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# Effect of curing method on characteristics of cold bonded fly ash aggregates

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ABSTRACT

This paper discusses the influence of normal water curing, autoclaving and steam curing on the properties of a typical class-C fly ash aggregate. The 10% fines value, water absorption, and porosity of aggregates are correlated with SEM and XRD results to understand the influence of various factors and material characteristics. An increase in duration of normal water curing significantly improved the aggregate properties. Autoclaving and steam curing resulted in relatively lower enhancement in the properties as compared to normal water cured aggregate. Between the accelerated curing methods, autoclaved aggregate possessed properties closer to the normal water cured aggregate due to the dense microstructure formation. Continuation of normal water curing, after initially subjecting the aggregates to accelerated curing, exhibited only a marginal improvement in the properties.



## 1. Introduction

As aggregates occupy large volume in concrete, conversion of fly ash into aggregate facilitates high-volume utilization of fly ash and saves depleting natural aggregates. Incorporation of sintered and cold bonded fly ash aggregates in concrete not only reduces the density of concrete [1,2] but also increases the thermal and acoustical insulation [3,4] and reduces the autogenous shrinkage [5]. Bijen [6] presented an overview of the manufacturing process of artificial fly ash aggregates. Baykal and Doven [7] have investigated the parameters influencing pelletization of fly ash and reported that the angle and speed of the disc considerably influenced the density and strength of the fly ash aggregate. Harikrishnan and Ramamurthy [8] have reported that the speed of disc has significant influence on strength and water absorption of aggregate followed by the angle of disc, while moisture content was an important factor influencing the size growth of aggregate.

In order to improve the properties of fly ash aggregates (strength and water absorption) various hardening methods viz., sintering, normal water curing, autoclaving and steam curing, and binders like cement, lime, bentonite and chemical admixtures have been used [9]. Show et al. [10] have reported that an increase in sintering temperature up to 1300 °C along with increase in proportion of marine clay from 0% to 30% significantly reduced the density and water absorption of industrial sludge aggregate. Cheeseman et al. [11] also reported similar behaviour of reduction in density and water absorption with increase in sintering temperature up to 1080 °C for sintered sewage sludge ash aggregate. Addition of binders like cement and lime along with increase in duration of normal water curing (i.e., cold bonding) was reported to significantly improve the properties of fly ash aggregate [5,7,12] and requires minimum energy as compared to sintered fly ash aggregate. However, the properties of cold bonded fly ash aggregate were observed to be comparatively lower than those of sintered fly ash aggregate. This is because of use of low calcium (class-F) fly ash, which requires higher binder content and accelerated curing methods for enhancing strength and reducing water absorption. Limited studies have been reported on use of high calcium fly ash in the production of cold bonded fly ash aggregates.

## 2. Research significance

The properties of cold bonded fly ash aggregate were reported to be comparatively lower than those of sintered fly ash aggregate due to the use of low calcium fly ash and only limited studies have been carried out on high calcium fly ash. Preliminary investigation on pelletization of class-C fly ash showed that almost all the materials were converted into aggregates of size greater than 4.75 mm without any binders. Further, statistically designed two level fractional factorial experiments showed that angle and speed of the disc significantly influence the pelletization of class-C fly ash while the other factors, moisture content and duration were insignificant due to the narrow range of the factors [13]. As sintering of Class-C fly ash causes aggregate disintegration, cold bonding is the appropriate technique. Such cold bonded aggregates require curing. This study is mainly directed towards exploring the influence of various hardening methods (i.e., normal water curing, autoclaving and

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steam curing) on the properties of cold bonded fly ash aggregate made from class-C fly ash. The 10% fines value, water absorption, and porosity of aggregates cured under various curing conditions were determined. Differences in performance of the aggregates were discussed with the support of SEM imaging and X-ray Diffraction (XRD) results.

## 3. Materials and methodology

Fly ash conforming to class-C as per ASTM C 618 [14] (collected from a nearby thermal power station) having Blaine's fineness of 414 $m^2$/kg and specific gravity of 2.64 and chemical composition as shown in Table 1 was used. Pelletization of fly ash was carried out on a disc pelletizer of 560 mm diameter and 250 mm depth, which has a provision for controlling the angle, speed and moisture content. The optimum levels of these pelletization parameters (angle-55°, speed-55 rpm and moisture content-31%) were determined through a systematic experimental design to achieve maximum pelletization efficiency [13].

### 3.1. Methods of curing

Green pellets or raw pelletized fly ash aggregate, due to its lower strength, was inadequate for its use in concrete or any other application, and hence the relative effectiveness of various curing methods has been studied. The variation in properties of fly ash aggregates with age of normal water curing (at 24 °C) has been studied by soaking aggregates packed in jute bag for 3, 7, 14 and 28 days before testing. Adoption of normal water curing of aggregate requires large curing/stocking facility within the production premises. If some form of accelerated curing method can be employed to meet the performance requirements, then the aggregates can be produced and marketed quickly thereby saving the space requirement even though additional energy requirement is higher. Hence the influence of accelerated curing methods such as autoclaving and steam curing were employed to reduce the period of curing. Autoclaving of fly ash aggregates was done at a pressure of 1 MPa with 2 h pressure built-up period and steam curing at 70 °C with 90 min temperature rise period. After the pressure built-up and temperature rise period, duration of autoclaving and steam curing was varied from 5 h to 10 h with 1 h interval and than cooled for 5 h.

### 3.2. Properties evaluated

The influences of above-mentioned curing methods on the properties of aggregates have been discussed through (i) physical (porosity, and water absorption) (ii) mechanical (strength) and (iii) chemical (XRD) properties. The variation in properties of aggregates with change in curing period was also supported with microstructural studies like SEM. The properties of fly ash aggregates were measured on an oven-dried aggregate at 105 °C for 24 h, after the prescribed curing period.

#### 3.2.1. Physical properties

Water absorption: 500 g of 12.5–10 mm size oven-dried aggregate was immersed in water for 24 h and then the increased mass of saturated surface dry aggregate was measured. The water absorption is expressed as increase in mass as a percentage of oven-dried mass. The pore system of lightweight aggregate is conventionally classified as open and closed pores [15]. The permeable/open porosity was determined using the below mentioned formulae after subjecting the aggregate samples under vacuum saturation as per ASTM C 1202-97 [16]

$$\text{Open porosity (OP)} = 100 \times (W_{sat} - W_{dry})/(W_{sat} - W_{wat})$$
$$\text{Total porosity (TP)} = 100 \times (\rho_t - \rho_d)/\rho_t$$

where, ($W_{sat}$) and ($W_{wat}$) are the weight of vacuum saturated aggregates in air and water respectively (in gram), $W_{dry}$ is weight of oven-dried sample (in gram), $\rho_t$ is the true specific gravity of fly ash aggregate (g/$cm^3$) measured by pycnometer method using powdered sample passing 75 μm

$$\text{Dry particle density } \rho_d = ((W_{dry}/V) \times 100) \text{in g/cm}^3, \text{ where } V \text{ is volume of particles} = (W_{sat} - W_{wat}) \text{ in cm}^3.$$

SEM micrographs were taken on a fractured surface of 12.5–10 mm oven-dried aggregate. Oven-dried samples were mounted on metal stubs and sputtered with gold under vacuum, before subjecting to the electron beam from a JEOL–JSM 840A Scanning Electron Microscope.

#### 3.2.2. Mechanical properties

For aggregates whose crushing value is above 25% (Neville, 2000), 10% fines value (TPFV) is recommended by BS 812-110 [17] for relative measure of strength. Hence the strength of fly ash aggregate was determined through 10% fines value as per BS 812-110 [17], on 12.5–10 mm size aggregates. TPFV was determined using 75 mm crushing cylinder with depth of penetration of 8 mm on 12.5–10 mm size aggregate [17].

#### 3.2.3. Chemical properties (XRD)

Powder diffractometer using Cu Kα radiation at 40 KV and 30 mA was used for recording the XRD data with a range of measurement (2$\theta$) 5–60°. Powdered sample passing 75 μm was used in XRD analysis.

## 4. Results and discussion

### 4.1. Influence of curing methods on the properties of fly ash aggregates

#### 4.1.1. Normal water curing

Figs. 1a and 1b shows the influence of normal water curing on 10% fines value, water absorption and porosity. An increase in the duration of normal water curing (NWC) resulted in significant increase in 10% fines value (1.63–2.8 tonne) (Fig. 1a) and reduction

**Table 1**
Chemical characteristics of class-C fly ash

| Chemical content (% by mass) | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| $SiO_2$ | CaO | $Al_2O_3$ | $Fe_2O_3$ | MgO | $Na_2O$ | MnO | $K_2O$ | $SO_3$ | LoI |
| 31.62 | 17.17 | 30.11 | 8.94 | 3.71 | 0.74 | 0.02 | 0.10 | 5.72 | 3.18 |

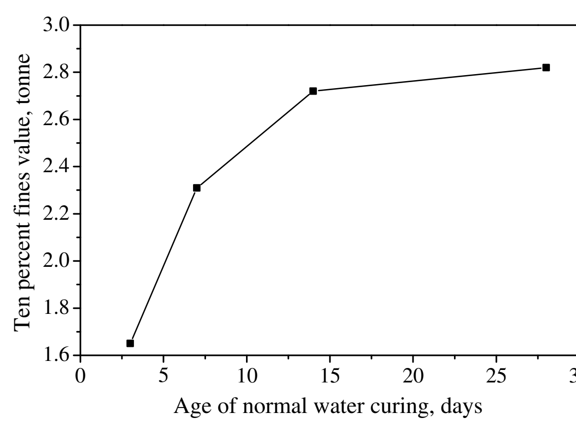



**Fig. 1a.** Variation of 10% fines value with age of normal water curing.

in water absorption (21–14%) (Fig. 1b). This is attributed to the significant reduction in open and total porosity of NWC aggregate (i.e., 52% total porosity and 43.5% open porosity at 3 days reduced to 46% and 38.5% at 28 days, respectively) as shown in Fig. 1b. These enhancements in properties were due to the formation of

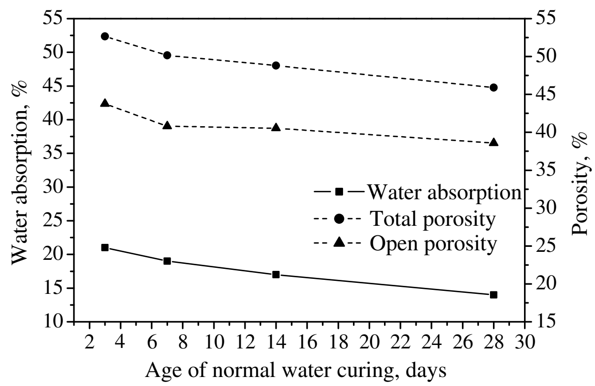



**Fig. 1b.** Variation of water absorption and porosity with age of normal water curing.

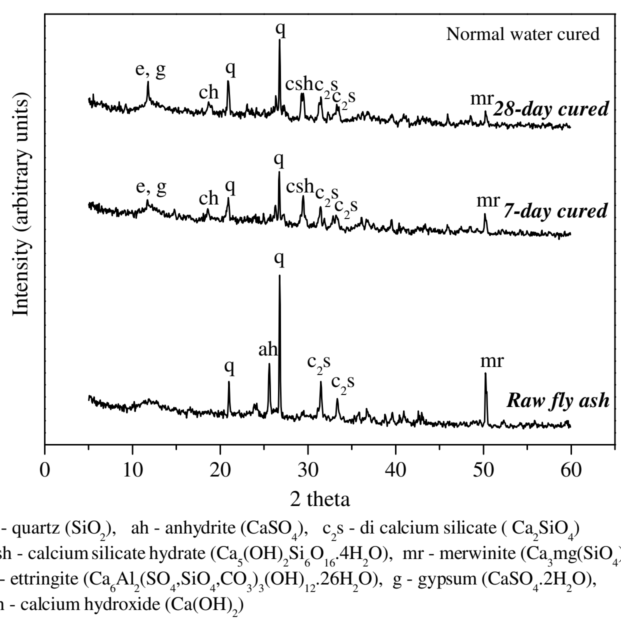



**Fig. 2.** XRD pattern for raw Class-C fly ash and aggregate cured at different age.

hydration products (calcium silicate hydrate, calcium hydroxide and ettringite) from the enhanced hydration of fly ash as seen in the XRD pattern (Fig. 2) and SEM (Figs. 3a and b).

When the duration of normal water curing was increased to 7 days, phases like anhydrite and di-calcium silicate present in the raw fly ash were converted into the hydration products like ettringite, gypsum and calcium silicate hydrate (Fig. 2), resulting in refinement of pores. The presence of unhydrated fly ash particles along with the voids in between the fly ash particles for aggregate cured at 7 days (Fig. 3a) are responsible for higher water absorption and lower 10% fines values. As the duration of curing is further increased to 28 days, maximum 10% fines value of 2.8 tonne was obtained. This is attributed to the formation of strength giving hydration products such as calcium silicate hydrate and needle shaped ettringite as shown in scanning electron micrograph Fig. 3b.

*4.1.2. Autoclaving and steam curing*

An increase in duration of autoclaving from 5 h to 10 h increased the 10% fines value from 1.85 to 2.4 tonne (Fig. 4a) and reduced the water absorption from 21% to 18.5% (Fig. 4b). For a similar increase in the same period of steam curing (between 5 and 10 h), an increase in 10% fines value (from 1.63 tonne to 1.92 tonne) and reduction in water absorption (from 24% to 20.5%) were observed (Fig. 4b). This behaviour is attributed to the reduction in percentage of total and open porosity (Fig. 4c) as result of formation of hydration products like calcium silicate hydrate and zcalcium hydroxide from the enhanced lime-silica reaction [18] as seen in XRD pattern Figs. 5a and b. The presence of unhydrated fly ash particles and interconnected pores (Figs. 6a and c) are responsible for higher porosity and water absorption and lower 10% fines value for aggregates autoclaved and steam cured at 5 h.

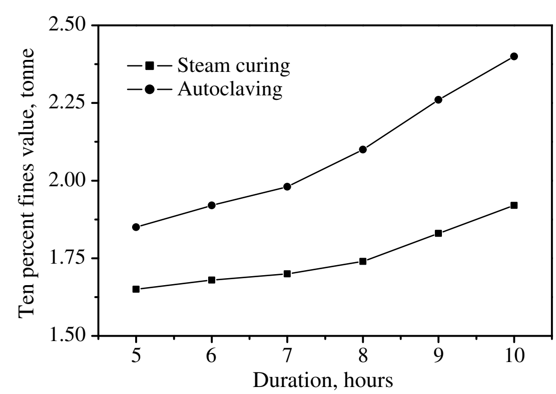
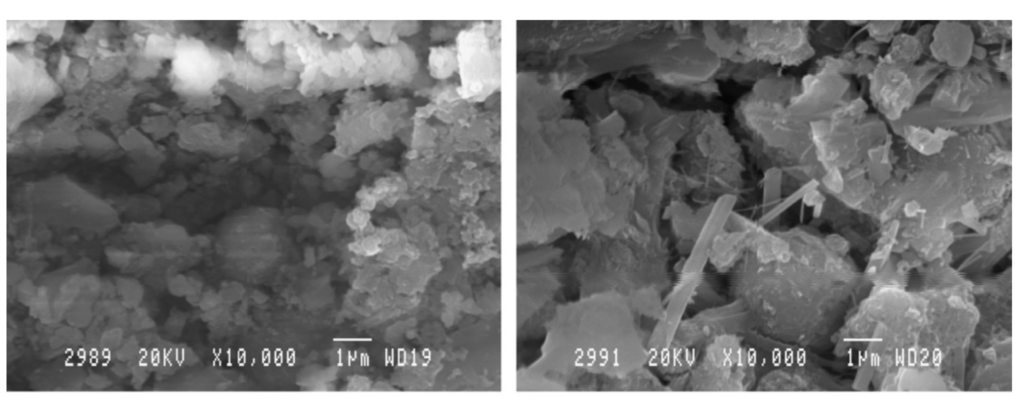



**Fig. 4a.** Variation of TPFV with duration of autoclaving and steam curing.




(a) 7 day-cured specimen (b) 28 day-cured specimen

**Fig. 3.** Micrographs of normal water-cured aggregate at different ages.

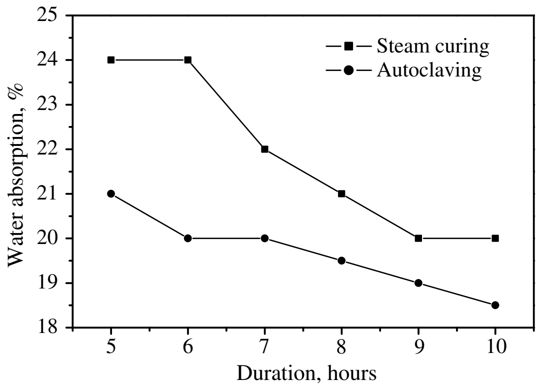



**Fig. 4b.** Variation of water absorption with duration of autoclaving and steam curing.

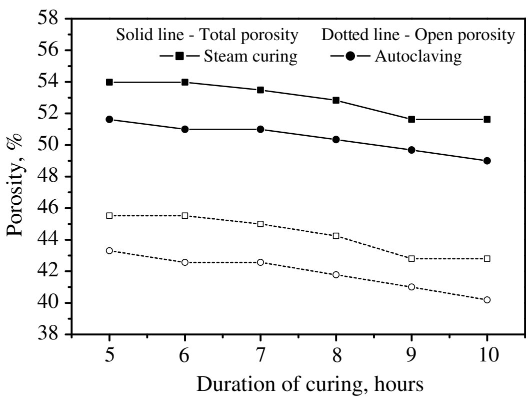



**Fig. 4c.** Variation of porosity with duration of autoclaving and steam curing.

Even though the XRD pattern exhibits the presence of strength giving hydration products (Fig. 5b), due to the formation of large hydration crystals (Figs. 6c and d) along with large interconnected pores, the 10% fines value of steam cured aggregate was lower than the autoclaved aggregate. As compared to the steam cured aggregate, autoclaved aggregate has 25% higher 10% fines value and 10% lower water absorption due to its dense microstructure formation (Fig. 6b).

It is interesting to note that even though the microstructure for autoclaved and steam cured fly ash aggregate showed the presence of calcium silicate hydrate, the change in shape of hydration products along with interconnected pores (Figs. 6a–d) can be attributed as the reason for possessing inferior properties as compared to normal water cured class-C fly ash aggregate.

In order to ascertain the variation in properties of fly ash aggregate after autoclaving and steam curing, the accelerated cured fly ash aggregate were further subjected to normal water curing up to 28 days. The properties were determined on the aggregate after the specified period of normal water curing and presented in Figs. 7a–c. An increase in duration of normal water curing of autoclaved and steam cured fly ash aggregates exhibit only marginal increase in 10% fines value (2.4 tonne to 2.55 tonne and 1.92 tonne to 2.12 tonne) (Fig. 7a) and reduction in water absorption (18.5–17.5% and 20–19%) (Fig. 7b). This is attributed to the marginal reduction in porosity of aggregate as shown in Fig. 7c.

*4.2. Comparison of different curing methods*

A comparison of the properties of aggregates subjected to different curing conditions is presented in Table 2. As compared to 28 day normal water cured aggregate, the properties of autoclaved and steam cured fly ash aggregate have relatively higher water absorption and lower 10% fines value. However, aggregates autoclaved for 10 h possessed properties closer to the normal water cured aggregate (i.e., 10% fines value approximately 86% of 28 days

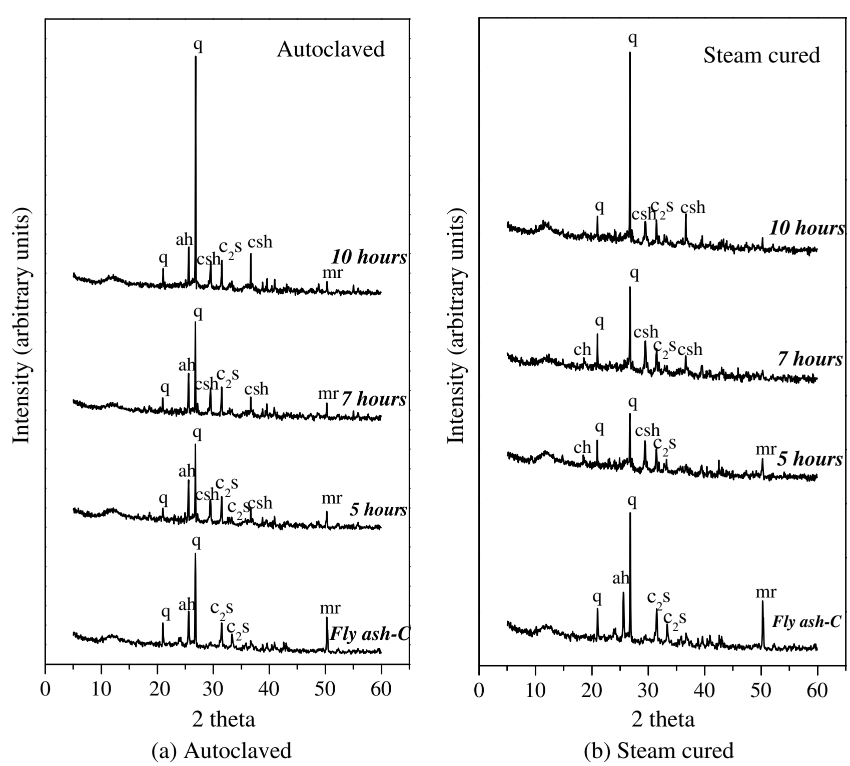



**Fig. 5.** XRD pattern for autoclaved and steam cured aggregate.

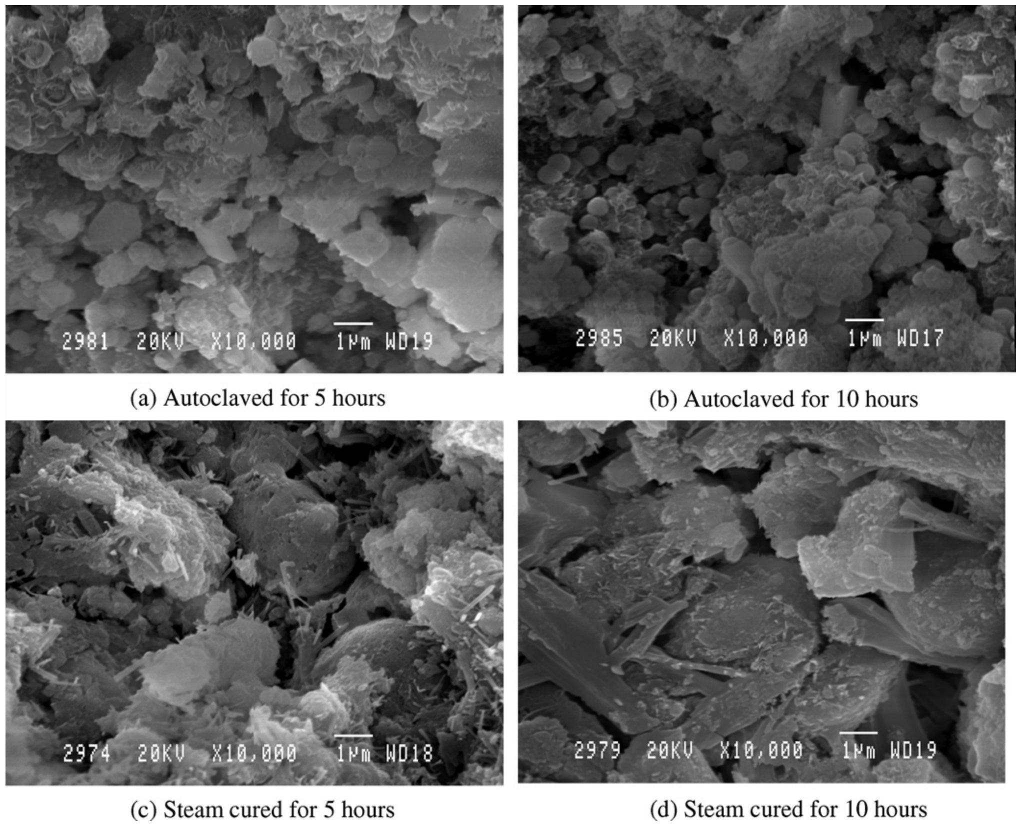



**Fig. 6.** Scanning electron micrograph for autoclaved and steam cured aggregate.

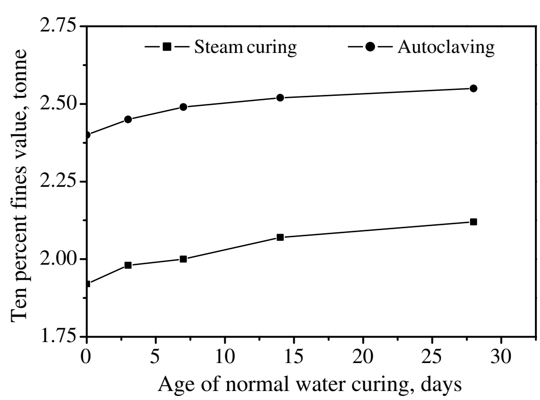



**Fig. 7a.** Variation in 10% fines value of accelerated cured aggregates with further normal water curing.

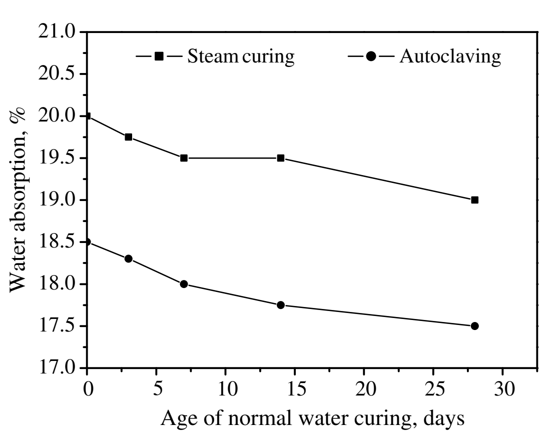



**Fig. 7b.** Variation in water absorption of accelerated cured aggregates with further normal water curing.

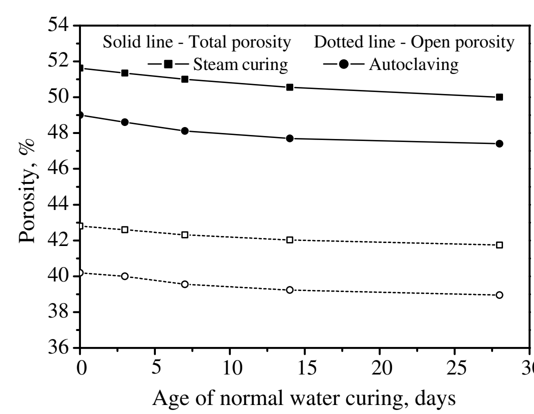



**Fig. 7c.** Variation in porosity of accelerated cured aggregates with further normal water curing.

**Table 2**
Properties of fly ash aggregates cured under various conditions

| Types of curing | Properties of aggregates subjected to different curing methods | | | | |
|---|---|---|---|---|---|
| | Water absorption (%) | 10% fines value (tonne) | Total porosity (%) | Open porosity (%) | Saturated surface dry specific gravity |
| 28 days NWC | 17.0 | 2.8 | 45.9 | 38.6 | 1.95 |
| Steam curing for 10 h | 20.0 | 1.9 | 51.6 | 42.8 | 1.87 |
| Steam cured aggregate after 28 days NWC | 19.0 | 2.1 | 50.0 | 41.7 | 1.89 |
| Autoclaving for 10 h | 18.5 | 2.4 | 49.0 | 40.2 | 1.91 |
| Autoclaved aggregate after 28 days NWC | 17.5 | 2.5 | 47.4 | 39.0 | 1.92 |

normal water cured aggregate). Saturated surface dry specific gravity of fly ash aggregate varies with the curing method, due to variation in the porosity of aggregate.

This study indicates that the type and duration of curing can be designed to suit the performance requirement of aggregates. If the space is not a constraint for storage of aggregate, normal water curing may be chosen as a curing method. Autoclaving may be adopted when high early strength is desirable.

## 5. Conclusions

The conclusions drawn from this study and summarized below are applicable to the properties of the materials used, methodology adopted and the range of parameters investigated:

1. An increase in duration of normal water curing significantly improved 10% fines value and reduced water absorption of class-C fly ash aggregate.
2. Autoclaving and steam curing resulted in relatively lower enhancement in the properties as compared to normal water cured aggregate. However, among the accelerated cured class-C fly ash aggregate, autoclaved aggregate possessed properties closer to the normal water cured aggregate due to the dense microstructure formation.
3. Continuation of normal water curing after initially subjecting the aggregates to accelerated curing exhibited only a marginal improvement in the properties.
4. The present investigation indicates that the type and duration of curing can be chosen based on the performance requirement of aggregate.

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