

MECHANICAL AND WATER TRANSPORT PERFORMANCE OF SUSTAINABLE GEOPOLYMER COMPOSITE USING BINARY BLENDS OF POZZOLANIC MATERIALS

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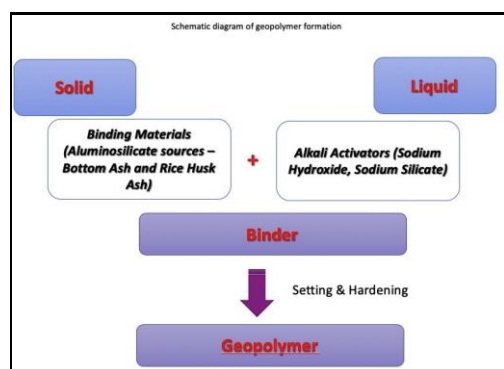
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Graphical abstract



Abstract

The focus of the researchers has now shifted towards the geopolymer based materials as it considered as sustainable alternative to the existing cementitious materials. This paper attempts to incorporate the binary blend of pozzolanic material to develop geopolymer composite and understand its mechanical and water transport performance as a building material. The combination of bottom ash and rice husk ash with fly ash as a common binder was used for development of geopolymer composite. Replacement levels of both bottom ash and rice husk ash was kept at 40%, 30% and 20% of total solid proportions. Also, the molarity of NaOH was provided at 12 M and 14 M levels respectively. The results showed that the blend of FA-BA blend to be better performed against the blend of FA-RHA blend for their mechanical and water transport properties. The compressive strength of the geopolymer composite having blend of fly ash and bottom ash reached to the value of 41.49 MPa due to its suitable Si/Al ratio for geopolymerization reaction, which is quite remarkable. Also, the results of water transport performance shows the blend of FA-BA to be 15-20% more resistant to percolation of water as compared to FA-RHA blended geopolymer composite, thus lowering the risk of damages to the structures.

Keywords: Geopolymer, Pozzolanic Materials, Bottom ash, Rice Husk Ash, Sustainable

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1.0 INTRODUCTION

The construction industry is the one of the fastest growing sector, which consumes abundant amount of mineralogical and biological resources, thus causing serious environmental concerns. Due to the rapid socio-economic growth and consumption of natural resources, there is urgent need to divert the attention

of construction industry towards sustainable materials and technologies. One of the most consumed material in construction industry is cement, which proves to be energy consumptive and unsustainable due to its production process. The amount of cement produced is estimated to be 4.0 billion tonnes per annum and it is supposed to be growing at 4% each year [1-2]. The amount of cement is equivalent to 626

kg per capita [3]. India is one of the fastest developing countries, having cement production at the rate of 190 kg per capita and it is estimated to increase to about 350 kg per capita by the year 2025 [4-5]. Due to enormous demand and production of cement as a raw material for the construction sector; it has raised serious environmental hazards as 0.66 to 0.82 kg of CO₂ is liberated in atmosphere for production of per kg of cement, contributing to an overall 7% of global CO₂ emission [6]. Various techniques and materials has been tried and tested to reduce the consumption of cement by using supplementary cementitious materials such as Fly Ash, Metakaolin, Silica Fume, Rice Husk Ash, etc. Furthermore to reduce the dependency on cement researcher has come up with one of the technique known as Geopolymerization to provide cementless binding material [7]. The main advantage of the geopolymer based material is that it does not use energy consumptive cement as a raw material for its preparation.

Geopolymer is one such material which has sustainable traits and it can replace cement as a binder in construction materials. Geopolymerization is the technique used to form an aluminosilicate inorganic polymeric compound developed by polymerization of materials rich in silica and alumina with alkaline (NaOH/KOH) solution [7]. The technical aspect shows improved mechanical and durability parameters by geopolymer based material as compared to energy consumptive conventional cementitious binder [8]. A geopolymer is formed by alkali activation of alumina and silica obtained from various pozzolanic materials which reacts at above the room temperature with an alkaline solution to form an aluminosilicate binder. Crucial factors such as chemical composition of source material, solid to liquid ratio, molarity of alkaline solution, curing temperature, etc has been identified for development of geopolymer materials [9-11]. Various researches [12-14] performed experiments to enhance the performance and applicability of geopolymer based products. It has been reported by Turner *et al.* [6] that geopolymer based products are more sustainable than the cement-based products due to the reduced embodied energy and carbon emission.

Various researchers [7, 8, 12, 13, 14, 15] has developed and analysed performances such as mechanical properties, physical properties, and microstructural characterization of geopolymer material. The brief overview of some literature has been discussed further to understand the behaviour of various alumino-silicate material as a primary ingredient and its effects on final product. Abdollahnejad *et al.* [15] analysed the capillarity and water absorption effects of geopolymer containing fly ash and calcined kaolin with sodium hydroxide as a alkali activator. The developed material showed high capillarity and water absorption with the addition of 8% of calcined kaolin due to its porous structure. In another research, Adak *et al.* [16] performed analysis on geopolymer materials by using 6% of nano-silica

with various molar concentration. The results indicated that due to the modified particle size and high reactivity the water absorption reduced. The compressive strength performance analysed by Chindaprasirt *et al.* [17] on of class C fly ash-based geopolymer mortar achieved the values of 52 Mpa when cured at 70 °C for 3 days using sand-fly ash ratio of 2.75 at workable flow of 135 ± 5%. It was also reported that prolonged curing lowers the compressive strength due loss of moisture causing weakening of bonding and increasing porosity. Huseien *et al.* [18] investigated the influence of metakaolin (MK) on the development of early strength of granulated blast furnace slag (GBFS) based geopolymer. GBFS was replaced up to 15% by metakaolin to develop geopolymer. In another research, tertiary blend of POFA, FA and GBFS was developed to understand its effect on bending strength. Similar types of research has been conducted on supplementary cementitious materials to enhance the performance of geopolymer for practical applications. The attempts made by various researchers has not given much emphasis on the water transport performance of geopolymer material. From the literature review, the gap has been identified that there has not been much effort to understand the behaviour of effects of binary blended geopolymer on water percolation through the material. It is important to understand the behaviour of material against the water permeability to reduce the susceptibility of durability attack as well as to enhance the life span of structure

Thus, the research focuses on the development of the geopolymer material using binary blend of water transport performance as well as mechanical performance. The research intends to improve the performance of geopolymer composite through binary blend of pozzolanic material. The study is inclined towards finding a sustainable alternative to the conventional energy consumptive construction materials using agro-industrial waste.

2.0 METHODOLOGY

The raw materials used for the development of the geopolymer composite were categorized as an alkali activator and binding material. The binders used were fly ash, bottom ash and rice husk ash along with crusher dust which acts as a filler material. The activation of binder was done by alkaline activators, i.e. combination of sodium silicate (Na₂SiO₃) and sodium hydroxide (NaOH). The molarity of sodium hydroxide was kept as 12 M and 14 M to understand the effect of the molarity of NaOH on geopolymerization reaction and its binding effect. The pozzolanic materials (RHA, BA and FA) were procured from locally available sources. Before using these pozzolanic materials, it was sieved through 90 µm to keep its fineness within limits. The crusher dust was also procured from locally available quarry sites.

The physical and chemical tests on fly ash, bottom ash and rice husk ash were conducted to analyse the suitability of the procured material to be used as a pozzolanic material in accordance to IS 1727 (2004). The result of the physical tests consisting of grain size analysis, consistency, specific gravity, drying shrinkage, initial & final setting time, compressive strength and soundness on fly ash, bottom ash and rice husk ash as shown in Table 1. The chemical composition of fly ash, bottom ash and rice ash was determined by

conducting X-ray fluorescence analysis and its results are shown in Table 2. Scanning electron microscopy (Figure 1) was conducted to understand the morphology of the raw materials used in the study. SEM image of rice husk ash shows fibrous irregular shape, whereas bottom ash has irregular spherical size with pores in it. Due to the nature of particles of bottom ash and its size, it is evident the reactivity will be greater in case of bottom ash as compared to rice husk ash due to its morphology.

Table 1 Physical test results of supplementary cementitious materials

Sr. No.	Name of Test	Test Method	Fly Ash	Rice Husk Ash	Bottom Ash
1	Grain Size Analysis	IS 2720:1985	Gravels	0	5.4
			Sand	23.1	71.05
			Silt & Clay	76.9	23.55
2	Consistency (%)	IS 1727:2004	29.5	54	30
3	Specific Gravity	IS 1727:2004	1.995	1.872	1.975
4	Drying Shrinkage (%)	IS 1727:2004	-0.025	-0.0378	-0.0565
5	Initial Setting Time (Mins.)	IS 1727:2004	120	160	140
6	Final Setting Time (Mins.)	IS 1727:2004	230	280	250
7	Compressive Strength (MPa)	IS 1727:2004	-	18.7	32.2
8	Soundness by Autoclave (%)	IS 1727:2004	-	0.0637	0.0608

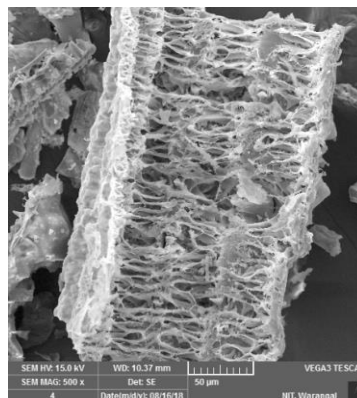
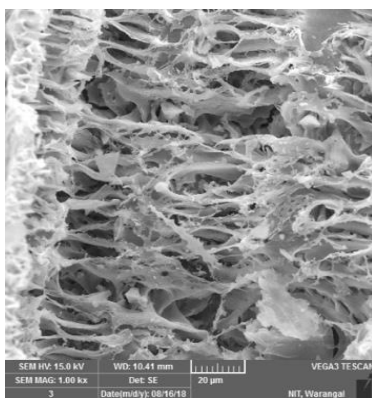


Figure 1(a) SEM image of Rice husk Ash

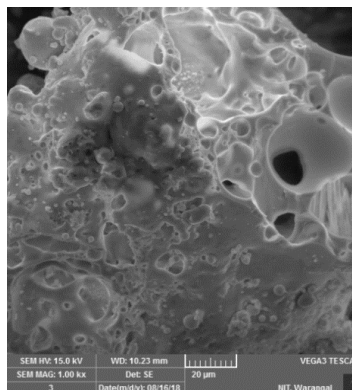
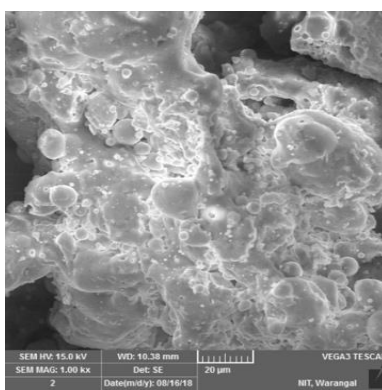


Figure 1(a) SEM image of Bottom Ash

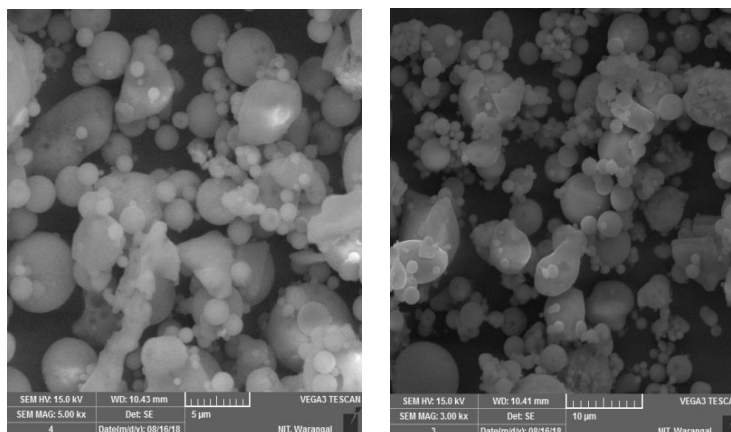


Figure 1(a) SEM image of Fly Ash

The rice husk ash and bottom ash was kept at 3 levels i.e. 40%, 30% and 20% of total weight of mix, according to the varying proportions of fly ash in the design mix. Whereas, filler materials (i.e. crusher dust) were kept constant at 30% of total weight of mix, due to its non-reactive nature with other raw materials. For an alkaline solution, a combination of sodium hydroxide in flake form and sodium silicate solution was used as the alkaline activator. The solution was prepared at least 1 day prior to its use to allow the exothermically heated liquid to cool to ambient temperature. The alkali activator solution was prepared using 12 M and 14 M solution of sodium hydroxide.

The solids were stirred in a rotary mixer for approximately 2 - 5 minutes to form a homogenous dry mix; it was then followed by the addition of alkaline solution which was prepared separately one day prior to the casting. The mix was then poured into the steel moulds and the specimens were covered with plastic sheets to prevent the surface water from evaporating. The specimens were then transferred to the curing chamber and cured at a temperature of 65°C for 48 h before being de-moulded and tested. The solids were stirred in a rotary mixer for approximately 2 - 5 minutes to form a homogenous dry mix; it was then followed by the addition of alkaline solution which was prepared separately one day prior to the casting. The mix was then poured into the steel moulds and the specimens were covered with plastic sheets to prevent the surface water from evaporating. The specimens were then transferred to the curing chamber and cured at a temperature of 65°C for 48 h before being de-moulded and tested.

2.1 Materials Testing

The developed geopolymer composite mixes were subjected to various types of tests to check its compliance with various building material standards. The physical traits such as bulk density, water absorption and apparent porosity of geopolymer composite was tested in accordance to ASTM C373-88

standard [19]. The mechanical performance of the geopolymer composite was analysed by using unconfined compressive testing on cubes which had a sample size of 100 x 100 x 100 mm³, and a compression testing machine of 2000 kN capacity at a loading rate of 0.75 kN/s. The test was conducted for 7, 28 and 90 days of cured samples in accordance with ASTM C109 [20]. Whereas, flexural strength was conducted for 7, 28 and 90 days of curing in accordance with ASTM C348 [21] for all geopolymer composite samples having a prism size of 25 x 25 x 250 mm³.

The developed geopolymer composite samples were tested for measuring the rate of water absorption through its pores. The amount of water absorption mainly relies on the interconnected pores within the geopolymer matrix. Experimentation was conducted on the samples being sealed by epoxy coating on its sides to maintain the unidirectional flow of water, whereas the one of the face of the cube were exposed to the water. The tests were conducted in accordance with ASTM C 1585 [22]. The formula used for calculation for velocity of water absorption is given below;

$$I = \frac{\Delta m}{ad}$$

Where;

I = velocity of water absorption (m/s^{1/2}),

Δm = gain in mass (kg/s^{1/2}),

a = exposed area of specimen (m²),

d = density of water (kg/m³).

Coefficient of water absorption was calculated by determining the uptake of water through a sample for a time period of 1 hour. The geopolymer composite samples were oven dried at a temperature of 110°C for a week to remove moisture content within the samples. Then, the sides of the samples were covered with epoxy resin and placed in a container containing water with a depth of 5 mm. The amount of water absorbed during 1 hour was measured. The coefficient of water absorption was calculated by using the formula given below:

$$Ka = \left(\frac{Q}{A}\right)^2 \frac{1}{t}$$

Where,

Ka = Coefficient of water absorption (m/s²),

Q = Quantity of water absorbed (m³),

A = Surface area (m²)

Table 2 XRF analysis of supplementary cementitious materials

Sample	Bottom Ash (%)	Fly Ash (%)	Rice Husk Ash(%)
SiO ₂	44.77	50.47	46.91
TiO ₂	22	9.6	23.37
Al ₂ O ₃	20.37	28.76	0.08
MnO	0.09	0.04	0.11
Fe ₂ O ₃	5.08	4.3	0.49
CaO	2.14	0.81	0.37
MgO	0.64	0.39	0.08
Na ₂ O	0.36	0.09	ND
K ₂ O	0.57	0.77	0.08
P ₂ O ₅	0.17	0.31	1.82
SO ₃	2.98	4.3	ND
BaO	ND	ND	24.48

Table 3 Mix design for geopolymer based material

Mix No.	Supplementary Cementitious Material	SCM (% wt.)	FA (% wt.)	CD (% wt.)	Na ₂ SiO ₃ (grams. In total wt. of sample)	Molarity of NaOH	NaOH Sol. (grams. In total wt. of sample)
CTRL	FA	0	70	30	1375	12M	178.2 g. NaOH + 371.8 ml water
M1	RHA	40	30	30	1375	12M	178.2 g. NaOH + 371.8 ml water
M2	RHA	30	40	30	1375	12M	178.2 g. NaOH + 371.8 ml water
M3	RHA	20	50	30	1375	12M	178.2 g. NaOH + 371.8 ml water
M4	RHA	40	30	30	1375	14M	157.1 g. NaOH + 392.9 ml water
M5	RHA	30	40	30	1375	14M	157.1 g. NaOH + 392.9 ml water
M6	RHA	20	50	30	1375	14M	157.1 g. NaOH + 392.9 ml water
M7	BA	40	30	30	1375	12M	178.2 g. NaOH + 371.8 ml water
M8	BA	30	40	30	1375	12M	178.2 g. NaOH + 371.8 ml water
M9	BA	20	50	30	1375	12M	178.2 g. NaOH + 371.8 ml water
M10	BA	40	30	30	1375	14M	157.1 g. NaOH + 392.9 ml water
M11	BA	30	40	30	1375	14M	157.1 g. NaOH + 392.9 ml water
M12	BA	20	50	30	1375	14M	157.1 gm NaOH + 392.9 ml water

3.0 RESULTS AND DISCUSSION

3.1 Effect on Compressive and Flexural Strength

The results of compressive strength of geopolymer composite samples developed from blending of FA-RHA and FA-BA are shown in Table 4. The increase of strength in geopolymer composites can be attributed to the formation of aluminium silicate hydrate/calcium silicate hydrate gel which has similar binding effects as C-S-H gel in cementitious materials [22, 23]. From the

Table 4, it is evident that the combination of FA-BA shows higher compressive strength than combination of FA-RHA. The highest strength for combination of FA-BA was found to be 41.49 MPa as compared to combination of FA-RHA having a compressive strength of 31.33 MPa with the same mix proportion. The strength gain in the geopolymer composite is due to the reaction between silica and aluminates in presences of highly alkaline activator. The ratio of Si/Al for strength in geopolymer composite should be $3.3 < \text{Si/Al} < 4.5$ [24], for BA-FA combination it is around 3.88 to

4.16, whereas for RHA-FA combination it is 5.03 to 8.33. Thus, due to imbalance in Si/Al ratio of RHA blended geopolymer composite mix, it does not gain much bonding than that of BA blended geopolymer composite mix. Also, from the morphology of SEM images (Figure 1), the surface area and particle size plays important role in the reactivity, as mentioned earlier BA has more reactivity due to its morphology (spherical porous nature) in addition to chemical composition as well. Also, from the Figure 2, it can be observed that fly ash (FA) plays a dominant role in developing compressive strength. From the Figure 2 it is clear that compressive strength of geopolymer composite decreases with a decrease in the fly ash (FA) content. The compressive strength is higher when the fly ash content is 50% in all solids. However, the control mix geopolymer composite (CTRL mix) solely developed from fly ash (FA) shows less strength compared to the blended mix of supplementary cementitious materials (please refer Table 4). Also, higher molarity of NaOH shows higher compressive

strength which has also been reported in previous researches [8, 9]. The higher molarity of NaOH provides an excess of Na⁺ cation for Na/Al and Na/Si ratios. Higher Na⁺ cation readily dissolves with silica and alumina to form a thicker geopolymer binder which eventually provides improved binding property in developed product [8]. Table 4 shows the results of flexural strength in geopolymer composite samples. The results are in proportion to the compressive strength observed in similar samples. The compressive strength is imparted due to the reactivity of aluminosilicate with the alkaline solution to form a binder. The flexural strength is caused by the intermolecular bond formed due to the continuous polymerization. Also, the increased flexural strength might be due to the denser interfacial zone formed between aggregate and geopolymer paste as suggested by Singh *et al.* [25]. The highest flexural strength found was M12 mix having a value of approximately 4.31 MPa.

Table 4 Results of Compressive strength test and Flexural Strength test for different stages of curing

Mix No.	Compressive Strength (MPa)			Flexural Strength (MPa)		
	7 days	28 days	90 days	7 days	28 days	90 days
CTRL	27.2	35.14	45.66	2.42	3.77	4.11
M1	18.9	26.3	30.1	1.91	2.66	2.92
M2	20.2	27.2	33.24	2.21	2.76	3.17
M3	23.4	31.12	37.4	2.56	2.31	3.32
M4	19.1	24.55	31.42	1.89	2.56	2.98
M5	22.5	28.67	36.69	2.63	2.89	3.41
M6	24.8	31.33	39.31	2.91	2.33	3.11
M7	18.1	31.24	39.98	1.97	3.45	3.71
M8	26.3	34.97	44.77	2.52	3.52	4.46
M9	27.1	36.04	46.13	2.89	3.64	4.53
M10	22.9	30.45	38.98	2.55	3.59	4.06
M11	28.3	37.63	48.17	2.97	3.89	4.58
M12	31.2	41.49	53.11	3.33	4.31	4.97

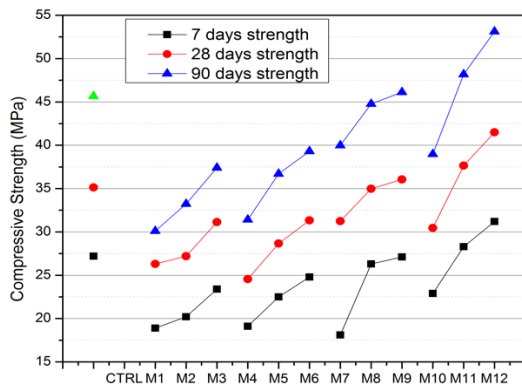


Figure 2 The analysis of compressive strength with respect to varying percentage of RHA/BA

3.2 Effect on Bulk Density and Porosity

Figure 3 shows the results obtained for bulk density and porosity test of the developed geopolymer composites. It is evident that specific gravity of the rice husk ash (value - 1.872) has played a vital role in the density of the material as the results shows lower density for rice husk ash incorporated geopolymer composite samples. Furthermore, it can be inferred that the increase in fly ash (value - 1.975) content results in a higher density due to the formation of geopolymer matrix. The highest value of density was observed in M12 mix having a blend of bottom ash (BA) and fly ash (FA) in 20:50 ratio. The density of the

mix having a higher molarity shows high density due to the higher degree of geopolymerization.

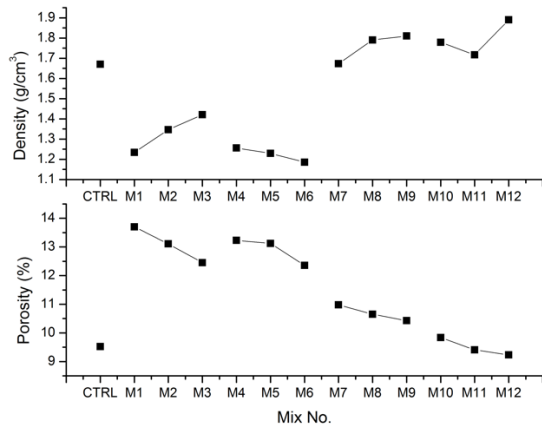


Figure 3 The results of bulk density and porosity of the developed geopolymer samples

During the lapse in curing days, the filling of pores within the geopolymer composite samples occurred due to the formation of geopolymer binder. The FA-RHA blended geopolymer had porosity values in the range of 14-12 %, whereas the FA-BA blend showed higher porosity with values ranging from 11-9%. The porosity plays a vital role in water transport properties of the building material. Thus, it is better to have less porous material to reduce the water percolation through the materials thus, improving the mechanical traits and durability performance of the proposed material.

3.2 Effects on Water Transport Properties

The results presented in Figure 4 shows the percentage of water absorbed by the developed geopolymer samples. The calculation is done for different stages i.e. 7, 28 and 90 days of curing period, to understand the influence of water absorption of geopolymer over the ageing of concrete. During the early stages, geopolymer shows a higher percentage of water absorption as compared to the geopolymer composite samples at later age. This might be due to the filling of the pore spaces in the materials due to formation of geopolymer matrix as discussed in the earlier section. The samples blended with FA-RHA shows a higher rate of water absorption due to the fibrous nature having hygroscopic nature of rice husk ash (RHA) which can be evident from the SEM image shown in Figure 1. The RHA samples shows the percentage of water absorption is in the range of 8.41-7 %, whereas samples having bottom ash (BA) is within the range of 6.89 – 6%. As the amount of Rice husk ash (RHA) increases, the water absorption also increases. The curing period has influence on the filling of the pores within the geopolymer matrix so it can be suggested that the geopolymer composite should be cured for a long duration to reduce the water absorption. The molarity of NaOH is also one of the

crucial factors to reduce the pore structure of the material, so the development of the geopolymer has to be done with a higher molarity of alkaline solution and by keeping in mind other factors influenced by molarity of NaOH.

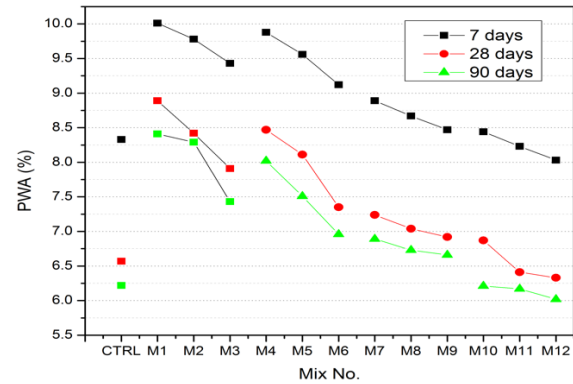


Figure 4 The results of percentage of water absorption for developed geopolymer

The velocity of water absorption at various stages are shown in Figure 5. From the aforementioned graph, it is evident that with the passing stage of geopolymer sample, there is gradual reduction in the velocity of water absorption. The RHA blended geopolymer was found to have a higher velocity of water absorption, with the increase in the capillary pores present due to the hygroscopic nature of RHA, the velocity increases. The velocity of water absorption ranges from 15×10^{-6} – 12×10^{-6} m/s^{1/2} for FA-RHA blended mix proportions, whereas FA-BA shows lower values in the range of 13×10^{-6} – 10×10^{-6} m/s^{1/2}. The FA-BA blend has lower values due to its fineness and reactivity, thus improving the microstructure of the developed material. It is of utmost importance to have a lower velocity of water absorption to reduce the percolation of water through the material. The velocity of water absorption mainly depends on the interconnectivity of pores within the microstructure of material. The service life of the structure can be prolonged if the percolation of water through it is minimal. This can be attained by developing the materials which have low velocity of water absorption. The discontinuous pores in the materials can be obtained by enhancing the densification of the microstructure by improving the fineness of the material and its reactivity.

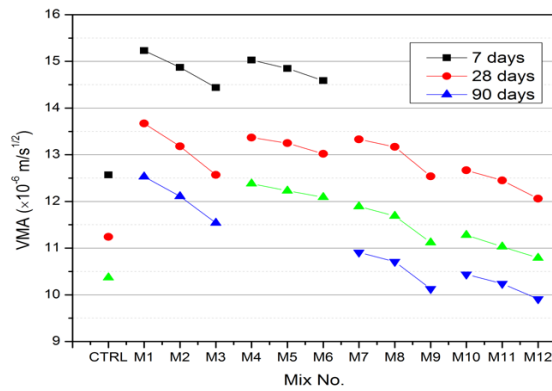


Figure 5 The results of velocity of water absorption for developed geopolymer

The coefficient of water absorption is considered as an evaluation of the permeability of water [24]. It is an evaluation of rate of uptake of the materials in a period of 1 hr. The developed samples show similar trends for coefficient of water absorption as compared to the velocity of water absorption, as both properties are regulated by the pore structure of the material. The determination of the coefficient of water absorption is very important to prevent the material susceptibility towards the durability attacks. The moisture movement with the pore structure is regulated by the interconnectivity of voids. As discussed earlier, rice husk has a fibrous nature with hygroscopic characteristics which promotes the capillary action for water to pass through the material at a much faster rate. The values for FA-RHA blended geopolymer composites are within the range of 4×10^{-10} – 2.75×10^{-10} m/s as compared to the much lower values of FA-BA within the range of 3.25×10^{-10} – 2.25×10^{-10} m/s. From the Figure 6, it is evident that the curing process of geopolymer material lowers the coefficient of water absorption, as the value seems to decrease with the curing time.

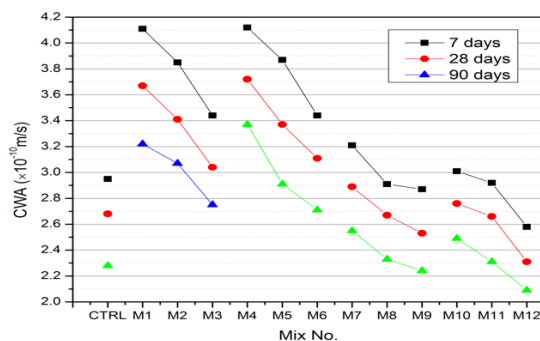


Figure 6 The coefficient of water absorption for developed geopolymer

4.0 CONCLUSION

The prototype geopolymer composite samples developed from the blend of two different waste materials i.e. rice husk ash and Bottom ash along with

fly as a primary binder, proves to be a sustainable alternative to the existing energy consumptive cement based materials. These developed geopolymer composite samples were analysed to understand their effects on mechanical and water transport performance. From the conducted research, the following conclusion can be drawn which has been explained below.

The blending of Bottom ash and Fly ash (BA-FA) shows better mechanical performance as compared to the control mix developed from fly ash. Also, the compressive strength value for BA-FA blended geopolymer composite was determined as 41.49 MPa. The blend of BA-FA was found to be superior to the blend of RHA-FA for development of geopolymer composite, due to the higher proportion of alumina-silica in the blend. The fibrous nature of rice husk has proved to be a problematic issue while dealing with the water transport performance. On the other hand, bottom ash proves beneficial for restricting the water transport through the material due to its dense pore structure. The higher molarity of NaOH has shown enhanced strength and resistance towards the water transport performance in both the RHA and BA blends. The utilization of the agro-industrial waste materials to develop sustainable geopolymer product will eventually provide an eco-friendly alternative to construction industry.

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