Effect of Duration of Heat Curing on the Artificially Produced Fly Ash Aggregates

K N Shivaprasad 1,*, B B Das 2
1 Research Scholar, Department of Civil Engineering, NITK, Surathkal 575 025, India.
2 Associate Professor, Department of Civil Engineering, NITK, Surathkal 575 025, India.

*corresponding author: shivaprasad.god@gmail.com

Abstract. This paper presents the results of an experimental investigation on the production of artificial fly ash aggregates through the process of pelletization. The alkaline solutions like sodium silicate and sodium hydroxide was used to activate the fly ash. The alkaline solution contains 4.5% of Na2O, Si2O/Na2O ratio of 0.3 and 20% of water content with respect to mass of fly ash were used as binder in the production of fly ash geopolymer aggregates. The experiments were conducted by varying with different parameters. Curing of artificial fly ash aggregates was done with ambient temperature and heat curing (60 to 80 °C). Six levels of duration of heat curing were considered for this study. The optimum temperature and duration of curing is essential in geopolymerization reaction to achieve good characteristics of the produced aggregates. Grey relation analysis was performed to identify the effect of heat curing. Test results and grey relation analysis shows that the characteristics of the artificial produced fly ash aggregates has significantly improved with increase in temperature and duration of oven curing.

1. Introduction

In 1940s, the pelletization theory was developed and grownup significantly in the many industrial applications. In the process of extraction of metals, pelletization process is used in metallurgical industries and produced the pellets about 435 million tons in 1985. However, in construction industry for production of artificial lightweight aggregate was only about 1.2 million tons in 1989 [1]. Even it is a well-known method to produce artificial aggregates, it has not been widely used in construction sector. Since availability of natural resource and production costs relatively high for the production of artificial aggregate when compared to natural aggregate.

From last two decades the research has been going on utilizing the fly ash in the production of artificial aggregates using different additives such as cement, lime, clay and glass powder [2-5]. Factors affecting the pelletization process in producing the artificial aggregates mainly depends on the raw materials, moisture content, type and dosage of binder [2-8]. Hardening process adopted for producing aggregates such as sintering, normal water curing, autoclaving and steam curing are also one the important factor. [2-5, 9]. The engineering properties of produced aggregates is largely depends on the type of binder used and hardening process adopted [1-9].

This review indicated that the existing literature on aggregate production dealt mostly with different binders and different hardening methods adopted in the process and no study has been reported on use of alkaline solution as binder and heat cured hardening process for the production of the aggregates.
Thus, this paper discuss about the production of artificial aggregates using alkaline solution as a binder and the effect of the heat curing hardening process on the produced fly ash aggregates.

2. Materials and methodology

2.1. Materials
Fly ash was collected from Udupi thermal power plant, Karnataka, India. The physical properties and chemical compositions of the fly ash were analysed and it is presented in Table 1. Fly ash was classified as class F as per IS 3812:2013 part 1 classification [10]. Particle size distribution of fly ash is presented in the Figure 1.

Table 1: Physical and chemical properties of the fly ash.

| Parameters         | Fly ash |
|--------------------|---------|
| Specific gravity   | 2.2     |
| Blaine's fineness (m²/kg) | 260.3  |
| Chemical properties|         |
| SiO₂               | 60.65   |
| Al₂O₃              | 28.62   |
| Fe₂O₃              | 3.95    |
| CaO                | 1.70    |
| MgO                | 1.84    |
| SO₃                | 1.26    |
| Na₂O               | 1.11    |
| K₂O                | 0.11    |

Laboratory grade sodium silicate solution with silica modulus of 3.3 (8.0% Na₂O, 26.5% SiO₂, 65.5% H₂O by mass) and sodium hydroxide flakes of 98% purity were used in the preparation of alkali solution. Sodium hydroxide flakes was dissolved in distilled water to produce a sodium hydroxide solution. The alkaline solution prepared by mixing both sodium silicate and sodium hydroxide in the required proportions for the mixes and transferred to an air tight container with cap and allowed to cool for 24 h, before using in the mix.

In the present study, the Na₂O content of 4.5%, Na₂O/SiO₂ ratio of 0.3 and water content of 20% were used in the mixes. The dosage of Na₂O and water content in the alkaline solution is represented as a percentage of fly ash in the mix.

2.2. Pelletization - Production of aggregates
The pelletization of fly ash has been carried out in a laboratory disc pelletizer. A fabricated disc pelletizer as shown in Figure 2 was used in this study which has a disc diameter of 500 mm and depth 125 mm. The angle of disc maintained at 45° [1-2, 5] and 15 min duration of pelletization is used in the process of pelletization [5]. For this present investigation rotational speed of disc is maintained at 40 rotations per minute for all mixes. The pelletization process included: i) transferring of uniform fly ash which is free from lumps to the pelletizing disc, ii) alkali solution is sprayed within 3 min to the fly ash mix during pelletization.
2.3 Curing of aggregates
The produced fly ash aggregates were cured under different curing regimes as follows.

*Ambient curing:* The fly ash aggregates produced in pelletization process are kept ambient temperature conditions of $28 \pm 2 \, ^\circ\text{C}$ and relative humidity of 80% until it is tested for aggregates properties.

*Heat curing:* The fly ash aggregates produced in pelletization process are subjected to temperature of $60 \, ^\circ\text{C}$ and $80 \, ^\circ\text{C}$ for different durations such as 1, 3, 6, 12, 18 and 24 h and removed pellets after heat curing are kept in ambient temperature conditions until it is tested for aggregates properties.

3. Testing of aggregates

3.1 Efficiency and particle size distribution
The efficiency of pelletization is expressed in percentage weight of aggregates of size greater than 4.75mm (amount of pellets retained on IS sieve no 480) produced against the total weight pellets produced and it is calculated using the following equation.

$$Efficiency \ of \ pelletization = \frac{\text{Weight retained on the IS sieve no 480}}{\text{Total weight of aggregates produced}} \times 100 \quad (Eq. \ 1)$$

The distribution of particle sizes present was determined using standard set of sieves in accordance with the Bureau of Indian Standards – IS 383: 2016 [11].

3.2 Specific gravity, water absorption value and impact value
Specific gravity and water absorption tests were carried out in accordance with the Bureau of Indian Standards - IS 2386:1963 part 3 [12]. The aggregates impact value is a measure of resistance to sudden impact, which may differ from its resistance to gradually applied compressive load. The test sample consists of aggregates sized 10mm to 12.5mm. Impact value was carried out in accordance with Bureau of Indian Standards - IS 2386:1963 part 4 [13].

3.3 Crushing strength of individual pellets
The crushing strength of individual pelletized fly ash aggregate was determined using a crushing testing machine in which compressive load is applied gradually as shown in Figure 3 and crushing strength ($\sigma$) of pellet was obtained from the following equation [14] and the average of the batch is reported as the crushing strength of the pellets.

$$\sigma = \frac{2.8 \times P}{\pi \times x^2} \quad (Eq. \ 2)$$

Where, $P$ represents the failure load for the sample and $x$ is the size of aggregates or distance between the two plates. The crushing strength of pellets was tested in a batch which has a sample size of 20 numbers and the diameter of the pellets in the range of 6 to 18mm.

![Figure 2: Laboratory scale disc pelletizer.](image-url)
4. Results and discussion

The aggregates properties of the produced fly ash aggregates are accessed as described in the section 3. The obtained results of the aggregates properties for the different curing conditions are reported in the following sections.

4.1. Efficiency and particle size distribution

Based on the equation 1, the pelletization efficiency for the produced aggregates were determined and it is found to be 98 to 99 %. The results of the particle size distribution of the produced pelletized aggregates are presented in Figure 4. The average size of the artificial produced fly ash aggregates is 10.8 mm. The particle size distribution of produced fly ash aggregates were checked with the lower limit and higher limit of the specified grading for 20 mm size of IS 383 – 2016 [11] is also plotted in Figure 4.

4.2. Specific gravity

The average specific gravity of ambient cured and heat cured fly ash aggregates were found to be 2.03 and 1.95 respectively. The effect of heat curing on the average specific gravity of the artificial produced fly ash aggregates are presented in the Table 2. The heat curing aggregates shows that, the increase in the curing temperature and duration will decrease slightly in the specific gravity of the aggregates when compared to ambient cured aggregates.

| Curing Regime       | Duration of curing |  |
|---------------------|--------------------|--|
|                     | 1h     | 3h   | 6h   | 12h  | 18h  | 24h  |
| Ambient Cured       | 2.03   |      |      |      |      |      |
| Heat Cured - 60 °C  | 2.02   | 2.01 | 1.99 | 1.97 | 1.95 | 1.95 |
| Heat Cured - 80 °C  | 1.99   | 2.00 | 1.98 | 1.97 | 1.95 | 1.94 |

Figure 3: Crushing strength testing arrangement

Figure 4: Particle size distribution of the artificial produced fly ash aggregates
4.3. Water absorption (WA)
The water absorption of the artificial produced fly ash aggregates are evaluated as described in the section 3.2. It is observed that water absorption of ambient cured fly ash aggregates was 6.6% and it has increased to 11.53% and 12.40% when fly ash aggregates subjected to heat curing of 60 °C and 80 °C for 24h respectively. The effect of heat curing of fly ash aggregates on the water absorption were presented in the Figure 5 with respect to different temperature and duration of curing. It is clear that increase in the curing temperature will increase the water absorption of the aggregates by 1.75 to 1.88 times of water absorption of ambient cured aggregates. As the aggregates are subjected to heat curing, the water present in the aggregates is getting participated in the chemical reaction process which helps in improvement of strength of the pellets. However, the surface of fly ash aggregates remain dry and hence the absorption percentage increases.

![Figure 5: Effect of heat curing on water absorption of the fly ash aggregates](image)

4.4. Impact value of aggregates (IV)
The impact value of the artificial produced fly ash aggregates are accessed as described in the section 3.2. It is observed that impact value of ambient cured fly ash aggregates was 27.57% and it has improved to 24.10 and 23.50 when fly ash aggregates subjected to heat curing of 60 °C and 80 °C for 24h respectively. The effect of heat curing of fly ash aggregates on the impact value were presented in the Figure 6 with respect to different temperature and duration of curing. It is clear that increase in the curing temperature will improve the impact value of the aggregates by 12.5% to 14.75% of impact value of ambient cured aggregates. Impact value of aggregates improved as the temperature increases, because the increase in the chemical reaction of alkaline solution used.

![Figure 6: Effect of heat curing on Impact value of aggregates](image)
4.5. Crushing strength of individual pellets (CS)

The crushing strength of individual pellets are accessed as described in the section 3.3. It is observed that crushing strength of individual pellets was 2.87 MPa and it has improved to 3.68 MPa and 4.01 MPa when fly ash aggregates subjected to heat curing of 60 °C and 80 °C for 24h respectively. The effect of heat curing on crushing strength of individual pellets were presented in the Figure 7 with respect to different temperature and duration of curing. It is clear that increase in the curing temperature will improve the crushing strength of individual pellets by 28.2% to 39.7% of crushing strength of individual pellets of ambient cured aggregates. As the alkaline solution reaction with fly ash increases at higher temperature which results in the improved crushing strength of individual pellets.

![Crushing Strength of Individual Pellets](image)

**Figure 7:** Effect of heat curing on crushing strength of individual pellets

5. Grey relation analysis

In the present study, water absorption, impact value of aggregates, and crushing strength of individual pellets have been determined experimentally and considered as responses. Sahoo et al (2016) demonstrated the advantage of the Grey relational analysis in characterising the material properties [15]. Grey relational generation was carried out on a scale of 0 – 1 with the experimental data. The normalization of responses is done using the principle that lower the obtained value, it is better for impact value and water absorption. However, the higher value obtained is better for crushing strength of pellets. Grey relational generation was obtained using the following equation.

\[
x_i = \frac{\text{max } y_i(k) - y_i(k)}{\text{max } y_i(k) - \text{min } y_i(k)}
\]

(Eq. 3)

Where, \( x_i \) = value after the Grey relational generation; \( \text{min } y_i(k) \) = smallest value of \( y_i(k) \) for the \( k^{th} \) response; and \( \text{max } y_i(k) \) = largest value of \( y_i(k) \) for the \( k^{th} \) response.

Grey relational coefficients provide information on the correlation between the desired and actual experimental data. The Grey relational coefficient was computed by using the following equation.

\[
\xi_i(k) = \frac{\Delta_{\text{min}} + \psi \Delta_{\text{max}}}{\Delta_0(k) + \psi \Delta_{\text{max}}}
\]

(Eq. 4)

Where, \( \Delta_0 \) = \( \| x_0(k) - x_i(k) \| \) is the difference of the absolute value between \( x_0(k) \) and \( x_i(k) \) and \( \Delta_{\text{min}} \) and \( \Delta_{\text{max}} \) = minimum and maximum values of the absolute differences of all comparing sequences, respectively. \( \psi \) is a distinguishing coefficient (0 ≤ \( \psi \) ≤ 1) and in the present study, \( \psi = 0.5 \) is taken.

Grey relational generations (GRG) and \( \Delta_0 \) are presented in Table 3, while the Grey relational coefficients (GRC) are furnished in Table 4.
Table 3: Test results of aggregates and grey relational generations

| Curing Regime       | Duration of curing | WA   | IV   | CS   | Grey relational generations | Δ0i   |
|---------------------|--------------------|------|------|------|-------------------------------|-------|
| Natural Aggregates  |                    |      |      |      |                               |       |
|                     | 1.00               | 21.42|      |      | 1.00 1.00 1.00 0.00 0.00 0.00 |       |
| Ambient Cured       |                    | 6.60 | 27.57| 2.87 | 0.51 0.00 0.00 0.49 1.00 1.00 |       |
| Heat Cured - 60 °C  | 1h                 | 6.53 | 27.43| 2.91 | 0.51 0.02 0.03 0.49 0.98 0.97 |       |
|                     | 3h                 | 7.03 | 27.40| 2.99 | 0.47 0.03 0.10 0.53 0.97 0.90 |       |
|                     | 6h                 | 8.43 | 26.80| 3.07 | 0.35 0.12 0.18 0.65 0.88 0.82 |       |
|                     | 12h                | 9.13 | 24.63| 3.20 | 0.29 0.48 0.29 0.71 0.52 0.71 |       |
|                     | 18h                | 11.20| 24.27| 3.39 | 0.11 0.54 0.45 0.89 0.46 0.55 |       |
|                     | 24h                | 11.53| 24.10| 3.68 | 0.08 0.56 0.71 0.92 0.44 0.29 |       |
| Heat Cured - 80 °C  | 1h                 | 7.03 | 26.73| 3.01 | 0.47 0.14 0.13 0.53 0.86 0.87 |       |
|                     | 3h                 | 7.70 | 26.23| 3.11 | 0.41 0.22 0.21 0.59 0.78 0.79 |       |
|                     | 6h                 | 8.83 | 25.87| 3.31 | 0.31 0.28 0.39 0.69 0.72 0.61 |       |
|                     | 12h                | 9.53 | 24.33| 3.39 | 0.25 0.53 0.45 0.75 0.47 0.55 |       |
|                     | 18h                | 11.40| 23.93| 3.47 | 0.09 0.59 0.52 0.91 0.41 0.48 |       |
|                     | 24h                | 12.40| 23.50| 4.01 | 0.00 0.66 1.00 1.00 0.34 0.00 |       |

The Grey relational grade is calculated by summing up the weighted GRC corresponding to the responses. The Grey relational grade \( \gamma_i \) is computed using the following equation.

\[
\gamma_i = \frac{1}{n} \sum_{k=1}^{n} AP(k) \xi_i(k) \quad (Eq. 5)
\]

Where, \( n = \) number of process responses.
The Grey relational grades (GRG) computed using Eq. 5 are presented in Table 4. Higher Grey relational grade indicates ideal aggregates. Table 4 shows the response table for the Grey relational grade for various aggregates subjected to different curing regimes along with local available natural aggregates.

Table 4: Grey relation coefficients and Grey relation grades for aggregates

| Curing Regime       | Duration of curing | Grey relation coefficients | Grey relational grade |
|---------------------|--------------------|----------------------------|-----------------------|
| Natural Aggregates  |                    | WA  | IV  | CS  | 1.00 | 1.00 | 1.00 | 1.00 |
| Ambient Cured       |                    | 0.50| 0.33| 0.33| 0.39 |
| Heat Cured - 60 °C  | 1h                 | 0.51| 0.34| 0.34| 0.40 |
|                     | 3h                 | 0.49| 0.34| 0.36| 0.39 |
|                     | 6h                 | 0.43| 0.36| 0.38| 0.39 |
|                     | 12h                | 0.41| 0.49| 0.41| 0.44 |
|                     | 18h                | 0.36| 0.52| 0.48| 0.45 |
|                     | 24h                | 0.35| 0.53| 0.63| 0.51 |
| Heat Cured - 80 °C  | 1h                 | 0.49| 0.37| 0.36| 0.41 |
|                     | 3h                 | 0.46| 0.39| 0.39| 0.41 |
|                     | 6h                 | 0.42| 0.41| 0.45| 0.43 |
|                     | 12h                | 0.40| 0.51| 0.48| 0.46 |
|                     | 18h                | 0.35| 0.55| 0.51| 0.47 |
|                     | 24h                | 0.33| 0.60| 1.00| 0.64 |
In Grey relation grade Table, the GRG of the natural aggregates is 1 which is ideal aggregates and compared with the other artificial produced fly ash aggregates. The GRG of ambient cured aggregates is 0.39 and for heat cured aggregates GRG is has been increases with the temperature and duration of the heat curing. It can observed from the Table 4, GRG of the heat cured fly ash aggregates is increased from 0.39 to 0.64.

6. Conclusion
In production of artificial fly ash aggregates, it can be observed from test results that the heat curing has significantly improved the impact value of aggregates, crushing strength of individual pellets and specific gravity of the aggregates. However, the water absorption of the aggregates increased drastically. From Grey relation analysis, the overall engineering properties of the fly ash aggregates subjected to heat curing has improved upto 25% when compared to ambient cured fly ash aggregates.

7. References
[1] Baykal G, Döven AG. Utilization of fly ash by pelletization process; theory, application areas and research results. Resources, Conservation and Recycling. 2000 Jul 1; 30(1):59-77.
[2] Ramamurthy K, Harikrishnan KL. Influence of binders on properties of sintered fly ash aggregate. Cement and Concrete Composites. 2006 Jan 1; 28(1):33-8.
[3] Kockal NU, Ozturan T. Characteristics of lightweight fly ash aggregates produced with different binders and heat treatments. Cement and Concrete Composites. 2011 Jan 1; 33(1):61-7.
[4] Priyadharshini P, Ganesh MG, Santhi AS. Experimental study on cold bonded fly ash aggregates. International journal of Civil and Structural engineering. 2011 Oct 1; 2(2):493.
[5] Gomathi P, Sivakumar A. Fly ash based lightweight aggregates incorporating clay binders. 2014;
[6] Manikandan R, Ramamurthy K. Influence of fineness of fly ash on the aggregate pelletization process. Cement and Concrete Composites. 2007 Jul 1; 29(6):456-64.
[7] Harikrishnan KL, Ramamurthy K. Influence of pelletization process on the properties of fly ash aggregates. Waste management. 2006 Jan 1; 26(8):846-52.
[8] Gesoğlu M, Özturan T, Güneyisi E. Effects of fly ash properties on characteristics of cold-bonded fly ash lightweight aggregates. Construction and Building Materials. 2007 Sep 1; 21(9):1869-78.
[9] Manikandan R, Ramamurthy K. Effect of curing method on characteristics of cold bonded fly ash aggregates. Cement and Concrete Composites. 2008 Oct 1; 30(9):848-53.
[10] IS 3812-1: Specification for Pulverized Fuel Ash, Part 1: For Use as Pozzolana in Cement, Cement Mortar and Concrete. Bureau of Indian Standards, New Delhi, 2013.
[11] IS 383, Specification for Coarse and Fine Aggregates From Natural Sources For Concrete. Bureau of Indian Standards, New Delhi, 2016.
[12] IS 2386 – part 3, Methods of test for aggregates for concrete: Specific gravity, density, voids, absorption and bulking, Bureau of Indian Standards, New Delhi, 1963.
[13] IS 2386 – part 4, Methods of test for aggregates for concrete: Mechanical properties Bureau of Indian Standards, New Delhi, 1963.
[14] Gomathi P, Sivakumar A. Characterization on the strength properties of pelletized fly ash aggregate. ARPN J. Eng. Appl. Sci. 2012; 7(11):1523-32.
[15] Sahoo S, Das BB, Mustakim S. Acid, Alkali, and Chloride Resistance of Concrete Composed of Low-Carbonated Fly Ash. Journal of Materials in Civil Engineering. 2016 Oct 25; 29(3):04016242.