Evaluation of Pozzolanic activity and lime reactivity of fly ash, GGBS, mica powder and pumice as binders

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Abstract. This article presents the effect of the pozzolanic activity and lime reactivity of various siliceous materials, like fly ash, GGBS (Ground granulated blast furnace slag), mica and pumice powders. The pozzolanic activity is evaluated as per ASTM and lime reactivity test as per IS 1727. Based on the ASTM C311 tests, at 28 days GGBS, pumice and fly ash showed 71%,12% and 9% improvement in strength respectively in comparison with the control mix for 20% replacement of binder. Mica powder showed decrease of strength in comparison with the control mix. It has been observed that all binders satisfy the criteria of requirement of strength as per ASTM C311 activity index which is greater than 0.75. Based on the lime reactivity tests carried out as per IS 1727 carried out, maximum lime reactivity has been observed in case of GGBS (26%), followed by fly ash (25%), pumice (9%) and mica powder (8%) in comparison with the control mix with 20% replacement of hydrated lime.

Keywords: Pozzolana, strength activity index, lime reactivity test, fly ash, GGBS, mica powder

1 Introduction

Supplementary Cementitious Materials are pozzolans with a high amount of silica (SiO_2) and alumina (Al_2O_3). As a result, they react with calcium hydroxide (CH) at room temperature in the presence of water to produce calcium silicate hydrates (C-S-H) and calcium aluminosilicate hydrates (C-A-H). The C-S-H and C-A-H both bind the particles and provide mortar and concrete more strength. Often used SCMs include fly ash, silica fume, metakaolin, powdered granulated blast furnace slag, and diatomaceous earth. Other SCMs include rice husk ash (RHA) and sugar cane bagasse ash, which are derived from agricultural byproducts[1-6]. The widespread availability of SCMs, their advantages for the economy and environment, and the advancements in cement technology today are what motivate their usage. Donatello et. al [7] compared pozzolanic activity using fratinni test, saturated lime test

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and strength activity test of metakaolin, silica fume, coal fly ash, incinerated sewage sludge ash. It was observed that saturated lime test is not a reliable test because it does not correlate with other tests that could be explained by the different activator to pozzolan ratio. The researchers suggested usage of the frattini test and SAI methods combining with an independent determination of $Ca(OH)_2$ content (thermal or diffraction methods) for the assessment of the pozzolanic activity. Elyasigorji et al [8] reviewed the available literature related to direct and indirect pozzolanic reactivity test methods that have been utilized over the years. While indirect methods evaluate the changes in the specimen's physical qualities brought about by pozzolanic reactions, direct methods measure the amount of calcium hydroxide consumed. The frattini test, electrical conductivity test, and pH test are regarded as the fastest ways to evaluate pozzolanic materials, according to the authors. Tests for pH and electrical conductivity are also simple to conduct. Other approaches, however, require more time. Mboya et al [9] carried tests on scoria, pumice and rice husk ash according to ASTM C618 criteria and tested these binders to evaluate their potential as supplementary cementitious materials. The researchers have reported that scoria and rice husk ash fulfilled the specified minimum of 75% of pozzolanic activity index in 7 days, whereas pumice has achieved minimum pozzolanic index in 28 days.Kramar & Ducman [10] carried out research using a variety of direct and indirect techniques to evaluate the pozzolanic activity of five distinct types of ash. After curing for 7, 28, and 90 days, ash pozzolanicity was evaluated using differential thermal and thermogravimetric analyses (DTA/TG) in addition to the Frattini test and the calculation of the strength activity index (SAI). The findings demonstrated that the ashes displayed varying degrees of pozzolanic activity in terms of the amount of lime with which they could chemically interact as well as reaction kinetics, owing to their distinct mineralogical, chemical, and physical properties. Additionally, the results demonstrated that ashes with greater BET surface areas had a faster pozzolanic reaction rate, but those with higher reactive SiO_2 content were more reactive.

The use of various reactive pozzolanas as supplementary cementitious materials is fast growing in the development of more durable and high-performance construction concreting materials. [11,12]. Slag based binder, fly ash, silica fume, rice husk ash (RHA), metakaolin, pond ash, palm oil fly ash (POFA), etc., are being used as alternative mineral admixtures to cement. Pourkhorshidi et al [13] investigated the techniques and accepted practices for assessing a material's pozzolanicity. Pozzolana standards and specifications fall into two primary categories: direct methods and indirect methods. Using direct methods the existence of $Ca (OH)_2$ and its subsequent decrease in abundance over time as the pozzolanic reaction advances is observed. Certain mineralogical (like amorphous or crystalline minerals) and chemical (like insoluble residue) characteristics of the pozzolan are measured (like thermo-gravimetry) and traced (like X-ray diffraction, XRD) to offer a foundation for assessing the pozzolana quality. Agarwal studied the accelerated pozzolanic activity of a number of siliceous materials, including metakaolin, quartz, precipitated silica, fly ash, silica fume, and rice husk ash (RHA; different fineness and carbon content), has been investigated. Cubes cured in water for seven and twenty-eight days have been compared to the compressive strength of accelerated tests. The example of RHA (<45 μ) has shown the highest activity, followed by quartz and silica fume. Sand replaced 10% of cement has demonstrated an accelerated pozzolanic index of 92%, but the ASTM requires 85% for silica fume to be used as a mineral additive in order to assess pozzolana. Xia et al [14] conducted a study on concrete samples produced using 30% FA, 30% fine tuff (FT), 30% fine volcanic slag (FVS), or 10% FA and 20% FVS to replace cement. The researchers found out the concrete made with 30%

FT showed the lowest mechanical strength. Although the best mechanical properties were obtained with a cement replacement of 20% natural FVS and 10% FA, satisfactory performance was also achieved for the concrete with 30% FVS as cement replacement.

The objective of this study is to evaluate the pozzolanic activity of binders by ASTM C618 [15] and ASTM C 311 [16] and lime reactivity as per IS 1727 [17]. As discussed above, it is apparent that one or two or combination of test methods are to be carried out to assess the pozzolanic activity of the binders rather than employing any one test method. As such, the pozzolanic activity of binders fly ash, GGBS, mica powder and pumice powder has been determined for their use in geopolymer mortar either individually or in combination of one or two binders.

2 Experimental

2.1 Materials

2.1.1 Cement:

Ordinary Portland Cement 43 grade is used in the current study. The properties of cement are shown in table 1

Table 1. Properties of Cement

Binder	SiO_2 [% by wt.]	Fe_2O_3 [% by wt.]	Al_2O_3 [% by wt.]	CaO [% by wt.]	MgO [% by wt.]	SO_3 [% by wt.]	Na_2O [% by wt.]	K_2O [% by wt.]	LOI [% by wt.]
Cement	20.16	3.99	4.12	61.91	2.82	2.06	0.41	-	1.28

2.1.2 Fine aggregate:

Manufactured sand is sourced from local supplier. The fine aggregate confirms to gradation requirements of IS 383-1979. The fine aggregate falls in zone II and its fineness modulus is 2.9.

2.1.3 Fly ash:

It is obtained when pulverised coal is burnt in thermal plants. Coal is burnt to heat water and convert it into steam, which runs the turbines. Fly ash is pozzolanic in nature due to the presence of high amount of amorphous silica. Silica, alumina, calcium, iron oxides make up nearly 90%-95% of fly ash. Further, fly ash is classified based on percentage of CaO present. Fly ash is termed as low calcium fly ash (Class F < 7% CaO) which is pozzolanic in nature and high calcium fly ash (Class C > 20% CaO) which is cementitious. The specific gravity of fly ash is 2.24 and specific surface area of 362 m^2/kg.

2.1.4 GGBS (Ground granulated blast slag):

Ground granulated blast furnace slag This is bye product generated during iron extraction in blast furnace. It is non- metallic. It consists mainly of silicates, alumina and calcium oxide. GGBFS – Granulated blast furnace slag is obtained when molten blast furnace slag is rapidly chilled in cold water. It is granular in appearance. When GGBFS is ground to fineness of cement it is called GGBS which is cementitious in nature. The specific gravity is 2.76 and specific surface area of 410m^2/kg

2.1.5 Mica powder:

Muscovite mica powder is a mineral name given to a group of minerals that are physically and chemically similar. They are all silicate minerals, usually known as sheet silicates because they form in distinct layers. They are fairly light and relatively soft, and the sheets and flakes are flexible. It is a grey/white coloured Potassium-Aluminium phyllosilicate mineral with the chemical formula $(KF)_2(Al_2O_3)3(SiO_2)6(H_2O)$. Muscovite is the most common mineral of the mica family. It is an important rock-forming mineral present in igneous, metamorphic, and sedimentary rocks. Mica's are used in a variety of products ranging from drywalls, paints, fillers, especially in parts for automobiles, roofing and shingles, electronics etc. The specific gravity is 2.80.

2.1.6 Pumice:

Pumice is an extrusive volcanic rock material, produced when lava, which is full of water and gases, is ejected on the earth surface by volcanic action. It has a pale color, is very vesicular, has a low iron and magnesium content, and a high silica and alumina content. Pumice from lavas with high silica content are white, whereas those from lavas with intermediate silica content are more uncommon and are typically yellow or brown. While most of the pumice is made of glass, there are occasionally tiny crystals of other minerals. The specific gravity of pumice is 2.70 and specific surface area is 480m^2/kg.

2.1.7 Hydrated lime:

Hydrated lime is a type of dry powder made from limestone. It is made by combining quick lime with water to convert oxides to hydroxides. Hydrated lime is most commonly used to create plasters and mortars when mixed with cement, sand, or water. Its chemical name is calcium hydroxide $Ca(OH)_2$. It consists of about 90 % of Ca (OH)2, magnesium MgO 0.8 % and insoluble acid as SiO_2 0.50%. As per supplier information, the density of hydrated lime is 2.31 gm/cm$^{3.}$ and pH value of is 12.4. Refer to the chemical composition of materials in Table 2.

Table 2. Chemical properties of cement and various binders

Binder	SiO_2 [% by wt.]	Fe_2O_3 [% by wt.]	Al_2O_3 [% by wt.]	CaO [% by wt.]	MgO [% by wt.]	SO_3 [% by wt.]	Na_2O [% by wt.]	K_2O [% by wt.]	LOI [% by wt.]
	Min 70%					Max – 4%			Max - 10%
Cement	20.16	3.99	4.12	61.91	2.82	2.06	0.41	-	1.28
Fly Ash	49.13	-	34.21	5.22	3.07	-	3.17	5.2	0.27
GGBS	28.82	-	16.44	48.82	5.49	-	-	0.44	2.2
Mica Powder	46.27	-	39.87	-	-	-	1.47	12.39	4
Pumice	69.37		22.74				2.11	5.78	5

Flyash

Cement

GGBS

Mica Powder

Pumice powder

Hydrated Lime

Fig 1: Materials

3 Test Methodology

3.1 Pozzolanic activity index with Portland cement

The procedure used is based on ASTM C311. The control mixture was prepared with 500 gm of 43 grade cement with 1375gm of graded sand and 242 ml of water and the various test mixes for fly ash, GGBS, mica powder and pumice were prepared with 10%, 20% & 30%

replacement of cement with respective binders. 6 cubes are prepared for each mix for testing at 7 days & 28 days.The mix proportions are shown in table 3. The identification of the mixes are R0 is control mixture, RF10 is test mix with 10% replacement of fly ash, RF 20 is 20% replacement with fly ash and so on. RG10 is test mix with 10% replacement of GGBS. Similarly, RM10 and RP10 is 10% replacement of mica powder and pumice respectively. The mortar mixes were prepared using pan mixer. Cubes of size 50mmx50mm x 50mm were cast. In total 78 cubes were cast for testing at 7 day and 28 day compressive strength. After 24 hours of initial curing at room temperature the cubes were stored and placed for testing at 7 days and 28 days. The compressive strength of the mortar cubes after 7 days and 28 days of demoulding of control and test mixture was determined as per below equation. The results of tests are shown in table 5.

Fig 2. Cubes for testing

Pozzolanic activity index with Portland cement = A/B x 100

(where, A is the average compressive strength of test mix cubes containing pozzolana (MPa), and B is the average compressive strength of control mix cubes mix without pozzolana (MPa).

Table 3. Mix proportions of materials for determination of pozzolanic activity index.

S.No	Mix Id	Cement [gm]	Pozzolanic Material [gm]	Sand [gm]	Water [ml]
1	R0	500	0	1375	242
2	RF10	450	50	1375	242
3	RF20	400	100	1375	242
4	RF30	350	150	1375	242
5	RG10	450	50	1375	242
6	RG20	400	100	1375	242
7	RG30	350	150	1375	242
8	RM10	450	50	1375	242
9	RM20	400	100	1375	242
10	RM30	350	150	1375	242
11	RP10	450	50	1375	242
12	RP20	400	100	1375	242
13	RP30	350	150	1375	242

3.2 Lime reactivity test as per IS 1727 (2004)

In this method, a volume based mix design is used to keep the same volume of the binder in each mix. The materials of the standard test mortar shall be lime: pozzolana: sand in proportion of 1: 2M: 9 by weight. (where 'M' indicates the ratio of specific gravities of pozzolana to lime). The mortars are cast and kept in RH saturated conditions and at 27 C until 2 days, after which the samples are demoulded and further cured at 90–100% RH and 50^0 C. The compressive strength of the mortar cubes is measured after 10 days of curing which should achieve a minimum strength of 4.5 MPa at 10 days. The strength data are taken as indication of the reactivity of the pozzolana.

The identification of the mixes RF0 is control mix with flyash, RF10 is test mix with 10% replacement of fly ash with hydrated lime, RF 20 is 20% replacement with hydrated lime and so on. RG10 is test mix with 10% replacement of GGBS with hydrated lime. Similarly, RM10 and RP 10 is 10% replacement of mica powder and pumice respectively. The mix proportions are shown in table 4. Cubes of size 50mm x 50mm x 50mm were cast. 6 cubes are prepared for each mix. In total 144 cubes are cast for testing at 7 days, 10 days & 28 days. The compressive strength of test specimens are carried out using a compression testing machine. at a rate of 35 kg/cm^2/min. The results of the testing are shown in table 6.

Table 4. Mix proportions of materials for determination of lime reactivity test

S.No	Mix Id	Hydrated Lime [gm]	Pozzolanic Material [gm]	Sand [gm]	Water [ml]
1	RF0	150	300	1350	242
2	RF10	165	285	1350	242
3	RF20	180	270	1350	242
4	RF30	195	255	1350	242
5	RG0	150	370	1350	242
6	RG10	165	355	1350	242
7	RG20	180	340	1350	242
8	RG30	195	325	1350	242
9	RM0	150	375	1350	242
10	RM10	165	360	1350	242
11	RM20	180	345	1350	242
12	RM30	195	330	1350	242
13	RP0	150	385	1350	242
14	RP10	165	370	1350	242
15	RP20	180	355	1350	242
16	RP30	195	340	1350	242

4 Results and discussion

Numerous techniques have been developed to assess the pozzolanic activity of different pozzolanas based on their mechanical, chemical, and physical characteristics. In the present study, the pozzolanic activity of various pozzolanic materials has been determined based on chemical composition based on ASTM C618, compressive strength based on ASTM C311 and lime reactivity test based on IS 1727.

4.1 Oxide Composition

As can be seen from the table 2 chemical composition. The binders consisted predominantly of Al_2O_3 (16 - 40%) and SiO_2 (28 - 70%). All the binders except GGBS met the ASTM standard requirement of a minimum of 70.0% for SiO_2+Al_2O_3+Fe_2O_3. Additionally, the SO3 content of all the binders was negligible, below 0.1 %, which was well below the maximum limit of 4%.

The combined percentage of SiO_2+Al_2O_3+Fe_2O_3 for fly ash is 83.34%, mica powder is 86.14% and pumice is 92.11% which satisfy the requirements of the standard. Contrary to these materials, the combined percentage of SiO_2+Al_2O_3+Fe_2O_3 for GGBS is 45.26%. However, GGBS has high percentage of CaO (48%). As GGBS is latent hydraulic material, GGBS can improve the reactivity during cement hydration and as a result it can be used widely as supplementary cement material. [18]

4.2 Pozzolanic activity index with Portland cement

The 7 day and 28-day strength results are shown in table 10. The control mortar strength was 20.4 MPa at 7 days and 26.40 MPa at 28 days. At 7 days, GGBS, pumice and fly ash showed 64% ,7% & 7 % improvement in strength respectively in comparison with the control mix with 20% replacement of cement. Mica powder showed decrease of 21% in strength compared to control mortar. After 28 days, there is improvement of pozzolanic activity in all the binders. GGBS, pumice and fly ash showed 71%, 12% & 9 % improvement in strength respectively in comparison with the control mix with 20% replacement with binder. Mica powder showed decrease of 19% in strength compared to control mortar. it may be concluded that GGBS show high pozzolanic activity, whereas pumice and fly ash show moderate pozzolanic activity and mica powder show slight pozzolanic activity.

One of the most crucial characteristics influencing pozzolanic activity is fly ash fineness. Fly ash with a greater median particle size may be more reactive than predicted by the model, according to Watt and Thorne [19]. This had to do with the larger particles' spongy shape. The bigger particle becomes perforated during the reaction with lime, as demonstrated by the microscopic inspection. It has been noted that in the case of fly ash, the carbon content has no discernible effect on the compressive strength ratio of the pozzolanic activity index [20,21]. Experiments show that more than 20% of active chemicals, such as SiO_2 and Al_2O_3, are present in FA. A study on fly ash [22] made it abundantly evident that the material had a strong pozzolanic activity. Based on the studies that were conducted, it was determined that: in cement composites with the addition of FA, the hydration reaction is primarily responsible for the development of the material structure up until the third day of curing; the first signs of the pozzolanic reaction on FA grains appear between the seventh and fourteenth day after sample preparation; however, between the fourteenth and twenty-eighth day, there is a noticeable homogenization of the structure of the cement composite with the addition of FA, resulting from the transformation of disordered phases into compact and homogeneous forms and filling in the composite of porous places with pozzolanic reaction products.

In case of mica powder, the lower value of strength could be due to very high demand of water content. Mica powder being inert, there has been a decrease in the strength development owing to the dilution effect. However, it may be noted that it still satisfies the requirement of 75% of control mix to be considered as pozzolanic material.

As seen from the figure 3, all binders satisfy the criteria of requirement of strength as per ASTM standard pozzolana activity index greater than 0.75.

Table 5: Compressive strength of binders for 7 day & 28 day.

S. No.	Mix Id	7 Day compressive strength [MPa]	28 Day compressive strength [MPa]
1	R0	20.40	26.40
2	RF10	26.80	35.20
3	RF20	22.00	28.80
4	RF30	24.00	26.00
5	RG10	32.00	41.60
6	RG20	33.60	45.20
7	RG30	31.20	38.80
8	RM10	18.00	24.00
9	RM20	16.00	21.20
10	RM30	12.40	16.80
11	RP10	26.00	36.40
12	RP20	22.00	29.60
13	RP30	20.40	27.20

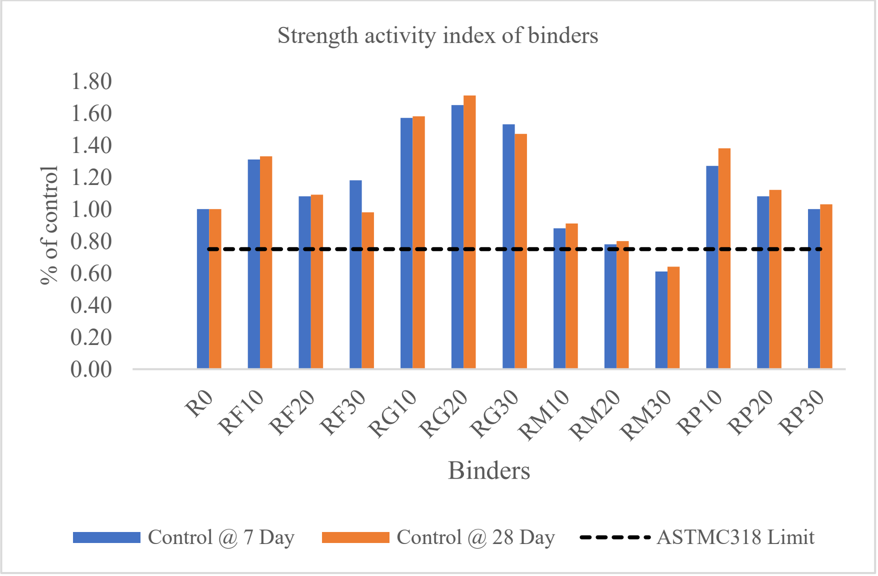

Fig.3. Strength activity index of binders after 7 and 28 days.

Results are averages of cube strength and expressed as % of control. Sample strengths, which were 20.4 MPa after 7 days and 26.40 MPa after 28 days

4.3 Lime Reactivity

The compressive strength results at 7 days,10 days & 28 days for the various materials are shown in table 6.

Table 6. Compressive Strength of Mortars with Various Proportions of Pozzolanic Material

Mix Id	Compressive Strength [MPa]		
	7 days	**10 days**	**28 days**
RF0	2.67	3.10	4.49
RF10	3.06	3.41	4.59
RF20	3.80	3.88	4.90
RF30	2.86	3.27	4.69
RG0	2.80	3.61	4.80
RG10	3.59	4.10	4.90
RG20	4.10	4.55	5.20
RG30	3.20	3.80	4.69
RM0	3.00	3.41	4.37
RM10	3.37	4.08	4.59
RM20	3.20	3.67	4.39
RM30	3.16	3.63	4.08
RP0	2.80	3.80	4.49
RP10	3.41	4.10	4.59
RP20	3.80	4.16	4.65
RP30	3.10	4.04	4.04

The 7 days,10 days and 28-day strength results are shown in table 7. The control mortar strength at 10 days with various binders fly ash, GGBS, Mica powder and pumice powder is 3.10 MPa,3.61MPa,3.41MPa and 3.80 MPa and at 28 days the strength is 4.49 MPa,4.80 MPa, 4.37 MPa and 4.49 MPa respectively.

At 10 days, fly ash with 20% replacement of hydrated lime at 10 days has shown improvement of 25% than the control mix and 9% increase than the control mix at 28 days.

GGBS with 20% hydrated lime at 10 days has shown improvement of 26% than the control mix and 8% than the control mix for 28 days. GGBS showed improvement in strength up to 20% replacement with hydrated lime. Mica powder with 20% hydrated lime at 10 days has shown improvement of 8% than the control mix and, it has shown has shown marginal improvement of 1% than the control mix at 28 days. Mica powder showed improvement in strength up to 10% replacement with hydrated lime and it decreased with 20% replacement and beyond. Pumice with replacement of 20% hydrated lime has shown improvement of 9% than the control mix. For 28 days, it has shown has shown improvement of 3.5% than the control mix. Similar to fly ash and GGBS, pumice showed improvement in strength up to 20% replacement with hydrated lime.

In general, all binders showed decrease in compressive strength at 30% replacement with hydrated lime. GGBS, mica powder and pumice showed 2%, 7 % and 10% decrease in compressive strength with reference to the control mix.

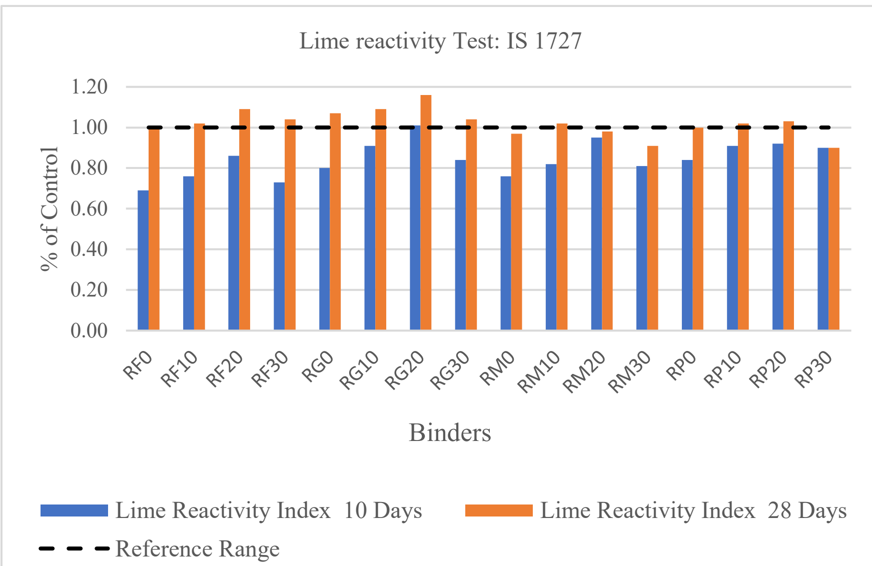

Fig.4 Lime reactivity index of binders for 10 days and 28 days. Results are averages of cube strength and expressed as % of control. Not less than 4.5 MPa after 10 days

As seen from the figure 4, GGBS has shown pozzolanic activity based on the lime reactivity test for 10 days whereas other binders fly ash, mica powder and pumice have shown less pozzolanic activity than the limit of 4.5 MPa. When the samples are tested at 28 days, fly ash, GGBS, mica powder and pumice have shown improved pozzolanic activity and satisfied the IS requirements.

4. Conclusion

Based on the research carried out the following conclusions can be drawn.

- GGBS has shown maximum pozzolanic activity of 71% followed by pumice which has shown 12% and fly ash shown 9% improvement in comparison with the control mortar specimen.

- Contrary to the above, mica powder showed 19% decrease in the strength than control mortar specimen. However, it still satisfies the requirement of 75% of the strength of control mortar specimen.
- Based on IS standard, lime reactivity test, for 10-day period, GGBS and fly ash showed maximum pozzolanic activity of 26% and 25% respectively with pumice and mica powder showing 5% than the control mortar specimen. For 28-day period, all binders satisfy the criteria of achievement of strength not less than 4.5MPa.
- From the above, it can be concluded that mica powder and pumice can be used as binder material in geopolymer mortar.

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