cement demand will grow up to approximately 600 million tonnes per annum by 2025 due to share contribution in various domain as prescribed above [4]. Cement as a binding material is an energy intensive product that consume natural resources and release harmful gases into the atmosphere [5]–[7]. These gases are the main sources for depleting the environmental ozone layer [8]. Hence, several concerns have been taken to
reduce the effect of harmful gases. One of them is the application of the pozzolana based materials in the preparation of cement based construction materials [9]–[11]. Some of widely used pozzolans are calcined clay, metakaolin, fly ash, silica fume, palm oil fuel ash and ceramic waste powder [3], [8], [12]–[14]. The reactivity and diffusivity of the pozzolanic ions depends upon the type of pozzolana used, concentration, and the control environment during the manufacturing of structural samples. Several admixtures such as water reducers, retarders, and accelerators are also utilized along with pozzolans to improve the properties of the cement products by various researchers[15], [16].

Paiva et al [11] demonstrate the influence of the metakaolin and diatomite pozzolana on the durable characteristic of the specimens. They revealed that metakaolin was capable of high reactivity against diatomite. Although, both the pozzolans were capable of decreasing the penetrability of chloride ions in the structure but metakaolin at a greater rate. Soriano et al [17] carried out his study on metakaolin and fluid catalyst based cement paste and mortar. The study was focused on determination of the hydration rate of cement paste and mortar cured at different curing conditions of temperatures. They reported that the replacement of the cement with fluid catalyst at 15% shows excellent results in comparison with the other pozzolans in visualizing the pozzolanic reaction and hydration reactions. They concluded that the fluid catalyst at low temperature of 5°C exhibit enhanced strength and more dense structure of the system investigated by scanning electron microscopy. Wu et al [18] investigated the role of fly ash morphology on the behaviour of paste made from magnesium oxychloride cement (MOC). Total six proportions of fly ash as 0%, 15%, 20%, 25%, 30%, and 35% by weight of magnesium oxide were selected and investigated for setting times, fluidity and mechanical properties of the magnesium oxychloride cement paste and were subjected to the analysis through SEM and XRD. They revealed that the setting time of the paste was retarded while the phase composition of the paste was not affected by fly ash in paste. In another study, Görhan and Kavasoğlu [19] also used fly ash as a substitution of the cement in making mortars cured for 28 day and 90 days. The mortars were subjected to the investigations for mechanical strength and durability properties with the provision of microfibers. They showed that mortar cured for 90 days possess highest strength and replacement of fly ash above 30% indicates lowest strength in the mortar specimens. Also, the length of microfiber influences the mortar system. They experimentally propose the 12 mm length of fibre as best selection in achieving the greater durability of the mortar. Tangpagasit et al [20] demonstrated the pozzolanic reaction and packing effect of fly ash in making mortar samples when fly ash replaced with 20% mass of cement. They revealed that the curing period of mortar and median particle size of the fly ash influences the strength. Also, the bigger sized particles of fly ash are not recommended to use as a pozzolanic material in the construction. Frías et al [21] investigated the heat evolution of the pozzolans based on the pozzolanic activity of the materials. They studied the difference of pozzolanic hydration rate in mortar, concrete and simultaneously compared those results with different pozzolanic hydration rates. They revealed that the rates of hydration of metakaolin and silica fume were found to be similar while the pozzolanic activity of silica fume was greater than metakaolin. Tomar et al. [22] shows in his study that the external environment affects the nature of the cement paste, mortar, and concrete. Such phenomenon could lead to the deterioration of the structures.

In this research article, it is made possible to study the interaction of different pozzolans. The consistency, setting times, compressive strength and acid attack are some of the highlighted tests to understand the role of calcined clay and fly ash. This study demonstrates the action of acid on the pozzolana prepared mortar cubes. The action of sulphuric acid in the
desired curing period is well demonstrated in the present study. The experimental study could be beneficial in analysing the behaviour of pozzolana based mortar cubes in the controlled environment.

2. Experimental

2.1. Materials Used

2.1.1 Cement: Cement is the essential part of the study. Cement is generally required to bind the aggregate particles and to harden the mortar system. In the present study, Ordinary Portland Cement (OPC) of 43 grade was used which ensure the specifications suggested by the IS 8112-2013 [23]. The cement has the specific gravity of 3.14. OPC of grade 43 was provided by the GLA University, Mathura itself. The chemical property of the cement is shown in Table 1.

| Compound | OPC, (%) | Class F Fly Ash, (%) | Calcined Clay, (%) |
|----------|---------|---------------------|-------------------|
| SiO₂     | 26.34   | 52.65               | 48.12             |
| Al₂O₃    | 8.5     | 23.23               | 32.03             |
| Fe₂O₃    | 4.8     | 11.15               | 1.65              |
| CaO      | 45.41   | 5.05                | 0.65              |
| SO₃      | 2.7     | 0.8                 | -                 |
| Na₂O     | 0.15    | 1.0                 | 0.15              |
| K₂O      | 0.05    | 2.0                 | 0.11              |
| LOI      | 2.52    | 2.8                 | 14.20             |

2.1.2 Fly Ash: Fly ash is an industrial waste by-product and a pozzolanic material, arises due to combustion of pulverized coal in thermal power plant. According to ASTM-C618, there are two classes of fly ash; Class C and Class F fly ash. Class-F fly ash has low calcium content (usually lime) than Class-C fly ash [24], [25]. This difference in lime content is arises due to the pulverization process which utilized different coal to produce electricity. This dissimilar profile of coal produces the variation in fly ash in terms of morphology, physical and chemical composition of the fly ash [26], [27]. In the present study, Class F fly ash is used for the preparation of mortar samples. The chemical composition of the Class F fly ash is shown in Table 1.

2.1.3 Calcined Clay: Finer particles are added to the system to denser the medium resulting in prevention of undesirable agents into the matrix. As a result, improved serviceability and durability of the structure obtained. Calcined clay is added to the mortar specimen in the fixed proportion. The application of calcined clay reduces the impact of CO₂ emission on the environment. Presently, calcined clay is used as a substitution of the cement in 04%, 08%, 12%, 16%, and 20% by mass of cement. Usually, calcined clay is whitish in colour and has finer particles than cement. The fineness modulus and specific gravity of calcined clay was 2.20 and 2.65 respectively. The chemical composition of calcined clay is shown in Table 1.
2.1.4 Fine Aggregate: Locally available sand passed through IS sieve size 2.36 mm was used in the present study. The sand was provided by the GLA University, Mathura itself from a local vendor. The various properties of fine aggregate were tested in accordance with IS 383: 2016 [28] and can be seen in Table 2. The sand was clean and free from impurities like vegetation, leaf, and mud etc. Fine aggregate acts a filler material in mortar specimen and contribute to the denseness of the structure.

| Physical Property          | Results                                      |
|----------------------------|----------------------------------------------|
| Compacted Bulk Density     | 1645 kg/m$^3$                                |
| Specific Gravity           | 2.67                                         |
| Fineness Modulus           | 2.45                                         |
| Water Absorption, %        | 1.0                                          |
| Particle Size              | Particles passing through IS Sieve size 4.75 mm |

2.1.5 Water: Water is essential for the hydration of cement and the pozzolanic particles. The strength and durable features of the specimen are greatly depending upon the quality of the applied water. Water used in this study was free from impurities from the external sources. Clean potable water with total dissolved solids contents up to 1665 ppm and an average pH of 7.2; taken from GLA University, Mathura, India; was used in the present study. The quality characteristic of the curing water can be seen in Table 3.
Table 3: Tests on Tap Water

| Quality Characteristic | Alkalinity | Hardness | Chloride Content | pH | TDS |
|------------------------|------------|----------|------------------|----|-----|
| Result                 | 450 mg/l   | 1480 mg/l| 955 mg/l         | 7.2| 1665 mg/l |

2.2. Testing Methods and Preparation

2.2.1. Mortar Composition and Ingredients: In order to calculate the compressive strength, several mortar cubes of size 70.6*70.6*70.6 (in mm.) were casted in accordance with IS 516 [29]. The cement used in; was also replaced in 04%, 08%, 12%, 16%, and 20% of calcined clay and fly ash content. A fixed proportion of 1:3 of cement to fine aggregate was also kept constant during the experiment. The cement mortar cubes were subjected to the curing at an age of 3, 7, 28 and 56 days. Also, some mortar cubes were immersed in 3% sulphuric acid for 14, 56, and 90 days respectively for observing the behaviour in extreme environment.

2.2.2. Consistency and Setting Time: Cement paste was used for checking the consistency and setting time of the cement. Approximately 200 gm of dry cement were utilized for preparing the cement paste. The consistency of the cement is the minimum amount of water required to hydrate the cement particles as a consequence of chemical reaction between water and cement. Consistency of cement were checked by replacing calcined clay and fly ash in 04%, 08%, 12%, 16%, and 20% respectively as per recommendations of IS 4031 Part 5.

Consistency and setting times were calculate using Vicat apparatus confirming to IS 5513-1976 of the cement pastes. Around 200 gm of cement was needed initially. Afterward, water was added to the cement for preparing cement. The prepared cement was then filled in Vicat’s mould to determine the consistency, initial setting time, and final setting time of the cement. A little vibration is generally applied to this mould for ensuring the complete homogeneity of the paste in the mould. Now, release the needle and observe the penetration for consistency and setting times. During the measurements, the room temperature was recorded as 27±2 °C.

2.2.3. Compressive Strength: A total of 144 mortar cubes were prepared for the analysis of compressive strength of the mortar. The compressive strength of the mortar cubes were checked on compression testing machine in accordance with IS 516: 2014 [29]. Before implementing the compression test on cubes, the cubes were well cured for 3, 7, 28, and 56 days. After curing, the cubes were sun dried and then consider for the test. Averages of three were taken as the compressive strength of the mortar cubes.

3. Results and Discussion

3.1. Consistency: Determination of consistency is very essential for the analysis of the workability and compressive strength test. It determines the resistance to shear deformation. An optimum content of water is necessary to hydrate the cement particle which in turn initiates the chemical reaction. The adoption of water less than this quantity can hinder the chemical reaction between cement and water [30]. Such reduction results in reduced strength. Fig. 2 shows the consistency of various substitutions of calcined clay and fly ash respectively. Consistency of each mix has been calculated with pozzolana replacement. From Fig. 2, it can
be seen that fly ash up to 12% by weight of cement shows decreased consistency as compared to the others while calcined clay up to 16% by weight of cement exhibit increased consistency. Beyond 12% fly ash replacement, gradual increase in consistency was observed. Also, beyond 16% calcined clay replacement, consistency of the paste decreases. Consistency of cement is affected by several factors such as temperature, humidity, fineness of cement and pozzolana, and method of water mixing [26]. As a result, the changes in the consistency are general in its nature.

![Figure 2: Data analysis for Consistency of mortar prepared with fly ash and calcined clay](image)

3.2. Setting Times: Setting time of a cement paste is an essential test to understand the rate of setting time after period of time. Setting time is affected by the relative humidity of the atmosphere, temperature variation and the moisture content present in the sample. Initial and final setting times were calculated on 4, 8, 12, 16, and 20% replacement of the cement with calcined clay and fly ash respectively. The progress of initial and final setting can be seen in Fig 3 and 4 respectively. From Fig 3, it can be concluded that the fly ash has the maximum initial setting time at 16% replacement. Afterwards, the initial setting time of fly ash decreases significantly. On the other hand, initial setting time of calcined clay was seen to be increasing as the proportions increases. The fly ash has the range of initial setting time from 32 min. to 40 min. while the calcined clay has 40 min. to 58 min respectively.

![Figure 3: Initial setting time statistics for fly ash and calcined clay](image)
The final setting time is the time at which needle makes an impression over the paste. Final setting time is essential to observe the hardening rate of the structure. Presently, the final setting time of both the pozzolana were seen to be increasing however, calcined clay shows the higher rate of setting time. Fig. 4 shows the final setting times of the paste composed of fly ash and calcined clay substitution.

![Final Setting Time Analysis](image)

**Figure 4:** Final setting time analysis of mortar prepared with fly ash and calcined clay

3.3. Compressive Strength: The effect of the fly ash and calcined clay on compressive strength can be seen in Fig. 5 and 6 respectively. From Fig. 5, it can be seen that in case of mortars containing fly ash, the compressive strength was lower than conventional mortar cubes at 3 days. However, such reduction in strength can be a measure of slower pozzolanic activity of the fly ash. The curing periods of the mortar cubes enhances the compressive strength. From Fig. 5, it can be concluded that the 12% fly ash mix proportion exhibit the maximum compressive strength over the other fractions. The compressive strength of mortar prepared with calcined clay and fly ash exhibit maximum strength at 12% replacement at 56 days. The strength beyond 12% replacement was seen to be decreasing. Both the materials were not able to gain strength beyond 12% substitution.
3.4. Observations for Acid Attack: Table 4 represent the percentage mass loss of various mortar cubes after submerging the samples into 3% sulphuric acid for an interval of 7, 14, 28, 56, and 90 days. Similarly, Table 4 showing the percentage loss of mortar cubes in compressive strength when samples were subjected to the 3% sulphuric acid immersion for curing period as specified above. Presently, the mortar samples were subjected to the observations for percentage mass loss and percentage loss in compressive strength under acidic conditions. As a result, reduced strength and deteriorate surface can be achieved as shown in Fig. 7 due to the chemical reaction of sulphuric acid with the calcium silicate hydrate gel available in mortar composition. The sulphuric acid (H$_2$SO$_4$) breaks into H$^+$ and SO$_4^{2-}$ which in turns attacks to the pore structure of the mortar thereby starts the deterioration process. The breakdown of H$^+$ can directly affects the CSH formation and reduces the pH.

Figure 5: Compressive strength analysis of mortars prepared with fly ash

Figure 6: Compressive strength analysis of mortars prepared with calcined clay
This reduced pH results in the dissolution of the CSH and CH products. Subsequently, lower strength and reduced mass loss is achieved.

### Table 4: Change in mass of cement and compressive strength of mortar specimen

| Curing Period | Percentage loss in weight | Percentage loss in Compressive Strength |
|---------------|---------------------------|----------------------------------------|
|               | OPC    | Fly Ash | Calcined Clay | OPC    | Fly Ash | Calcined Clay |
| 7 Days        | 22%    | 23%     | 23%            | 23%    | 18%     | 14%           |
| 14 Days       | 27%    | 26%     | 26%            | 26%    | 21%     | 16%           |
| 28 Days       | 31%    | 30%     | 30%            | 30%    | 25%     | 19%           |
| 56 Days       | 33%    | 34%     | 34%            | 34%    | 34%     | 26%           |
| 90 Days       | 40%    | 45%     | 45%            | 45%    | 36%     | 32%           |

From Table 4, it can be concluded that Ordinary Portland cement showing maximum loss in strength and mass respectively. Table 4 is drawn with a fixed proportion of 12% calcined clay and fly ash only as the higher strength obtained in Fig. 6 & 7 respectively. Moreover, change in w/c ratio to higher one may show higher reduction in strength and a greater loss of specimen weights. Calcined clay mortar shows better results in resisting the acidic environment over fly ash and OPC mortar specimens. At 90 days, the loss in strength and mass loss were achieved as 32%, 35% and 23%, 35% respectively.

### 4. Conclusions

Following conclusions can be made based on the study:

1) Fly ash up to 12% by weight of cement shows decreased consistency as compared to the others while calcined clay up to 16% by weight of cement exhibit increased consistency.

2) Both initial and final setting of the calcined clay and fly ash paste increases with increasing the percentage of replacement. Paste with 16% fly ash substitution shows maximum initial setting time while paste with calcined clay showing gradual increasing initial setting time with varying fractions.

3) The compressive strength of mortar prepared with calcined clay and fly ash exhibit maximum strength at 12% replacement at 56 days. The strength beyond 12% replacement was seen to be decreasing.

4) OPC specimens were seen to be deteriorating due to more susceptibility of diffusion of sulphate ions in the formation. Calcined clay revealed better results over OPC and fly ash mortar samples in resisting sulphated environment.

5) At 90 days, 32% loss in strength were achieved in calcined clay mortar while it was 36% in fly ash and 45% in OPC mortar hence calcined clay based mortar exhibit better resistance over fly ash and conventional mortar samples.
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