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RESEARCH ARTICLE

Feasibility Studies on Compressive Strength of Ground Coal Ash Geopolymer Mortar

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Abstract

Coal ash obtained from the Thermal Power plants consists of a major portion of Flyash and less amount of Bottom ash. As a major portion of the coal ash, fly ash has been recognized widely as a source material for geopolymers while the utilization of bottom ash has received very less attention owing to its coarser size.Hence to promote greater utilisation of bottom ash in construction sector, this work proposes the use of ground bottom ash in geopolymer mortar. The effect of molar ratio of SiO_2/Na_2O, $Na_2SiO_3/NaOH$ ratio and curing mode were studied on compressive strength of coal ash geopolymer mortar. Molarity of sodium silicate solution was kept as 8M. Ambient curing and steam curing at 60ºC was attempted. Test results indicate that $Na_2SiO_3/NaOH$ of ratio 2 with SiO_2/Na_2O molar ratio 1 mortar achieved higher compressive strength under both ambient curing and steam curing.

Keywords

ground bottom ash · curing · molarity · molar ratio · compressive strength

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1 Introduction

There has been a growing demand for new construction materials with low green house gas emissions and effect on sustainability for the past few decades. Moreover, the boom in the industrial sectors has paved way for the consumption of cement in a massive manner. Although cement is an inevitable part in the construction sectors, its deleterious effects on the environment has made it less viable for the present and future scenario, mainly because of its enormous CO_2 emissions. Accordingly, the cement industry strives to reduce this carbon footprint by the usage of industrial by-products like fly ash, GGBS, silica fume, rice husk ash, etc. Further, an alternate class of binders namely geopolymer which is a cementless one is emerged in the recent decades.

Prof. Davidovits was the first to coin the term geopolymer for the alternative binders which could be formed with in the presence of alkaline conditions. Geopolymerisation involves the dissolution of silicates and aluminates in alkaline solutions which may be sodium or potassium based. During the early stages of geopolymer invention, only naturally occurring raw materials had been traditionally used. As of now any material which is rich in silica and alumina could be used for geopolymerisation reaction. Nowadays major emphasis has been made to utilize waste materials such as fly ash, GGBS, rice husk ash, etc economically instead of naturally occurring raw materials. Geopolymers are now emerging as a potential solution to lower the emission of greenhouse gases and energy consumption. Further, geopolymers have many advantages such as good compressive strength and resistance to aggressive environment and long term durability. [9, 18].

Coal ash refers to the residue remaining after the combustion of coal at 1500°C in suspended state and it collectively represents fly ash and bottom ash. Fly ash is collected in the chimney (Electrostatic precipitator) whereas bottom ash is collected from the boilers. Coal ash has been moved from the category of “hazardous industrial waste” to “waste material” category in 2000 and has become a scalable commodity. Indian coal is of low grade having ash content upto 30 - 40% in comparison to imported coals which have ash content of 10 - 15%. Coal ash generates about 80% fly ash and 20% bottom ash.

The total ash generated in the year 2011 - 12 is 66.5 MT and the ash utilized is 36.3 MT, the percentage of utilization being 55% [Source -" Report on Fly ash generation, Central electricity Authority, 2011 - 12]. However, not much focus has thrown on use of bottom ash in construction work similar to fly ash. As a major portion of the coal ash, only fly ash has been recognized widely as a source material for geopolymers while the utilization of bottom ash has received very less attention.

Bottom ash is also rich in silica and alumina similar to fly ash [2, 10, 23, 24] and hence geopolymer concrete using bottom ash has similar emphasis in reducing CO_2 emission and recycling of bottom ash. Moreover bottom ash contains toxic elements like lead, cadmium, copper which leach out in due course and contaminate the soil [1, 10, 11]. However, bottom ash as such cannot be used directly owing to its coarser size and porous structure. Although fly ash and bottom ash are obtained from the same source, their physical and phase properties are different with bottom ash having less glassy constituent phase [6]. Furthermore, bottom ash as obtained has angular fragments and less spherical particles [7]. It has to be ground finely as that of fly ash to increase the pozzolanic activity for using as partial replacement of Portland cement [1, 4, 21]. The utilization of bottom ash as a replacement to cement has not yet been well acknowledged as it itself is porous in nature and increases the water requirement of the mixture [13]. Whereas on the other hand, grinding of bottom ash has the consequences of prolonged setting time and reduces the workability of the mix [1, 10, 12]. Consequently, several efforts are in progress on use of bottom ash in geopolymer concrete [1, 17, 21, 27, 28].

Some of the important parameters that influence the strength of geopolymer concretes are alkaline liquid to binder ratio, molar ratio of SiO_2 / Na_2O in sodium silicate solution, molarity of sodium hydroxide, ratio of $Na_2SiO_3 / NaOH$, and the curing mode. Researchers reported that an increase in the alkaline liquid to binder ratio increases the compressive strength as well as workability of geopolymer mortar. The increase in the sodium silicate to sodium hydroxide ratio increased the reaction higher rate and thereby develops long chain polymerization when compared to sodium hydroxide [19, 26]. The composition of sodium silicate is predominant in improving the polymerization process. The ratio of SiO_2 / Na_2O is the key factor in developing the geopolymerization process. [25] found that both dosage and molar ratio of SiO_2 / Na_2O have significant role on the strength of geopolymer mortar. [3] mentioned that lower molar ratio of SiO_2 / Na_2O can exhibit higher compressive strength. Lower ratio of silicates with higher alkali concentration is recommended for activating pozzolans.

The increase in sodium silicate solution affected the strength because of the larger interparticle distance [1, 28]. The molarity of sodium hydroxide solution increases the strength [28]. This may be due to the fact that higher molarity breaks the glassy structure of the source material which aids in the internal Si and Al contents to activate the geopolymerisation.

The alkaline liquid plays an important role in polymerization process. The addition sodium silicate and sodium hydroxide solutions as the alkaline liquid enhanced the polymerization reaction. Water does not play a significant role as it is expelled during the polymerization reaction. Also addition of excess water makes the structure porous and reduces strength. Hence a controlled quantity of water was used in production of bottom ash geopolymer mortar.

Mustafa Al Bakri et al (2011) reported that heat is required to increase the geopolymerisation process to obtain higher compressive strength. Kirschner et al. (2004) states that, geopolymerisation at ambient temperature was unfeasible due to a delayed beginning of setting [29]. He also reported that curing at 75ºC for 4 hrs completed a major part of geopolymerization process and resulted in satisfactory properties of the material. The temperature during curing depends upon the source materials and activating solution, heat often must be applied to facilitate polymerization, although some systems have been developed that are designed to be cured at room temperature [5].

Although geopolymer concrete has Geopolymer coexistence much promise, the use of geopolymer concrete is not as common as would be expected considering the potential benefits. A major challenge in implementing the geopolymer concrete is its use in the cast in situ construction work. From the above literatures it is evident that alkaline liquid to binder ratio, molarity of sodium silica solution, molar ratio of alkaline liquids and mode of curing play an important role in geopolymerisation.

Accordingly, the present work undertakes a feasibility study on the effect of various parameters such as molar ratio of SiO_2 / Na_2O in sodium silicate solution, ratio of $Na_2SiO_3 / NaOH$, and the curing mode on the compressive strength of bottom ash based geopolymer mortar

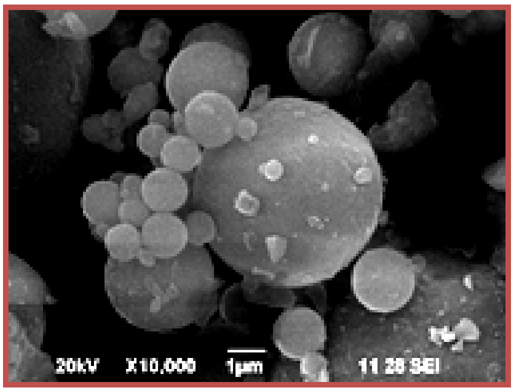
2 Materials and Experimental Details

2.1 Materials

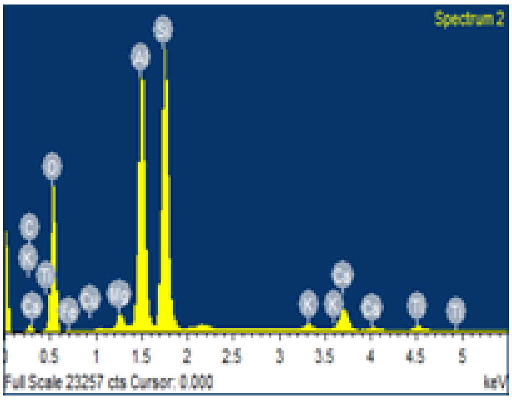
Bottom ash: Bottom ash used in this study was obtained from Mettur thermal power Station. It was obtained in wet condition and it was coarser. Therefore, it was ground in the pulveriser to the fineness with 95% fraction passing through 45 micron sieve. Grinding of bottom ash increases the surface area similar to Flyash (Fig. 2.a) and hence improves the reactivity. After grinding the specific surface area was 3460 cm^2 / gm. The specific gravity of the bottom ash was 2.17. The chemical properties and SEM image of bottom ash are presented in Table 1 and Fig. 2.a.

Alkaline Activators: A combination of sodium silicate solution and sodium hydroxide solution was chosen as the alkaline liquid activators. The sodium silicate solution and sodium hydroxide (NaOH) in flakes 97% to 98% purity were purchased from a local supplier in bulk. The NaOH flakes were dissolved in water to make the solution.The properties of Na_2SiO_3 and NaOH are shown in Table 2 and 3.

River Sand: Locally available river sand conforming to grading zone III as per BIS 383-1970 was used as filler for bottom ash geopolymer mortar in this work. The properties of Fine aggregate are shown in Table 4.



(a)



(b)

Fig. 1. SEM and EDAX of Bottom ash

Tab. 1. Chemical Properties of Bottom ash

Chemical Compositions	Percentage by Weight
SiO_2	51.50
Al_2O_3	32.58
SO_3	5.19
Fe2O3	7.28
CaO	0.50
MgO	0.21
Na_2O	1.35
K_2O	0.58
LOI (Loss of Ignition)	1.50

2.2 Experimental Details

2.2.1 Mix Composition

The parameters such as, molar ratio of SiO_2 / Na_2O in sodium silicate solution, ratio of Na_2SiO_3 / NaOH and curing mode were considered to find out the effect of bottom ash geopolymer (GP) mortar on the compressive strength. The ratio of bottom ash to sand was selected as 1:3 based essentially on a measurement of the 'voids by volume' within a measure of dry sand. The method used to measure the voids involves half-filling a graduated laboratory flask with an oven-dried sample of the specified sand,

Tab. 2. Properties of Na_2SiO_3 solution

Chemical Compositions	Percentage by Weight
Na_2O	19.6%
SiO_2	28.9%
Water	51.5%
Molar ratio (SiO_2/Na_2O)	1.5
Appearance	Light yellow liquid (Gel)
Specific gravity	1.54

Tab. 3. Properties of NaOH flakes

Chemical Compositions	Percentage by Weight
Assay	97%
Na_2CO_3	1.8%
Cl	0.01%
SO_2	0.05%
Pb	0.001%
Fe	0.001%
K	0.1%
Zn	0.02%

and then carefully pouring clean (potable) water into it from another identical graduated flask until all the voids are filled and the surface of the water rises level with the surface of the sand. The volume of water required to fill all the voids in this volume of sand can then be calculated by subtracting the volume of water left in the water flask from the volume it contained at the start, this being determined as the minimum volume of binder required for producing a good mortar. Typically this is found to be one-third of the original volume of the water and hence the ratio is determined as 1:3. The alkaline liquid/binder ratio plays a very important role in geoplymeristion. Rangan, (2008) suggested the alkaline liquid/binder ratio between 0.35 and 0.55 for fly ash based geopolymer [22]. For mix design of BAGPM the following parameters were considered to arrive at the quantities of the constituent materials.

Alkaline liquid to binder ratio: 0.4

Ratio of Na_2 SiO_3/NaOH: 1, 1.5, 2, 2.5

Water/Binder ratio: 0.3

Molarity of NaOH solution: 8M

Molar ratio of Na_2 SiO_3 Solution: 1,1.2,1.4,1.6,1.8,2

Curing Temperature: 60°C

Curing Mode: Ambient and Steam curing

Tab. 4. Properties of Fine aggregate

Properties	***Value***
Fineness Modulus Zone III	2.26
Water absorption	0.58%
Bulk density(SSD)	1.54 gm/cc
Specific gravity (SSD)	2.63

3 Mix Design of Geopolymer mortar

The mix design of Geopolymer mortar is presented below

Ratio of Bottom ash: Sand = 1:3

Bottom ash = 200 gm, Sand = 600 gm

Alkaline liquid/Binder ratio = 0.4

Alkaline liquid = 0.4* Binder = 80 gm

Na_2SiO_3 / NaOH = 2.5

Alkaline liquid = Na2SiO3+NaOH solutions

Na_2SiO_3 + NaOH = 80

Na_2SiO_3 solution = 57.14 gm

NaOH solution = 22.86 gm

For NaOH solution of molarity 8,

Quantity of NaOH solids = 8*40 = 320 gm of solids in

1 litre of water.Hence for 22.86 gm of NaOH solution

Quantity of NaOH solids = 5.63 gm

Quantity of water = 17.6 ml

Water/Binder ratio = 0.3

Water content = 60 gm

Extra water to be added if needed = 13 gm

Water to Geopolymer solids ratio = 0.2

Tab. 5. Mix proportions for geopolymer mortar

S.no	Description	Quantity/ Mortar cube (0.0003579 m^3)
1	Bottom ash	200 gm
2	Sand	600 gm
3	NaOH solution	22.86 gm
	NaOH solids	5.63 gm
	Water for NaOH solution	17.6 ml
4	Na_2SiO_3 solution	57.14 gm
5	Water/Geopolymer solids ratio	0.2
6	Extra water to be added	13 ml

3.1 Specimen Preparation

The method of mixing for GP mortar was followed as per the procedure given by Rangan (2008) [22]. The raw materials bottom ash and sand were thoroughly mixed for few minutes in the pan mixer. This was followed by the addition of the alkaline liquid and the mixing was continued for another 4 - 5 minutes. The preparation of the solution of alkaline liquid was immediate to the mixing with the source material. The fresh geopolymer paste is then filled in 70.6 x 70.6 x 70.6 mm moulds and are compacted by vibration to expel the air voids. Then the moulds are sealed to avoid any water loss in evaporation.

3.2 Curing

Two curing modes were attempted, ambient curing and steam curing. Ambient curing was achieved by placing the specimens completely sealed or covered for 24 hours under room temperature. The specimens were then demoulded and left at room temperature till the day of testing. Steam curing was achieved by placing the sealed specimens in a steam curing chamber provided with an electrical boiler. Water was supplied continuously and the temperature inside the steam curing chamber was maintained at 60°C for 24 hours. Upon curing, the specimens were demoulded and allowed to cure in room temperature till the period of testing.

3.3 Compressive Strength

The compressive strength of the bottom ash geopolymer mortar cubes were taken for each mix proportion and the strength at 3, 7 and 28 days were evaluated for the two curing modes, ambient curing and steam curing at 60°C.

4 Results and Discussions

4.1 Compressive Strength of Bottom ash Geopolymer Mortar (BAGPM)

The various parameters such as ratio of Na_2SiO_3 / NaOH, molar ratio of SiO_2 / Na_2O of Sodium hydroxide solution (Na_2SiO_3 solution), and curing mode on compressive strength of bottom ash geopolymer mortar were studied. The compressive strength of bottom ash geopolymer mortar (BAGPM) were determined at the age of 3, 7 and 28 days are given in Table 5. In order to indentify the various mixes, alphabetical letters A, B, C, & D are given for ratios of Na_2SiO_3 / NaOH solutions from 1 to 2.5 respectively. Each alphabetical letter is provided with numerical to denote the molar ratio of SiO_2 / Na_2O in sodium silicate solution.

a)Ratio of Na_2SiO_3 / NaOH solutions:

Bottom ash-based mortar achieves its strength development by the activation of the geopolymerization under highly alkaline environment. From the results it can be observed that increasing the Na_2SiO_3 / NaOH solution ratio from 1 to 2.5, increased the compressive strength for most of the mixes. Maximum strength was attained for a ratio of 2.0 (Identity C).The increase in compressive strength may be attributed to the increase in the SiO_2 content of Na_2SiO_3 solution than the NaOH solution. But increasing the Na_2SiO_3 content to 2.5 decreases the compressive strength. This may be due to the reason that greater amount of soluble silica in sodium silicate solution retards the geopolymerisation reaction.This is in similar opinion of Xu et al (2000) that the higher amount of sodium silicate improves the geoplymerisation when compared to sodium hydroxide content [26].

b)Molar ratio of SiO_2/Na_2O of Sodium silicate solution

In all the mixes, molar ratio of SiO_2 / Na_2O of sodium silicate solution showed remarkable compressive strength in both ambient and steam curing condition. A mixed trend was observed while varying the molar ratio of SiO_2 / Na_2O for the varying ratios of Na_2SiO_3 to NaOH solutions. For Na_2SiO_3 / NaOH ratios of 1 and 2, decrease in the molar ratio increased the compressive strength whereas for ratios of 1.5 and 2.5 increase in the molar ratio decreased the compressive strength. Maximum strength of 41.53 MPa(under ambient curing) and 48.55 MPa(under steam curing)was attained for a molar ratio of 1 for Na_2SiO_3 / NaOH ratios of 2.0.

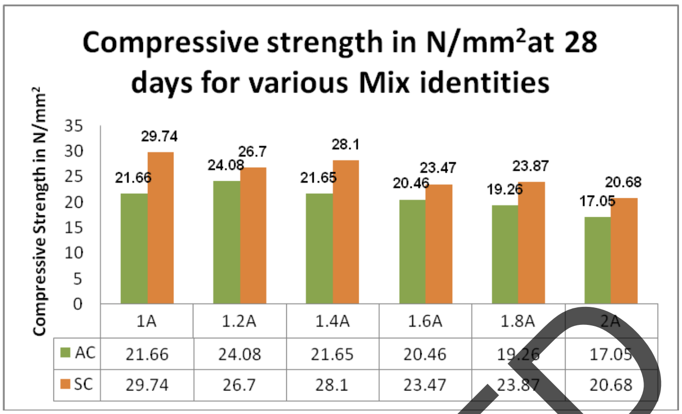
A mixed trend is noticed when SiO_2 / Na_2O ratio was increased in the BAGPM for different Na_2SiO_3 / NaOH. Rangan (2008) mentioned that increasing the ratio of SiO_2 / Na_2O significantly improves the degree of polymerization [22]. This is confirmed for Na_2SiO_3 / NaOH = 1.5 mixes. This is also in opinion of Kirgiz.M (2011,2014) who has mentioned that the fluctuations in SiO_2 and Na_2O content greatly affects the compressive strength of mortars wherein continuous increase in SiO_2 content increases the compressive strength proportionally [14, 15]. However, in all other mixes the best activation was noticed for SiO_2 / Na_2O with lower ratio 1. This is confirmed with the findings of Dali Bondar et al (2011) [3].

c) Curing Mode

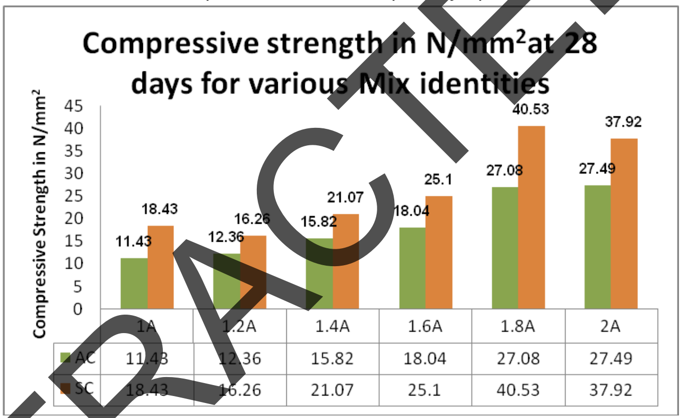
From the results it can be seen that nearly similar maximum compressive strengths are achieved at the age of 28 days under both ambient and steam curing conditions for mix 1C,while in all the other mixes there is sufficient difference between the strengths at ambient and steam curing. This is mainly due to the fact theat steam curing enhances geopolymerisation as mentioned by B.V.Rangan(2008) [22]. Kirschner et al (2004) reported that ambient temperature was not viable for geopolymerisation [29]. This is to some extent true for many BAGPM mixes especially at early periods. However, with the proper selection of SiO_2 / Na_2O with suitable Na_2SiO_3 / NaOH, it is possible to obtain the enhanced geopolymeric reaction at later ages. The mix 1C achieved remarkable compressive strength even at early age under ambient temperature. This is a beneficial finding with respect to practical constraints in cast-in-situ construction.

4.2 Microstructre Analysis of Bottom ash Geopolymer Mortar (BAGPM)

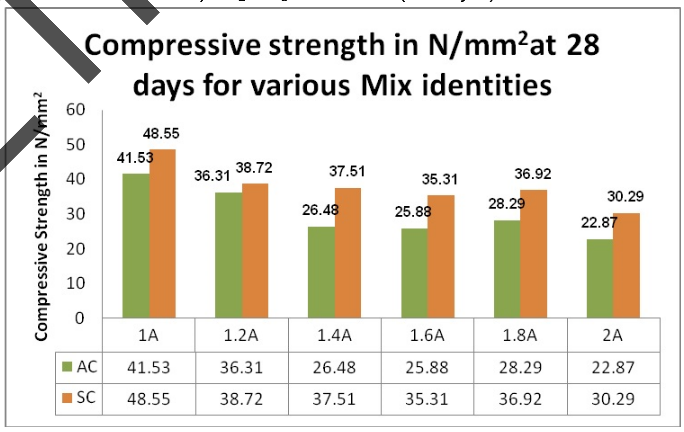
The microstructure study was undertaken to observe the microstructure, study the effect of different mix formulations and identify pores and cracks, if any. Scanning electron Microscopy (SEM) was used to record micrographs and the microstructure of bottom ash geopolymer mortar was found to contain mainly amorphous aluminosilicate gel and unreacted spheres of bottom ash. SEM imaging was conducted using Philips XL-30 using secondary as well as backscatter electron detectors. Most of the Bottom ash particles were found to be dissolved in the alkaline solution and formed C-S-H gel with the addition of silica, while some did not dissolve due to less quantity of silica available for C-S-H formation Uniformly distributed microcracks were also found on the surface of the bottom ash geopolymer mortar specimens. The SEM image of geopolymer mortar are shown in



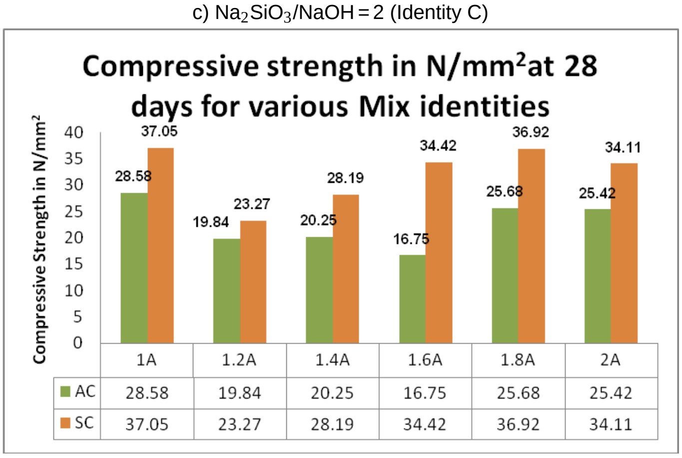
a) Na_2SiO_3/NaOH = 1(Identity A)



b) Na_2SiO_3/NaOH = 1.5(Identity B)



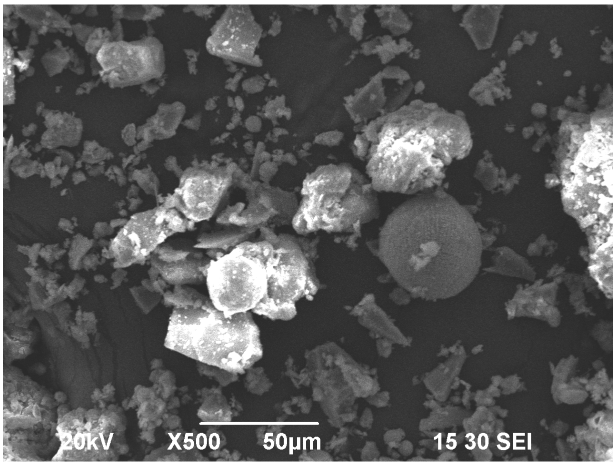
c) Na_2SiO_3/NaOH = 2 (Identity C)



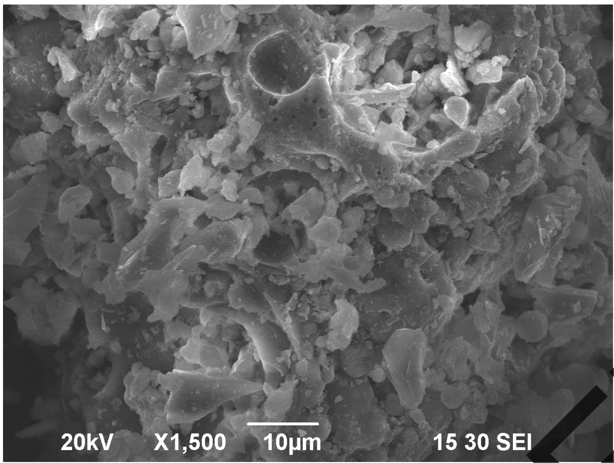
d) Na_2SiO_3/NaOH = 2.5 (Identity D)

Fig. 2. Compressive Strength of BAGPM at the age 28 days for Ambient and Steam curing

Fig. 2(a)&(b).



(a) SEM image of Geopolymer mortar



(b) Geopolymer coexistence and CSH phase

Fig. 3. SEM image of Bottom ash geopolymer mortar

5 Conclusion

The following concluding remarks are presented on the study of effect of molar ratio of SiO_2/Na_2O, $Na_2SiO_3/NaOH$, and curing mode on compressive strength of bottom ash geopolymer mortar :

- Low SiO_2/Na_2O of 1 attained greater compressive strength for BAGPM which indicates that sufficient amount of alkalis Na_2O is required for geopolymerisation.
- Increasing $Na_2SiO_3/NaOH$ ratio achieved maximum compressive strength for BAGPM. The ideal ratio of $Na_2SiO_3/NaOH$ is 2.
- The molar ratio of SiO_2/Na_2O of 1.0 with $Na_2SiO_3/NaOH$ ratio 2 was the one that yielded the greatest compressive strength It developed compressive strength of 41.53 MPa under ambient curing and 48.55 MPa under steam curing at 60°C at the age of 28 days. The findings obtained from potential geopolymer mortar with respect to molar ratio of SiO_2/Na_2O, $Na_2SiO_3/NaOH$ can be extended for geopolymer concrete.
- Comparable compressive strength was achieved for the mix 1C under ambient and steam curing at 60°C. It is also revealed that with proper selection of SiO_2/Na_2O and $Na_2SiO_3/NaOH$ ratio, best geopolymerisation could be achieved even at ambient temperature. It is the positive aspect to use geopolymer technology in cast in situ concrete construction.

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