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Development of Novel Constructional Material from Industrial Solid Waste as Geopolymer

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Abstract

In present investigations, Geopolymer is made from thermal power plant waste obtained such as Pond Ash. The mechanism involved is that the silicon and aluminium present in the waste reacts with alkali liquid, forming Geopolymer which binds other non-reactive materials in the waste. The highest strength level achieved for as-prepared Geopolymers 19 MPa by curing 1 day. The strength level of Geopolymeric products are 22 MPa (Geopolymeric Motar) and 31 MPa (Geopolymer concrete), respectively. SEM micrographs reveal that there is a gradual transformation from irregular spherical shape to compacted mass which is due to polymeric transformation with increase in curing time which also corroborate with mechanical properties such as compressive strength. DSC isotherms show oozing out of inbuilt water which has accumulated during condensation polymerization reaction. The strength level achieved for optimum combination of variable is found to be comparable to that of standard motar of grade (M15) as is used for constructional purpose.

Key words: Geopolymer, Pond ash, Cement and Compressive strength
1.0. INTRODUCTION

In the recent time, fly Ash based Geopolymer (GP) has emerged as promising new cement-like materials which is alternative in the field of building and construction materials [1-3]. Geopolymers show many superior properties such as high compressive strength, low creep, good acid resistance and low shrinkage [3] compared with OPC based constructional materials. The main ingredient of Geopolymers is Fly Ash which is obtained from coal burning thermal power stations and is thus very beneficial in terms of environmental impacts. Fly Ash/Bottom Ash/ Pond Ash based Geopolymer gives emphasis in reducing carbon dioxide emission [1]. Since Pond Ash is the waste products, this research can lead to the awareness of sustainable development to the society. This is very advisable in sustainable developments to reduce carbon dioxide. At present time, there is limited information on the influence of parameters on Geopolymer available, especially Geopolymer with bottom ash as the fine aggregate. As a result, study on the effect of different parameters on the Fly Ash based Geopolymer with bottom ash as sand replacement is needed. Hardjito and Rangan demonstrated that the benefit of Geopolymer concrete which is cheaper than Portland cement concrete [2,3]. Fly ash based Geopolymer products are prepared as like Geopolymer pastes [4], mortars [5] and concretes [2,3]. The inclusion of naturally occurring materials such as quartz, basalt, granite, sandstone and limestone as aggregates to Geopolymer paste is not only economically favorable but also reduces pore density, reduces crack formation and improves durability [6]. In ordinary Portland cement (OPC) concrete and mortar there is a likelihood of an alkali-aggregate reaction between reactive aggregate and alkalis present in cement (Na₂O + K₂O) and Ca(OH)₂. The product of the alkali-aggregate reaction results in expansion and subsequent crumbling of the mortar and concretes. [7] found that the calcium in the OPC mortar plays an essential role in the expansive nature of
the gels. Fly ash based Geopolymer mortars are less susceptible to alkali-aggregate reaction because the lower calcium content in these systems results in a reaction product that is not expansive [6,7]. Previous researchers have described addition of aggregate to Geopolymer with binder: aggregate at constant ratio 0.5 [5].

The present research reports on the effects of sieving size Pond Ash, curing time and curing temperature on the Geopolymerization process within the binder phase and subsequent mechanical properties of the Geopolymer. Also, the work has done for morphological transformation from as-received Pond Ash to Geopolymeric products. DSC analysis is also done.

2.0. EXPERIMENTAL DETAILS

2.1. Chemicals and Materials

The variety of chemicals and materials such as Pond Ash, sodium silicate, sodium hydroxide, Sika are required for the preparation of GP products. Bulk quantity of Pond Ash is collected from NALCO Navratna Company (Damanjodi, Odisha) and the different percentage of composition is mentioned in Table 1.

| Table 1. Chemical composition of Pond Ash (PA) |
|------------------------------------------------|
| Raw materials | SiO2 | Al2O3 | CaO | MgO | Fe2O3 | TiO2 | Cr2O3 | MnO | P2O5 | C | LOI |
| Pond Ash      | 62.8 | 28.3  | 0.7 | 0.58 | 3.85  | 1.84 | 0.04  | 0.03 | 0.32 | 1.15 | 0.5 |

2.2. Cured as-prepared Geopolymer

Pond Ash based Geopolymer is prepared by taking in the amount of as-received Pond Ash, sodium silicate, sodium hydroxide, and water soluble plasticizers. The process is carried out in
following three steps such as Grinding, Mixing and Ramming followed by curing using hot air oven [9].

**Step 1**: The as received Pond Ash from NALCO Damanjodi is grinded by ball mill followed by sieving through different mesh size such as 240 meshes, 200 meshes and 150 meshes.

**Step 2**: Now, we are taken 240 meshes of Pond Ash for the preparation of Geopolymeric product. The sieved 240 meshes of Pond Ash is mixed with NaOH, Na$_2$SiO$_3$, small amount water and water soluble plasticizers (Sika) in appropriate amount and gel-like material is formed. The different compositions of the mixtures are shown in Table 2.

**Step 3**: Appropriate amount of gel-like materials are put in REMI system. REMI mould is made from iron in cylindrical form, which have 50 mm diameter by 70 mm height. The samples are RAMMING twenty (20) times. It is left 3 minutes for compacting and then de-molded it. It is called as Green samples.

The casted green samples are curried at 70 ºC for 24 h in oven. The cured samples wrapped within plastic zipper bags to prevent moisture loss and then stored until the samples are tested. In this way, varieties of Geopolymeric samples are prepared by considering the different parameters such as temperature, time and mesh size.

2.3. CHARACTERIZATION TECHNIQUES

The major testing of as prepared Geopolymeric product is compression testing. It is done in compression testing machine (AIMIL COMPTEST 2000, India). Total maximum load are recorded at the point of fracture. It is followed ASTM standard. Morphological analysis of 70 ºC cured Geopolymeric products with as-received Pond ash is carried out by Field Emission Scanning Electron Microscopy (FESEM) with Energy Dispersive X-ray. It is conducted using JEOL, Carl Zeiss Supra 40 field emission scanning electron microscope. Before FESEM experiment, gold coating is done using. Operating voltage is maintained at 30 kV. Differential scanning calorimetry (DSC) of 70 ºC cured as-prepared samples is performed using a Perkin Elmer Pyris Diamond analyzer at a heating rate of 10 ºC/min in nitrogen environment.

2.4. RESULTS AND DISCUSSION

The results of compressive strength of prepared Pond Ash based Geopolymeric sample after different length of curing time at 70 ºC are summarized in Table 2.
Table 2: Variation of curing time with other parameters as curing temperature (70°C), 240 mesh size, 12% SS and 3% SH

| Sample code | Curing Time (hour) | Pond Ash | Sod. Silicate | Alkali (8 M, NaOH) | Water soluble Plasticizer (Sika) | Compressive Strength (MPa) |
|-------------|--------------------|----------|---------------|-------------------|----------------------------------|---------------------------|
| S1          | 4                  | 85%      | 12%           | 3%                | 1-2ml                            | 10.6                      |
| S2          | 8                  | 85%      | 12%           | 3%                | 1-2ml                            | 13                        |
| S3          | 16                 | 85%      | 12%           | 3%                | 1-2ml                            | 15                        |
| S4          | 20                 | 85%      | 12%           | 3%                | 1-2ml                            | 18                        |
| S5          | 24                 | 85%      | 12%           | 3%                | 1-2ml                            | 19                        |
| S6          | 168                | 85%      | 12%           | 3%                | 1-2ml                            | 20.3                      |

In case of prepared Pond Ash based Geopolymer for 7 days (168 h) curing, the compressive strength is found to be 20.3 MPa; whereas the compressive strength of PA based GP curing at 70 °C for 24 h is found to be 19 MPa. There is slight difference in compressive strength which is better than Fly Ash based Geopolymer [10] and M15 grade concrete [11]. This may believe that it is happened due to the increase in Si/Al ratio and with increasing in NaOH (alkali) %. The compressive strength value is higher in samples with higher Si/Al ratio and high alkali % (i.e., Si/Al ratio 3 and alkali % 8) [11].

Table 3: Mix proportions of Pond Ash based Geopolymers at 24 h in different temperature

| Sample code | Curing Temp.(°C) | Pond Ash | Sod. Silicate | Alkali (8 M, NaOH) | Water soluble Plasticizer (Sika) | Compressive Strength (MPa) |
|-------------|------------------|----------|---------------|-------------------|----------------------------------|---------------------------|
| S1          | 50               | 85%      | 12%           | 3%                | 1-2ml                            | 15                        |
| S2          | 60               | 85%      | 12%           | 3%                | 1-2ml                            | 16.5                      |
| S3          | 70               | 85%      | 12%           | 3%                | 1-2ml                            | 19                        |
| S4          | 80               | 85%      | 12%           | 3%                | 1-2ml                            | 15                        |
In addition, the compressive strength of as-prepared Pond Ash based GP product in different curing temperature such as 50 ºC, 60 ºC, 70 º and 80 ºC, respectively is presented in Table 2. It is clearly indicated that as the curing time increases, the compressive strength increases at 70 ºC curing temperature. The highest compressive strength is obtained at 70 ºC in 24 h curing time, whereas the lowest compressive strength is estimated at 50 ºC and 80 ºC in 24 h curing. The corresponding highest value of compressive is 19 MPa. The reason may be due to the thermal energy. It may help to the progression of chain and that gives better strength [12,13].

Table 4 Variation of mesh size with other parameters as curing time 24 h, curing temperature 70 C, 12% SS and 3% SH

| Sample code | Mesh size (micron size) | Pond Ash | Sod. Silicate | Alkali (8 M, NaOH) | Water soluble Plasticizer (Sika) | Compressive Strength (MPa) |
|-------------|-------------------------|----------|---------------|-------------------|---------------------------------|---------------------------|
| S1          | 150                     | 85%      | 12%           | 3%                | 1-2ml                           | 15                        |
| S2          | 200                     | 85%      | 12%           | 3%                | 1-2ml                           | 16.5                      |
| S3          | 240                     | 85%      | 12%           | 3%                | 1-2ml                           | 19                        |

Table 4 shows different particle size of Pond Ash which is obtained from sieving of different mesh size. During the sieving, the mesh size is 240 meshes, 200 meshes and 150 meshes. Pond Ash can be taken same percentages during the preparation of Geopolymer. The highest compressive strength is estimated to be 19 MPa and lower one is 15 MPa. It happens may be due to the effect of densities and pores present in the prepared product [14].

For the morphological analysis of the as-received Pond Ash and cured Pond Ash based Geopolymers is shown in Fig 2. For this analysis, the powdered sample is used. FESEM image of as-received Pond Ash sample is shown in Fig 2A. It is observed that the arrangement of the irregular particles is relatively loose with high porosity and small particle size; whereas prepared Pond Ash based
Fig 1. FESEM images of (A) as-received Pond Ash, PA based GP cured at 70°C for 24 h with 240 meshes (B)

Geopolymer (Fig 1B) is compacted mass. The rate of compacted mass is formed in different curing time length at particular curing temperature i.e., 70 °C. This may believe that is the formation of Geopolymer because the particles are connected to each other. EDX analysis of the as-received Pond Ash ensures the presence of particular elements in it and is shown in Fig 2 (Left side).

Fig. 2. EDS of as-received Pond Ash (left side) and PA based GP cured at 70 °C for 24 h (right side)
The main elements observed are Si, Al, Fe, Na, Mg, Ca and O. **Fig 2** (Right side) shows the EDS image of as-prepared PA based GP cured at 70 °C for 24 h. The same elements are presented as-like as-received PA. Only the difference is different peak height.

**Fig. 3.** DSC isotherms of as-received PA (A) and Optimized GP product (B)

Differential scanning calorimetry (DSC) is used to measure a number of characteristic properties of the Geopolymer pastes. From this experiment, it is likely to observe exothermic and endothermic events as well as glass transition temperatures (Tg). DSC isotherm is carried out in the temperature range of 50 °C to 300 °C and is shown in **Fig. 3** [15]. In the thermograms, the as-prepared Geopolymer showed exothermic peaks. Similar report is reported [15]. From the **Fig. 3** indicates, the DSC thermogram of Pond Ash is smooth, there is no sign of Geopolymeric reaction. The as-prepared optimized Pond Ash based Geopolymeric product is demonstrated peak in the **Fig. 3**. When the appropriate proportion of Na₂SiO₃, NaOH, SiKA (water soluble
plasticizer) and distil water of the optimized product, the exothermal peaks is occurred at a temperature ~ 110 °C. The sign indicates the Geopolymeric reaction happened. The significant peak is indicated the water’s melting point in all Geopolymer sample at 0 °C [15].

3.0. CONCLUSIONS

Optimization mechanical properties of Geopolymeric process of Pond Ash is carried out with the addition of sodium silicate, sodium hydroxide and water soluble plasticizer with different parameters such as curing temperature, curing time and meshes size. The optimum condition of preparation of Geopolymeric product is 240 meshes, 70 °C curing temperature with 24 h curing. The optimum strength of developed Geopolymeric product is found to be 19 MPa. In DSC isotherm, the water of crystallization is found to be ~115 °C. FESEM image of as-received Pond Ash and optimized Geopolymeric product shows the morphological transformation from irregular spherical shape to compacted structure. EDX data indicate the presence of elements such as Si, Ca, Al, Fe, Na, Mg, and O. From the results, it concludes that the construction products such as brick and concrete will be more sustainable in terms of environment performance, economic viability and potential compared to OPC based products.

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