A Review on Ambient Cured Geopolymer Concrete- Sustainable Concrete for the Future

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Abstract. The cement industry is one of the major contributors to global warming due to the release of greenhouse gases. An alternative low emission binding agent is needed to reduce the environmental impact caused by cement production. Geopolymer binder is an ideal material to substitute cement binder. Geopolymer Concrete (GPC) is formed by the polymerisation of aluminates and silicates formed by the reaction of solid aluminosilicates with alkali activators. The source materials studied were waste by-products from industries. This paper reviews the strength and durability characteristics of Geopolymer Concrete under ambient curing conditions. The comparison and study of the morphology of different aluminosilicate source materials through X-ray diffraction (XRD) pattern and Scanning electron microscopy (SEM) analysis under different curing temperatures and the study on the chemical composition and its effect on alkali activators and aggregates helped to understand the nature and type of source materials and activators most desirable to develop GPC in ambient conditions. GPC formed in ambient curing conditions have considerable strength and durability characteristics and can be adopted as a sustainable replacement for conventional concrete.

1. Introduction

Concrete is the most widely used man-made substance. Ordinary Portland Cement (OPC) is the most commonly used primary binder in the conventional concrete. The production of OPC is an energy-intensive process and it releases huge amount of greenhouse gases to the atmosphere. For every tonne of cement produced, about one tonne of CO₂ is released into the atmosphere [1]. The annual global production of cement is about 4 billion tonnes [2]. The global demand for concrete is increasing rapidly and its production will increase to about 18 billion tonnes per annum by 2050 [3]. An alternative environment-friendly sustainable and low emission primary binding material needs to be widely adopted to replace OPC based concrete. For a developing country like India, there is a high demand for cement to meet the ongoing infrastructural activities. India is in the second position globally, in both production and consumption of cement with an annual production of 502 million tonnes per annum in 2018. Alternative materials rich in aluminates and silicates like Fly ash (FA), ground granulated blast furnace slag (GGBFS) etc. can be utilised to develop cement-like binders without compromising efficiency and quality of concrete. Geopolymer technology replaces OPC binder completely with an aluminosilicate binder material and about 80% of greenhouse gas emitted by the cement industries [4]. ‘ENVIS centre on fly ash’, estimated the production of fly ash in India to be around 600 million tons by 2030 from about 160 million tons in the year 2010. 70% of electricity demand in India is met by coal-based thermal
power plants [4]. India and China are the major producers but have less than 50% utilisation rate [5]. According to Central Electricity Authority's report 2018, the highest utilisation of fly ash was in cement sector with 25.6 per cent of the total fly ash used but the concrete had only 0.66 percent of the total fly ash used. Ground granulated slag is the waste generated during the steel manufacturing process and about 17.236 million tons were produced for the year 2017-18 in India [6]. The government of India has estimated steel production to hit 128.6 million tonnes by 2021. The Blast furnace slag specific waste generation is 320 kg/ton of steel. 40% of this is utilised in cement industry [7]. An average of 30000 tons per year of silica fume is produced by Steel Authority of India [8]. An average of 40 kg of Rice husk ash (RHA) can be produced for every tonne of rice [9]. According to the report by the United States Department of Agriculture (USDA), 114 MT of rice production in India was forecasted during the year 2019-20. There is an adequate generation of aluminosilicate industrial by-products in India. The utilisation to generation ratio of these materials is very less. Adopting GPC as a viable alternative to OPC concrete enables to minimise the impact of cement production-related environmental problems and creates a sustainable utilisation of these harmful industrial by-products.

2. Geopolymer concrete
During the history of the concrete, there was first-generation lime concrete, second-generation ordinary Portland and special cement-based concrete. Geopolymer concrete (GPC) can be considered as a third-generation type of concrete [10]. Geopolymer technology was first developed by Joseph Davidovits in 1972 by polycondensation reaction of kaolinite with NaOH [11]. In Geopolymer concrete, OPC binder is totally replaced by a polymerised aluminosilicate binder formed by the reaction of an alkaline solution with aluminosilicate rich source materials. Geopolymer compounds are crystalline in hydrothermal setting conditions and amorphous in ambient setting conditions [12]. The geopolymers are composed of source materials rich in silicates and aluminates which when dissolved in an alkaline activating solution, polymerises and forms the binder [13].

In Geopolymer concrete (GPC), these binders (geopolymer) binds the aggregates. The behaviour of geopolymer concrete and conventional Portland concrete are different due to the variation in the microstructural behaviour of source materials and the process of formation of binders [14]. The studies on geopolymer concrete were mostly based heat cured fly ash blended types. Elevated temperatures were required for fast geopolymerisation reaction [15]. Heat-cured geopolymer concrete possesses high compressive strength, low drying shrinkage and creep, and good resistance to sulphate and acid environments. The heat curing mechanism is difficult and costly. Thus, there occurred a need to research and develop ambient cured geopolymer concrete. In this review study, an attempt has been done to study the properties of different aluminosilicate sources materials and its effect on the properties of GPC under ambient curing and are detailed in the subsequent sections.

3. Geopolymer source materials
The geopolymer source materials are either natural minerals like kaolinite, clay or industrial by-products like fly ash, GGBFS, Silica fume, Rice husk Ash, etc. The reaction between the source materials and alkaline activators and their interaction affects the properties and behaviour of Geopolymer concrete [16]. The main factors affecting the properties of geopolymer are the chemical composition and fineness of various source materials. The generally adopted tests done to examine the microstructure and chemical composition are X-ray diffraction (XRD) pattern and Scanning electron microscopy (SEM) analysis. The chemical composition of various source materials and its specific surface area is given in table 1 [17]. A description of source and microstructure properties of different aluminosilicates sources and their behaviour are discussed below.

3.1. Fly ash
Fly ash is the most commonly used binder because of its pozzolanic character, less cost, availability, richness in Silicon and Aluminium oxides, low water demand and high workability. Class F fly ash is obtained by burning of pulverised coal extracted from anthracite and bituminous coal reserves [25]. Fly ash (Class C) is obtained on the burning of lignite and has Calcium more than 10% [26]. Class F fly ash showed better performance than class C fly ash when subjected to heat curing [27]. The SEM and XRD of various source materials are given in figure 1 and figure 2. From figure 1 (a) it can be seen than Class
F fly ash consisted mostly of hollow spherical particles (cenospheres) of size ranging from 1 to 300µm [28]. The SEM Analysis shows solid spheres with irregular shape particles [29]. The spherical shape imparts ball-bearing effect [30]. The spherical shape of fly ash particles will impart easy blending and free-flowing nature to the binder. From figure 2 (a), the XRD pattern of Class F Fly ash had sharp peaks of quartz, mullite, and mellite which indicate crystalline phase and broad humps showing amorphous phase. Class F Fly ash had a higher mullite content. This indicates less abundance of amorphous phase as there is an inverse relationship between mullite and amorphous phase. [31]. The presence of the crystalline phase in Class F Fly ash resulted to use heat curing as an effective method to accelerate geopolymerisation reaction. Figure 1 (b) shows the SEM analysis of Class C Fly ash, which consists of sphere, agglomerate and angular shape particles and figure 2 (b) shows XRD pattern of class C fly ash with smooth and less steep humps. This indicates the presence of both crystalline and amorphous phase [32]. The Fly ash-based GPC had considerable workability compared to GPC formed with other aluminosilicate-based materials.

3.2. Ground Granulated Blast furnace slag (GGBFS)
GGBFS is obtained during the manufacture of pig iron for the steel industry [33]. GGBFS is hydraulic in nature due to the presence of oxides of Calcium. When alkali-activated it resulted in the C-S-H microstructure formation compared to an Al-Si network typically formed during geopolymerisation [20]. But a faster rate of strength development and ambient temperature curing are the advantages of using GGBFS as aluminosilicate binder. Figure 1 (c) shows SEM images of GGBFS indicating very fine glassy and granular shaped particles and these particles were finer than 30µm [34] [36]. The granular shaped particles of GGBFS resulted in the formation of geopolymer binder with less workability and making the slag-based GPC difficult to work with. GGBFS based GPC had lower setting time for geopolymer binders. As in figure 2 (c), smooth peaks were observed in XRD patterns of GGBFS [35]. The smooth hump indicates the amorphous phase. C-S-H formation helps to accelerate geopolymerisation reaction at ambient temperature. Due to these properties, Fly Ash based GPC partially replaced with GGBFS had better strength compared to Fly Ash based GPC at ambient temperature.

3.3. Rice husk ash
Rice husk ash (RHA) is obtained by burning rice husk [37]. It contains SiO$_2$ greater than 90%. Since it is obtained from the agricultural industry, it is important to maintain efficient burning at a controlled temperature of 500 to 600°C and is then ground in jar mill to obtain required fineness. From figure 1 (d), the average size of RHA was found nearly three times smaller than fly ash particles and are irregular in shape [20]. Due to which GPC having RHA is found to have less setting time. RHA gets polymerised faster due to less size compared to Fly ash. From figure 2 (d), the majority of composition was found to be in the amorphous phase due to smooth humps [20]. The amorphous phase of silica in RHA reacts with an alkaline solution to produce binders at ambient temperature conditions.

3.4. Silica fume
Silica fume (SF) is obtained from the ferrosilicon industry [38]. Silica fume contains about 85-97% SiO$_2$. It is a highly reactive material suitable for geopolymerisation. Figure 1 (e) shows SEM analysis of silica fume and particle size were extremely small particles sizes [31]. The reactivity was higher due to a larger surface area to mass ratio. GPC with SF has a lower setting time. The SEM analysis showed dense microstructure. The dense microstructure improved the durability properties in Fly ash based GPC partially replaced by SF. Figure 2 (e) shows XRD pattern of SF as amorphous structured due to its board smooth humps and presence of a high amount of amorphous silicon dioxide which provide a highly reactive pozzolanic material [20][39]. SF reacts with an alkaline solution to produce binders at ambient curing conditions.

3.5. Alccofine
Alccofine is a low calcium micro-fine silicate slag. It is a fine powder obtained by powdering the dried granular material produced by quenching molten iron slag in a blast furnace in water or steam [40]. Figure 1 (f) of alccofine shows SEM image, which reveals ultra-fine and angular particles for alccofine...
Fly ash-based GPC with partial replacement of Alccofine had an early setting and dense microstructure was observed. The dense microstructure improved the durability properties in Fly ash-based GPC partially replaced by Alccofine. Figure 2 (f) shows XRD pattern of Alccofine as amorphous since it shows the Calcite phase with broad smooth humps [41]. Alccofine reacts with an alkaline solution to produce binders at ambient curing conditions.

3.6. Red mud
Red Mud is a silt muddy alkaline solid industrial by-product obtained during the extraction of alumina from bauxite ores by the Bayer process. The Presence of Alumina and high alkalinity makes them useful in geopolymer. Fe$_2$O$_3$, SiO$_2$ and Al$_2$O$_3$ constitute 75% of total composition [42]. Figure 1 (g) shows the SEM image of Red Mud. It contains superfine amorphous aragonite and a few calcite micrites and the particle sizes of the grinded Red Mud are less than 45μ [43]. The fineness imparts quick setting properties to the binder. Figure 2 (g) shows the XRD pattern of Red Mud. The presence of sharp peaks indicated the crystalline phases and smooth humps indicated amorphous phase [44].

Table 1. The chemical composition (% mass) of Class F Fly ash (FFA) [11], Class C Fly ash (CFA), GGBFS [19], Rice Husk Ash (RHA) [20], Nano silica (NS) [21], Alccofine [22], Silica fume (SF) [23] and Red Mud (RM) [24]

| Composition mass | FFA | CFA | GGBFS | RHA | NS | Alccofine | SF | RM |
|------------------|-----|-----|--------|-----|----|-----------|----|----|
| SiO$_2$          | 59.7| 38.4| 34.06  | 91  | 99.8| 35.3      | 92 | 8.88|
| Al$_2$O$_3$      | 30.2| 18.7| 20     | 0.35| -   | 21.4      | 0.46| 25.4|
| CaO              | 0.7 | 24.6| 32.6   | -   | -   | 32.2      | 0.29| 0.92|
| SO$_3$           | 0.02| 1.4 | 0.9    | 1.21| -   | 0.13      | 0.19| 0.22|
| Fe$_2$O$_3$      | 2.8 | 5.1 | 0.8    | 0.41| -   | 1.2       | 1.60| 40.03|
| MgO              | 0.8 | 5.1 | 7.89   | 0.81| -   | 8.2       | 0.28| -   |
| Na$_2$O          | -   | -   | -      | -   | -   | -         | -   | 8.75|
| LOI              | 0.8 | 0.3 | 0.9    | <1  | 0.2 | 1         | 8.7 |
| Surface area(m$^2$/kg) | 1280| 900 | 375    | 20000| 90000| 12000    | 8900| 655 |

(a) Class F Fly ash [29] (b) Class C Fly ash [32] (c) GGBFS [34] (d) Rice Husk Ash [20] (e) Silica Fume [31] (f) Alccofine [40] (g) Red Mud [43]

Figure 1. SEM analysis of aluminosilicate sources.
Previous Studies on Geopolymer concrete cured at ambient temperature

To enhance the strength of GPC in ambient curing conditions, the geopolymerisation process needs to be accelerated at the early stages. For this, amorphous structured aluminosilicate source materials need to be effectively used. A Literature survey was conducted to understand the factors affecting the strength, workability and durability of ambient cured geopolymer concrete. These factors are summarised on the following sections.

4. Effect of Aluminosilicate Sources

Geopolymer binder with fly ash as the source material required higher temperature for initiating the geopolymerisation reaction. The heat-cured GPC had high early-age strength gains and has better durability properties compared to conventional concrete. [46][47][48]. Partial replacement of fly ash with amorphous structured aluminosilicates or those binders having calcite enabled to develop GPC at ambient curing. Majority of earlier studies were done on heat-cured low calcium fly ash. This limited the applications to precast members and as a solution, further research was carried in developing cost effective ambient cured GPC using two or more aluminosilicate source materials. Class C fly ash showed better strength and performance at ambient cured conditions [35]. Class F fly ash geopolymer concrete (GPC) partially replaced by GGBFS, OPC or Calcium Hydroxide under ambient curing had setting time lower to a value similar to that of OPC based concrete. It had better early strength compared to fly ash GPC cured at ambient conditions but late age strength at 28 and 90 days showed not much significant difference [49]. This behaviour is due to the amorphous phase in GGBFS activated by the Calcium Silicate Hydrates (C-S-H) when OPC reacts with water during geopolymerisation at ambient temperature. The addition of GGBFS in fly ash GPC increased the strength and decreased the
workability of GPC [19][33][35][36][50]. The properties of ambient cured GPC were enhanced by the addition of GGBFS but the rate at which strength improved slowed down after 28 days [51]. GGBFS blended ambient cured GPC has strength comparable to that of OPC at the ambient cured condition. The setting time reduced while replacing more percent of fly ash with GGBFS [49]. This is mainly due to the variation in particle size and shape at the microstructural level. The spherical Fly ash particles were replaced by the angular and irregular shaped GGBFS particle, resulting in reduced workability. The addition of OPC accelerated the geopolymerisation reaction as well as affected the workability and setting time [49][52]. The strength of ambient cured fly ash GPC was also improved by the small percentage replacement by OPC. The micro cracks reduced as the hydration utilised expelled water in the geopolymerisation process [53]. Fly ash-based GPC cured at ambient temperature can also be blended with ordinary Portland Cement (OPC) and micro-silica (MS) and GPC with sufficient strength can be attained at ambient curing conditions [54]. Early strength and durability were achieved in ambient cured geopolymer concrete blended with red mud, GGBFS and fly ash [42]. Red Mud contains alkalies that help to reduce the molarity of NaOH activator solution and compressive strength comparable to OPC was obtained by partial replacement [42]. Fly ash geopolymer concrete blended with alccofine improved compressive strength and durability at ambient curing conditions [22]. The fly ash nano-silica modified GPC cured at ambient temperature showed higher compressive strength and enhanced durability properties compared to heat-cured GPC and OPC [55]. GGBS based fly ash had dense microstructure. It performed well under sulphate attack [56]. Aluminosilicate source materials having fineness in micro units formed very dense structured GPC. The compact binder formed by alkali activation improved durability properties of GPC. Using aluminosilicate source materials with amorphous structured microstructure and accelerating the reaction by adding calcite sources or very fine silicate sources, GPC with strength comparable to conventional OPC concrete were developed.

Another ongoing research was to modify the microstructure of fly ash before mixing during geopolymerisation. The compressive strength of geopolymer concrete was enhanced by the mechanical activation of fly ash material [30]. Mechanical activation results in increased rate of geopolymerisation by reducing particle size and increasing the amorphous phase [57]. The heat activation of fly ash was another method adopted and used for preparing ambient cured GPC. It improved the early strength properties of GPC. Here early activation of fly ash helped in early polymerisation and less amount of crystalline compound. About 9% less energy was required in heat-activated fly ash material blended geopolymer concrete than in heat-cured GPC for heat activation [55].

4.2. Effect of alkaline activators

The alkaline activators used to activate aluminosilicates are Sodium or Potassium based hydroxides or silicates. Sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium silicate (Na₂SiO₃) and potassium silicate (K₂SiO₃), and NaOH are more commonly used as it has the capacity to release more silicate and aluminate monomers [58]. The Sodium Silicate to Sodium Hydroxide ratio (SS/SH) and molarity of Sodium Hydroxide affects the properties of geopolymer concrete cured at ambient temperature. The slump value and flow value are reduced when SS/SH ratio is increased [49] and lower value of SS/SH ratio showed better later age strength in fly ash GPC blended with GGBFS. Increasing the molarity of NaOH from 4.0 to 14.5 increased the strength of geopolymer paste but further increase causes early precipitation of aluminosilicates, which hindered the development of strength [51]. For obtaining higher compressive strength, the molarity of NaOH must be very high [59]-[61]. Less concentration of NaOH is required when geopolymerisation was done on elevated temperatures [62]. The compressive strength was increased and the setting time was decreased when molarity of NaOH was increased subject to a subsequent increase in slag content [63]. Higher molarity for NaOH is required for faster geopolymerisation reaction for Ambient cured GPC compared to heat-cured GPC.

The current research on activators is focussed on developing GPC by using single activator. The behaviour of Geopolymer concrete using single activator NaOH showed that replacing binder with 100% GGBS gained sufficient strength in 28 days. Fly ash used as a sole aluminosilicate source in ambient cured geopolymer concrete resulted in very low strength [64].
4.3. Effect of aggregates
The strength of geopolymer binders is less affected by the interaction of aggregates and more on the chemical behaviour of the geopolymer binder. Compared to OPC, less treatment of C&DW is required for geopolymer based concrete. Thus, geopolymer technology was effective in developing concrete using recycled industrial construction waste [65]. The strength of GPC improved with the increase in the mean size of the coarse aggregates. There was a decrease when the mean size was increased after the mean size of 18.75 mm [66]. The aggregate–paste interface was bigger for large size aggregates. This resulted in wider cracks, which interacted with cracks in the binder as well as in other interfacial zones resulting in loss of the strength. When internal bleeding occurs, water gets trapped in between the bigger aggregates and when this water evaporates, voids were formed which reduced the strength of the concrete. The maximum value for compressive strength was obtained for aggregates of size range 12.5–25 mm [66]. Thus, the strength of fly ash-based geopolymer concrete increased with an increase in total aggregate size and proportioning of total aggregate content and ratio of fine aggregate to total aggregate affected the behaviour of GPC [67].

5. Conclusions
The paper reviewed the strength and durability studies on ambient cured geopolymer concrete formed from different aluminosilicate sources. Ambient cured GPC can be used as an alternative for the conventional OPC concrete as it had better durability and comparable strength parameters. The early strength is very less when compared heat-cured Fly ash GPC. India has an abundant quantity of aluminosilicate materials as industrial by-products. The source materials used in geopolymer binders must be pozzolanic in nature. The microstructure and chemical composition of aluminosilicate source materials played a major role in the determination the strength and durability behaviour of GPC. It also helped to understand the nature in which source materials behaves during geopolymerisation reaction. The oxides of silica, alumina and calcium were the critical elements affecting the behaviour of geopolymer binder. The crystalline phase in aluminosilicate source materials required a higher temperature to initiate geopolymerisation reaction compared to the amorphous phase in which geopolymerisation occurs at ambient temperature. The particle size and presence of amorphous phases determines the rate of geopolymerisation reaction. GGBFS, RHA and Alccofine blended with Fly ash GPC had sufficient strength at ambient curing condition. The particle shapes and size study were important to compare the reactivity and workability of the binders. The workability was reduced in angular shaped aluminosilicate source materials and often very fine sizes attributed to the early setting of geopolymer binder. This widened the research area on the effect of superplasticiser on the geopolymer binders. The addition of the calcium-based aluminosilicates enhanced the properties of the fly ash-based geopolymers cured at ambient temperature. The current research on ambient curing is mainly focused on low-cost blended geopolymer concrete using tertiary binders. Another area of research is on developing activated geopolymer source materials. Ambient GPC achieved by mechanical activation of aluminosilicate source materials or by developing ambient cured geopolymer concrete using aluminosilicate source materials having calcite, appropriate molarity for alkali activator and alkali to binder ratio is found to be the current area of interest. By avoiding heat curing and reducing the concentration of alkali activator, the cost of GPC can be reduced considerably. The current researches need to be focused on developing low-cost alkali activators.

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