THINK INDIA JOURNAL

ISSN: 0971-1260
Vol-22-Issue-10-November-2019

Microstructure Properties Of Geopolymer Concrete- An Overview

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Abstract— Concrete is the construction material of the highest exigency which, in turn, created a huge demand for ordinary Portland cement (OPC) as an essential binder. Lamentably, its current production process is not only highly energy-intensive and happens to be at elevated temperature for Calcination of Limestones to takes place but it also liberates embodied anthropogenic Carbon Dioxide (CO_2), a primary Green House Gas (GHG), answerable to global warming and pollution of earth's environment. Not only that, it is user-hostile and comparatively costly too. Therefore, construction and infrastructure industries are looking at concrete researchers to invent alternative, new-fangled, user and environmentally-compassionate durable, sustainable and desirably cost-effective edifice materials and binders. Nowadays, Geopolymer technology has emerged out as a brilliant possible substitute to conventional OPC-system whereby Geopolymer composites and binders are being synthesized by activating alkali solution with Silica and Alumina rich pozzolanic material such as Fly ash, through the process of Geopolymerization analogous to geo-synthesis of natural rocks. The aim of the current review is to investigate the micro-structure performance of geopolymer concrete with 100% substitution of cement with waste materials, i.e., in the absolute absence of cement.

Index Terms—*Geopolymer (GP), Geopolymer Concrete (GPC), Geopolymer Technology,* Global warming, *Green House Gas (GHG), Ordinary Portland cement (OPC).*

I. INTRODUCTION

The alarm bells have already initiated to ring in order to "save earth's environment". For that reason, industries are compelled to reduce the evils of polluting agents from all kind of productions including Ordinary Portland cement (OPC) since it emits CO_2- Carbon dioxide from its present production process. On the one hand, mushrooming world population demands vigorously for new brand residences, colonies and essential infrastructure to live in and on the other hand, construction and infrastructure industries necessitate titanic amount of concrete to construct them. This is a catch 22 situation because traditional concrete can't be manufactured without OPC, and in turn, OPC can't be produced without emission of anthropogenic CO_2 accountable for earth heating eventually contributing to global warming dilemma. Not only have that, degradation of restricted natural resources through haphazard mining of natural rocks like Limestones, etc.; and natural minerals such as Coals, etc.; along with natural aggregates viz., River sand, Gravel, Basalt, other crushed natural stones, etc. also imbalances the earth's environments.

What's more, it is a universal fact that more urbanization leads to exigency of more OPC and analogously more industrial development pilots to more generation of wastes as by-products or remains. Diverse industries across the planet are answerable for a variety of profuse wastes creating not merely their disposal concerns but also crafting gigantic quandaries like land spaces filling together with contamination of air, waters, soils, lot of health hazards, etc. Global environmentalists are worried greatly on these crises and can predict the cloud on the horizon. In these signaling situations, researchers, concrete scientists, engineers can't bury their head in the sand by declining the unpleasing reality the living beings facing today. Collectively, all these states of affairs have twisted the arms of the international researchers to search for novel, innovative alternative eco-benevolent composites for construction industries in order to get rid of all the referred predicaments. Quite recently, researchers have a ray of hope in form of an innovative inorganic Geopolymer technology through which novelties of construction composites can be produced possessing excellent required attributes. This novel technology was coined by a French Prof (Dr.) Davidovits, in 1978, which are competent enough to produce Geopolymers (GP) through an exothermic, complex, course of geopolymerization nine times low temperature and six times less energy requisites as compared to OPC system since the reaction kinetics can take place even at room temperature and at atmospheric pressure only [1]. On top of that, GP can be manufactured by utilizing copious diverse wastes from dissimilar sectors, lending a hand for their systematic and responsible disposal otherwise playing the role of pollution agent.

Additionally, GPs exhibit brilliant properties of resistances to heat, fire, freeze-thaw, acids, chemicals, strength, durability, sustainability, micro-structures, etc. besides being pleasingly affordable, user and eco-compassionate and economical too. This is the core reason why researchers of universe are attracted towards this ground-breaking technology which contributes so many construction composites like concrete, mortar, paste and binders devoid of cement amalgamating diverse wastes generating from a variety of fields. However, GPCs seems to be durable with acceptable strength, but the review of its other properties like micro-structures is also a significant parameter to study. Consequently, the current review manuscript is flashing lights on microstructural characteristics of Geopolymer concretes.

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II. SCANNING ELECTRON MICROSCOPY (SEM)

A lot of researchers have accounted on the topic of microstructural properties of GPCs especially on fly ash (FA)-based GPC with variables enclosing molarity, supplementary binders, ratio of alkali activator, and temperature as well as period for curing [2-4]. Pavithra et al. [3] studied GPCs based on fly ash and have suggested a technique to attain optimum mix-design. Little parameters viz., molarity [M], curing age and temperature for curing, ratio of Sodium silicate/sodium hydroxide (SS/SH), were maintained as constant variables previous to making a selection for the Alkali Activator Solution (AAS) in the investigations. The referred study has suggested that quantity of water utilized was found relying upon the highest size of aggregate, as it can be employed to govern the frontier of maximum amount of water. The computation on quantity of binder, amount of water, AAS, finer and coarser ingredients was performed based on the priority of mixture whether a ratio of AAS to binder or strength is to be centered on. A constant slimming down with regard to precipitation found as the AAS to binder ratio accelerates. When the referred ratio enhances, the strength of GPC mitigates owing to the growing quantity of water existing in GP-mix. This happening can be regarded as similar to OPC-concretes with elevated ratio of water/cement. The water-molecules have blocked the contact region for reaction, which in turn influenced the course of geopolymerization among the activator and the binder resulting in inferior compressive strength of GPC [4]. The non-reacted particles of FA were found in lowest quantity by keeping ratios of alkali activator to binder appropriately as 0.4 and 0.5, as displayed in Fig.1(a) and (b). What's more, meager number of voids is also portrayed through Fig. - 1(c) to (e). Also, the dense matrix is assigned to the fineness of FA, which is lending a hand to reduce micro-cracks development. Over and above that, in accordance with the study of Xie and Ozbakkaloglu [4] and Assi et al.[5], the strength of GPs was encountered as quite enhanced on account of the aptitude of fly ash to plug micro-cracks [5]. Abdulkareem and Ramli have concentrated on the influence of dissimilar ratios of mass of SS/SH of FA based GP were investigated micro-structurally [6]. The reacted FA was monitored with respect to GP possessing ratios of alkaline activator solution mass as 0.5 and 1.0. Although analogous micro-structure was encountered for both 0.5 and 1.0 ratio of mass, the compressive strength attained was recorded as 65 MPa and 35 MPa for mass ratio of 1.0 and 0.5 respectively. The preceding study suggested that elevated silicate concentration might have contributed for superior development with regard to strength owing to more quantity of accessible silicates for geopolymerization process [7]. Nevertheless, on comparing, elevated mass ratio of Na_2SiO_3 has resulted in non-reacted particles of FA. The application of mass ratios for activator solution as 2.0 and 3.0 has exhibited higher content of non-reacted and partly reacted micro-spheres of FA. The excessive quantity of AAS into the mixture has resulted in a hindrance to geopolymerization via confining the interaction among the AAS and binder [8]. As well, specimens of GP containing mass ratio of 3.0 for SS/SH has demonstrated the feeblest compressive strength figuring to 32 MPa. A majority of researchers found from past studies were found to employ the mass ratio limiting to 2.5 for SS/SH [4,9,10,11]. However, some studies have achieved parallel attributes for GP owning elevated mass ratio of alkali activator solution, predicting that the temperature for curing might be responsible for the dense structure [12]. The temperature of curing at 70° C or more has a propensity to encompass arrangement with loose-fitting because of evaporation of free H_2O that is answerable to incessant micro-cracks formation inside the matrix [13-15]. Chindaprasirt et al. [15], have revealed that enough water content is obligatory for geopolymerization kinetics to take place with a view to having higher growth of strength. Okoye et al. [9] have tested the microstructural properties of FA plus Silica Fumes (SF) based GP. Contrarily, GPC specimen enclosing FA was monitored with fractured surface. The voids monitoring unearthed the looser particle arrangement. While the even surface was recorded for 20 percent substitution of FA plus SF in FA-based GPC. A noteworthy quantity of either non-reacted or partly reacted FA particles were monitored in the case of pure GP. Nevertheless, number of non-reacted particles found mitigated. Other than that, micro-cracks have been noticed using dry mixture course were inferior to the pure GP and wet mixture. A pure GP has put on show the least quantity of amorphous phases that is in accordance with the number of non-reacted particles noticed. The supplement of nano-silica with nano-composite mixtures of GP has augmented amorphous content. The dry mixture of nano-composites is thought to represent superior owing to the capabilities to play a role as filler in matrix of GP. Accordingly, elevated compressive strength is encountered and wholly reacted particles have been monitored in dry mixture specimens than those wet mixtures. Interfacial Transition Zone (ITZ) among aggregate and matrix of GP. In harmony with Embong et al. [16], the improvement with regard to dissolution of particles of FA and poly-condensation of Alumino-silicate compound contributes bonding among matrix and ingredients. The formulation of alumino-silicate is filling up ITZ as illustrated. The sample's compressive strength was improved to a large extent. The existence of fiber straw in the matrix of GP is supposed to be capable of inferior dissolution of particles of FA. The non-reacted particles of FA were distributed in the matrix because the water-absorbing fiber straw has wrapped the surface. Contrasting to Pavithra et al. [3], the micro-structure of FA and bottom ash in GP was investigated by Xie and Ozbakkaloglu [4]. When mass ratio of FA to bottom ash (BA) escalates, the strength also gets increased. No doubt, the coverage of bigger surface area of BA and some associated foreign objects have not been reacted entirely in the GP resulting in mitigation of the strength of GP. A comparison that a smaller quantity of semi-spheres was found existing besides some objects possessing rectangular shape. Even as, composition testing has unveiled the existence of a chemical element Radium (Ra), which is the heaviest radioactive alkaline - earth metals representing group (II) of periodic table in the form of a foreign object associated with BA. On the other hand, there was an absence of foreign objects as well as particles of irregular shape in FA. Putting aside the association of foreign objects with BA, some of BAs had not reacted completely during geopolymerization contributing to inferior compressive strength in comparison with pure FA based GP. In harmony with the preceding studies, residues of non-reacted FA were noticed that merely smaller traces of sodium (Na) element are visible on the minerals which are not involved in reaction kinetics veiling the non-reacted particles of FA. A trivial

ISSN: 0971-1260
Vol-22-Issue-10-November-2019

Calcium (Ca) element content can be spotted because of lower amount of Ca associated with FA made available from "Gaston". The crystalline phases were disclosed, and new-fangled precipitant was developed subsequent to the Gaston FA dissolution employing alkali activator solution as represented by the image of SEM.

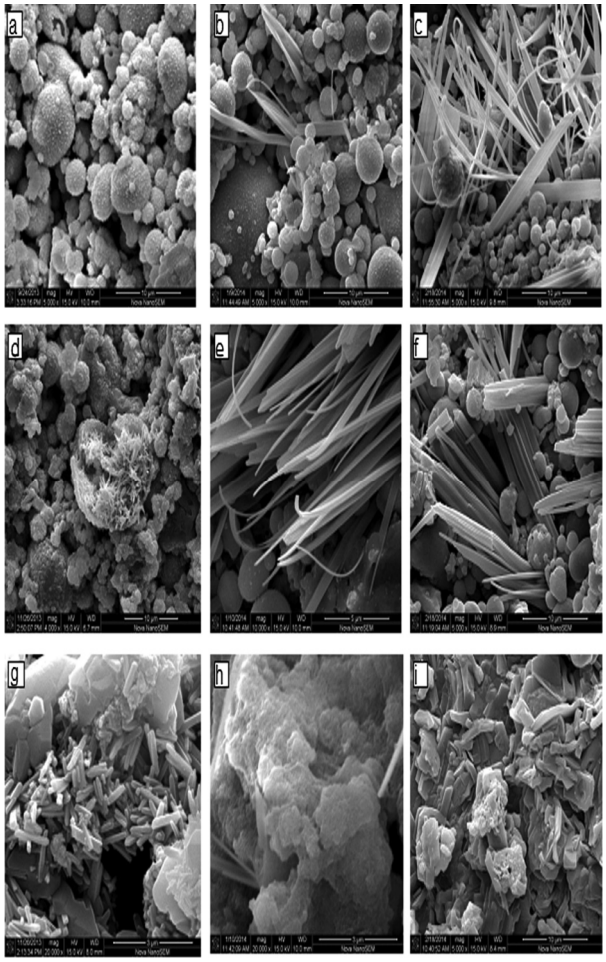

Fig.1 SEM image of fly ash-based geopolymer for different mixes [17]

III. X-RAY DIFFRACTION (XRD)

Abdulkareem et al. [18], have monitored a broad hump in the range of 20 to 35 signaling the presence of amorphous GP-products. The presence of mullite and quartz has reflected the characteristic FA chemistry as found in earlier studies. Previous studies show that greater than half of the FA was found enclosing Iron (Fe) and Ca in its composition. Accordingly, it helped to explain crystalline phases found accompanying GP- paste pattern. A comparison of cement-based controlled (CC) specimen with GPC sans supplementing nano-silica (12G C0H) exhibits a broad hump around 25 to 35 in the case of a sample of GPC enclosing nano-silica (12GC6). Quartz, mullite and hematite have displayed elevated intensity in 12GC6 owing to presence of supplementary nano-silica in matrix. A little extra peaks are exposed in nano-silica containing GPC (12GC6) indicating the formulation of the new-fangled phases of alite (Ca_3SiO_5) ; albite ($NaAlSi_3O_8$), kaolinite ($Al_2Si_2O_5$ $(OH)_4$), mullite ($3Al_2O_3{\cdot}2SiO_2$) ; Quartz (SiO_2), and $Ca(OH)_2$ crystallized compound in comparison with others (12GC0H and CC-concretes). The broad diffraction hump was spotted around 2h = 25 – 35 that has determined the presence of crystallized phases in 12GC6 GP- matrices. The crystallized phases were revealed following the supplement of alkali activator solution. The XRD analysis is found supporting the SEM image as illustrated in Fig 2 [19]. Zeolite was formulated subsequent to the dissolution whereas quartz, mullite, and hematite found present in all FAs. Tho-In et al. [20] have accounted the analysis of XRD on 7^{th} day age of FA based geopolymeric paste with ground container glass (CP). FA based geopolymeric paste has shown a broad hump at 25 to 35 and lower Calcite phases, which corresponds to elevated extent of the course of geopolymerization [21]. The tendencies of FA based CP paste XRD are analogous to 100 FA-paste. Nevertheless, calcite and quartz peaks were monitored as the percent of ground-CP boosted on account of the higher SiO_2 and CaO content [22]. Cristobalite and tridymite peaks were noticed at an elevated percent of CP substitution.

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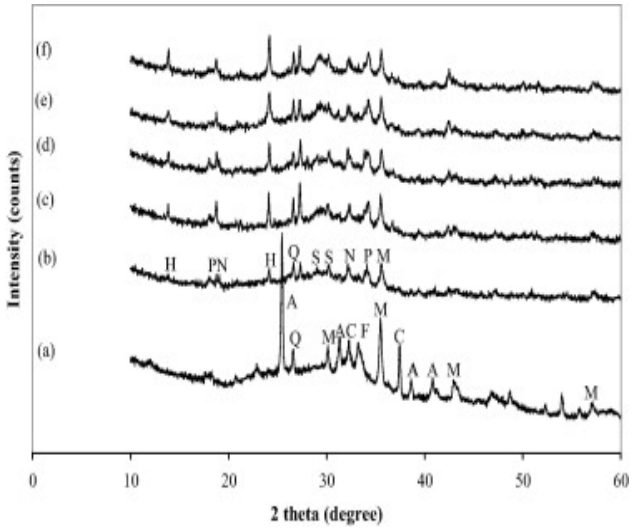

Fig.2 XRD analysis of fly ash-based geopolymer for different mixes [19]

IV. FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR)

In order to confirm GP-structure, Adak et al. [23] have conducted a FTIR analysis. The distinctive intensity band in the vicinity of 460 cm^{-1} was identified for SiA-O-ASi bonding vibration. The band from 750 cm^{-1} to 800 cm^{-1} was found observable on account of the AlO_4 vibration. One more peak for asymmetric stretching and vibration band of SiA-O-AT , whereby, T = Al, Si; which was explained as the most powerful band, registered in area between 950 cm^{-1} to 1050 cm^{-1} [24]. The position 1420 cm^{-1} of SiA-O-ASi in 12GC0H was reallocated to the right position 1485 cm^{-1} in 12GC6. Also, momentous band was placed at roughly 3450 cm^{-1} for OH stretching bonding. The FTIR analysis of FA based GP with container glass pastes and ground fluorescent lamp (FP) in accordance with the experiment by Tho-In et al. [20]. The absorption bands at more or less 3450 cm^{-1} and the feeble band at 1650 cm^{-1} are linked to the vibrations of HA-O-AH and OAH bonds in the H_2O-molecules. Tho-In et al. [20] have unearthed that the H_2O-molecules were either absorbed on the surface or entrenched in pores all through geopolymerization course. The band in the region of 1450 cm^1 has pointed toward the expansion of OA-C-AO in carbonate groups because of reaction between CO_2 in air and hydroxides of alkali metal [25,26]. The uneven stretching of SiA-O-AX, whereby, where "X" represents tetrahedral silicon or aluminium atomic bonds, was monitored in all samples positioned at 1050 cm^{-1}. The SiA-O-AX bonding has propped up course of geopolymerization with phases of amorphous alumino-silicates developed. Also, the researchers have accounted that the sharp absorption band shows a relationship with the number of tetrahedrally coordinated aluminium, which was present in GP-gel [27,28].

What's more, the stretching of SiA-O-ASi and OA-Si-AO bonds can be positioned at 450 cm^{-1} which is the same to group of SiAOAAl [29]. Furthermore, the harmonizing broad bands have been recorded for both FA with GP plus FP and 100 FA samples. Whereas, the sample of 100 FA have been found with accelerated rate of the process of geopolymerization on account of the absorption band at 1050 cm^{-1} was greater than the samples of FP and GP.

V. THERMOGRAVIMETRIC ANALYSIS

Abdulkareem et al.[18] have measured the mass loss as a temperature function among 25°C to 800°C during the thermal analysis. The curves for the GP-paste of and the derivative thermogravimetric analysis (DTG) and the thermogravimetric analysis (TGA). The loss of water owing to evaporation of both, i.e., a little of inside the GP as chemically bonded water and the free H_2O, was exhibited with a decline concerning mass swiftly previous to 150°C[18]. Approximately 55 to 60% of free H_2O existing within GP-matrix get evaporated prior to 100°Cin specimens was depicted by sharp loss in weight. Nevertheless, it is monitored that the weight loss rate was found stabilized in the temperature range of 150°C to 780°C on account of the evaporation of chemically bonded water and OH (hydroxyl) groups. When the temperature was elevated to 800°C, there was no detection of any further loss in mass. Kong and Sanjayan [13] have analyzed behaviour of FA-based GP-paste in very towering temperature conditions. According to Abdulkareem et al. [18], the sharp loss in context to weight was encountered at 250°Cin place of 150°Cthat can also be assigned to evaporation of hydroxyl (OH) group. The alteration concerning weight is rather incessant subsequent to 300°C. The sluggish rate of thermal shrinkage noted from 300°C, and further was found linked to the inferior rate of loss in weight in harmony with Kong and Sanjayan [20]. The average mass penned down was found 89% when subjected beyond 800°C. Duan et al. [53] have performed the thermal analysis of GP-paste incorporated with IOT, i.e., Iron Ore Tailing. The quantity of hydroxide of Calcium [$Ca(OH)_2$] found dropped when the IOT supplement escalated. This was assigned to smaller loss in mass in comparison with specimen for reference. It is comprehended that IOT supplement has assisted for the

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formulation of calcium-silicate-hydrate (C – S – H) by employing $Ca(OH)_2$. Nonetheless, even subsequent to 30% of IOT addition in the mixture, the breakdown of $Ca(OH)_2$ found reported matching to endothermic peak. It was revealed that Ca $(OH)_2$ still exists as merely a number of Ca was transformed into C – S – H. Analogously, Abdulkareem et al. [18] have accounted the rate of loss in mass at towering temperature further than 800°Cwas encountered as insignificant for each single specimen. Okoye et al. [10] have carried out thermal analysis to investigate thermal stability of GPs. The loss in weight for both specimens of pure FA based GP and kaolin blended FA based GP occurred at around 100°C. A rather sharp plunge in weight was monitored in the case of FA based GP when temperature was augmented while the another sample of kaolin amalgamated FA based GP represented a complex curve. The upshots of monitoring of both the specimens have escorted to end that an ideal temperature for curing can vary from 80°Cto 100°C.

CONCLUSION

The review of novel GP-composites for construction, especially, GPCs are found within acceptable norms with regard to their microstructural properties to establish them as potentially promising structural materials. The micro-structural performance of GPCs incorporating dissimilar wastes with total or partial replacement of cement was found extraordinarily practicable. Looking to the review of studies on SEM images of FA-based GPCs, it was found that the consideration of molarity, ratio of alkali solution, adding up binders, curing temperature and age of curing are influential factors on their parameters. The denser matrix is allocated to the FA fineness helping to lessen formulation of micro cracks. The strength of GPs was encountered as quite enhanced on account of the aptitude of fly ash to plug micro-cracks. The elevated measure of handy silicates for geopolymerization is possibly a reason to contribute better strength. An elevated mass ratio of alkaline activator along with the curing temperature has likely provided denser structure. Sufficient quantity of water for geopolymerization probably gives higher strength to GPs. The GPCs incorporating with nano-silica is found augmenting amorphous content. The dry mixes of nano-silica blending GP-composites possibly represent as superior filler in matrix of GPs.

On the other hand, the XRD analysis of GPs, particularly FA based, has exhibited a wide-ranging hump amongst 20 to 35 points toward the presence of amorphous GP-products. Demonstration of low Calcite phases found favourable for elevated degree of geopolymerization. While FTIR analysis are found with the strongest band recorded in area among 950 cm^{-1} to 1050 cm^{-1}. The 100 FA samples had been reported with augmented rate of geopolymerization. In context to thermogravimetric analysis, the loss in mass found as a function of temperature amongst 25 to 800°C. during the thermal examination. The H_2O loss on account of evaporation of petite H_2O within the structure of GP existing as chemically bonded water and also of the free H_2O had displayed a drop into mass weight quickly before touching 150°C. However, the rate of weight loss was stabilized in between 150°C to 780°C temperature as a result of evaporation of chemically bonded H_2O and hydroxyl groups. But, beyond 800°C temperature, there has been no spotting of any mass loss ahead. The review of upshots also leads to unveiling that an ideal temperature of curing may range from 80°C to 100°C. Thus, all the results of reviewed micro-structural properties are found under standard limits and permit GPCs to employ as novel construction materials, however, still advanced researches are needed to support them entirely as future edifice materials.

To sum up, GPCs are innovative, user and eco-friendly, durable, sustainable, owning excellent attributes viz., resistances to heat, fire, freeze-thaw, acids, chemicals, strength, durability, sustainability with agreeable standard of micro-structures and cost-effective and can be regarded as alternative building materials for construction and infrastructure industries. On the top of that, amalgamation of varied wastes from diverse sources is feasible to employ them for manufacturing of dissimilar GP-composites extending a solution to their disposal dilemma and consequential predicaments like landfilling, contamination of air, soils and waters, along with health hazards. Also, GPCs of this kind provide relief to crises such as degradation of confined natural resources, global warming, etc. These are the core reasons why world researchers are endeavoring to promote this pioneering technology using no OPC at all. Geopolymer technology has come forward like a ray of hope for possible stand-in of traditional OPC-system. The existing modern and potential research works in context of this earth-shattering novel Geopolymer technology will revolutionize infrastructure and construction industries and seems to coerce a modern era towards green concept adoption by establishing GP- composites like GPC as acceptable, most promising, building materials for days to come as a need of the hour with a view to breathe in fresh environments and have wealthy restricted resources to pass on to future generation. This will fruitfully progress the notation to “Live and Go Green” for times to come to save our beautiful planet earth!

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