



AN INVESTIGATION ON SERVICE LIFE & MgO USE IN GREEN CEMENT

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Abstract: The emission of Carbon dioxide is no exception in the construction industry, but rather extensive. Establishing an alternative Cementing material adds an advantage in addressing the subject. As part of this, attempts to produce Cement through the activation of alkalis, which is both eco-friendly, and viable is the major add-on. Through the literature available, it is evident that the preparation of Alkali Cement is advantageous in all aspects, but the best suitable combination of chemicals is still the concern. Moreover, there are not many conclusions stated regarding the storage period or the service life of the attained Alkali Cements. Thus, this study aims to determine the storage period of the Alkali/ Green Cements produced through the alkaline medium created using Sodium Silicate and Magnesium Oxide (Na_2SiO_3 & MgO), while disentangling the role of MgO . Three different weight combinations of Na_2SiO_3 and MgO were ventured, using Fly Ash (FA) and Ground Granulated Blast Furnace Slag (GGBS) by fixing the GGBS content to 30%. X-Ray Fluorescence (XRF) and Heat of hydration (HOH) were adopted to understand the performance variation of the Cement because of the age. Whereas, tests like Fineness by Blaine's Permeability, Soundness, Standard Consistency, Initial & Final Setting time (IST & FST), Compressive strength, and Density were conducted along with XRF and HOH to derive the ideal mix case. Further, it is undeniable that the use of MgO as an activator, in just the right amount can help enhance performance in terms of strength and shrinkage. Out of the combination of Alkalis used, the optimum percent of MgO is found out to be 10% when combined with 10% of Na_2SiO_3 . A compressive strength of 33MPa was attained, by mere ambient curing. Through the study, it is observed that Alkali Cement has a minimum service life of 1 month.

Index Terms - Carbon dioxide, Alkali Cement, Fly ash, GGBS, MgO , Na_2SiO_3 , XRF.

I. INTRODUCTION

In view of the extensive use of Cement, the CO_2 emitted is beyond control. The current priority among the researchers is to address this aspect. Geopolymer concrete is the outcome of one such studies, with the Geopolymerization given by Davidovits, 1994. Geopolymer concrete (GPC) is produced majorly by using the Fly Ash and/or GGBS as source material and chemicals for activation of these admixtures. GPC has even reached the extent to produce strengths greater than Ordinary Portland Cement (OPC), done by Annapurna, 2019 at different dosages of GGBS. However, the drawback of GPC is that excessive supervision is required to deal with the alkali activators, such as Sodium/ Potassium Hydroxide (NaOH / KOH), Sodium/ Potassium Silicate (Na_2SiO_3 / K_2SiO_3), Sodium Carbonate (Na_2CO_3) and many more as per IS 17452-2020. Further hazardous nature of alkaline solution is a challenging issue. Alkali Cement can be the best solution for these issues, through which a ready mix can be prepared without any groundwork as described by Abdel, 2016.

NaOH and Na_2SiO_3 are the combination of the alkali activators widely known for their compatibility and performance (Rakhimova, 2022 and Annapurna, 2016). This compatibility is because of their ability to make the FA/ GGBS destabilize to participate in the Geopolymerization process resulting into a binder as explained by Faris, 2017. NaOH being absorptive in nature, the need for the study of a better alternative in combination with Na_2SiO_3 or otherwise becomes enormous as studied by Kamal, 2021. This also explains the need for the study of the service life of the Alkali Cements obtained, as the chemicals used are generally hygroscopic in nature as suggested by Faris, 2018. In addition, with the inherent advantages like the reduction in the use of Cement, ambient curing conditions, reduction in energy consumption enhances the scope for Alkali Cement (Arash Nivkar, 2022 and Singh, 2020). This Alkali activation when adopted into Cement can make it highly desirable in the days to come, once the morphology and chemical contents are balanced optimistically. Hence, the challenge behind preparation of Alkali Cement is to identify the better alternative activators.

II. ALKALI ACTIVATION METHOD

The Alkali activation is ensured to take place by adopting either of the two methods, one being a wet process and other being the dry one. Though the wet approach is assumed to be more effective given the homogeneity that can be attained through the liquid phase, still the process of drying at elevated temperatures which is further to be followed by proper crushing cannot be ignored. Thus, the energy consumption and need for oven is high in the case of the liquid approach. Where as in the dry method, the oven drying and pulverising can be skipped as long as all the materials are ensured to be grounded and milled into each other thoroughly,

so that mechanical energy gets converted into chemical energy guaranteeing a proper inter mixing and a sure gateway for the formation of Geopolymer structure (Faris, 2017). Hence, the dry approach is used in the present study.

III. EXPERIMENTAL APPROACH

Experimental work is carried out in different phases, consisting of sub stages as explained in the below headings.

3.1 Phase - 1 Investigation

Primarily, in order to study the compatibility of the attained Fly Ash and GGBS with the chemical activators a minor study was taken up using manual mixing and grinding before using them directly in the ball mill. As part of this, 9 mix combinations were studied as shown in the figure 3.1. As soon as the raw materials were procured, the study was taken forward in the following 3 stages.

3.1.1 Stage-1

In this stage, all the ingredients were mixed manually. The detailed combination of activators is shown in Figure 3.1.

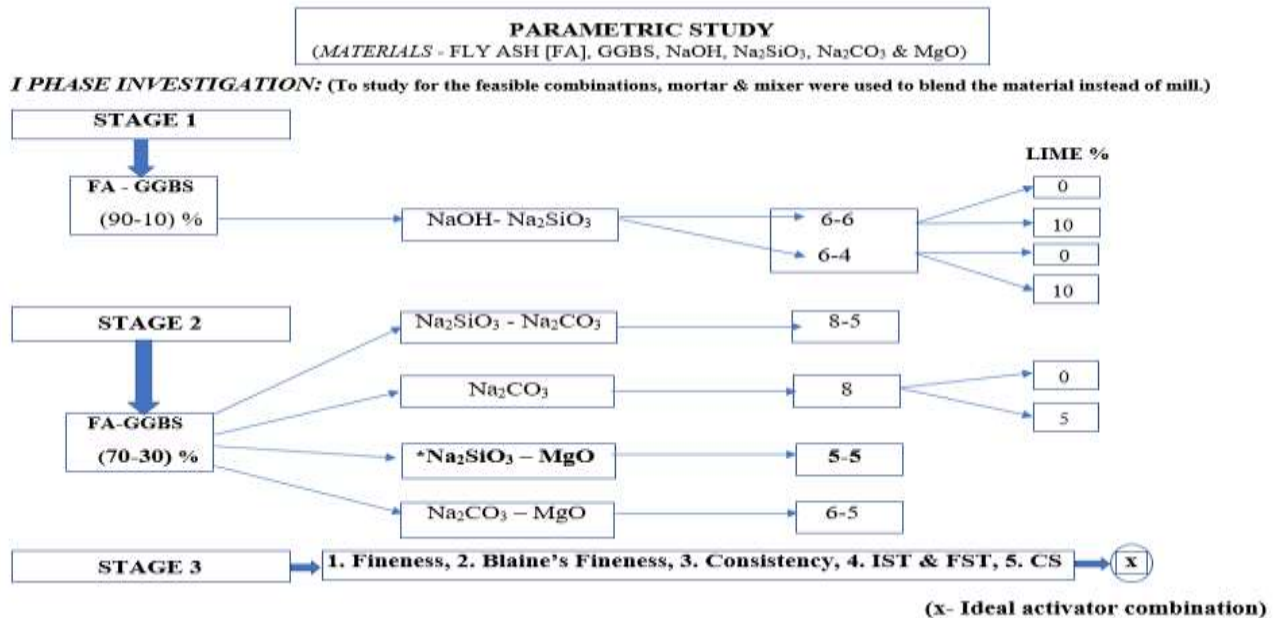


Figure 3.1 Phase-1 Investigation

To start with, the FA: GGBS content is kept as 90:10 for the basic activator combination of NaOH and Na₂SiO₃. The NaOH-Na₂SiO₃ is varied as 6-6 and 6-4, each case without lime initially and then in the presence of 10% of lime. NaOH, which was available in pallet form, was pounded manually in mortar as shown in the Figure 3.2. Therefore, four combinations were obtained.



Figure 3.2. Manually grounded NaOH.

3.1.2 Stage-2

Using 10% of GGBS did not seem to be advantageous as FA was slow in reacting. However, this was proved in stage-3 through the compressive strength test. Hence, in this stage, four different activator combinations were tried by upgrading the FA: GGBS content to 70:30. The activator cases being Na₂SiO₃-Na₂CO₃ (8-5) %, Na₂CO₃ (8%)- without lime and with 5% lime, Na₂SiO₃-MgO (5-5)% and Na₂CO₃-MgO (6-5)%, as described in table-4. All these mixes were attained using a mixer as shown in figure 3.3. Thus five more combinations were prepared.



Figure 3.3. Mixing of the material using a mixer.

3.1.3 Stage-3

As part of this stage, all the 9 attained combinations were evaluated through the physical tests such as Fineness, Blaine’s Air Permeability of Fineness, Standard Consistency, Initial and Final Setting time (IST & FST) and Compressive strength as given in table -3.1. Therefore, 45 tests were performed in total.

Table - 3.1: Physical tests carried.

STAGE - 3	
Physical Analysis	Part -1 Fineness (dry sieving)
	Part -2 Fineness (Blaine's Permeability)
	Part -4 Standard Consistency
	Part -5 IST & FST
	Part -6 Compressive strength

3.2 Phase - 2 Investigations

II PHASE INVESTIGATION: (Performed at NCCBM)

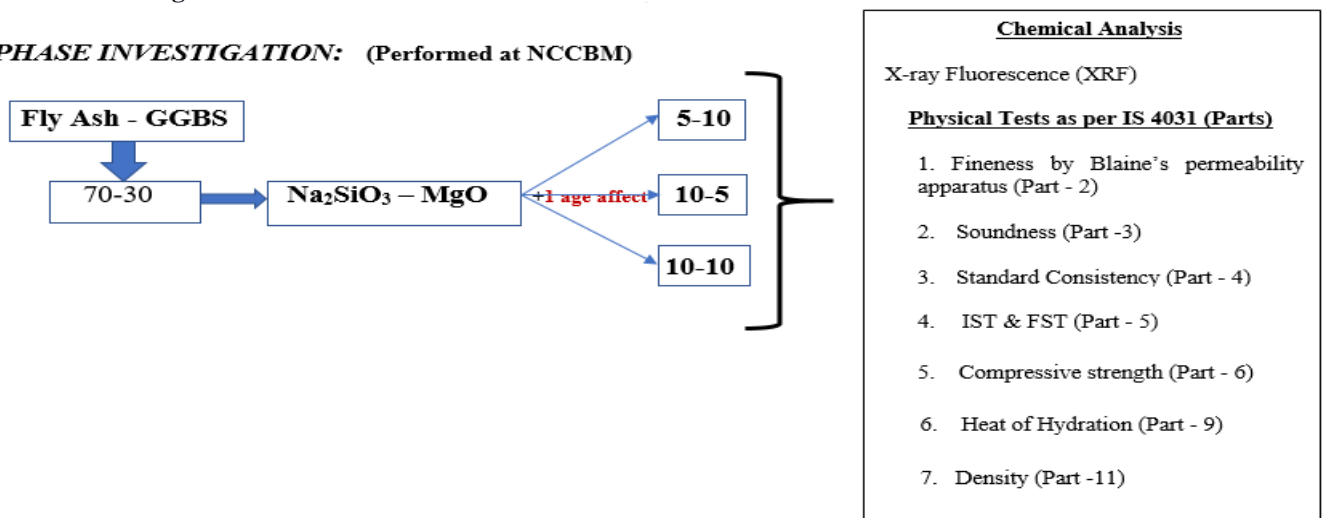


Figure 3.4 Phase - 2 Investigation

As part of this investigation, 3 weight variations were taken up for Na_2SiO_3 & MgO (S-M) combination as given in figure 3.4. Namely, (5-10)%, (10-5)% and (10-10)% and named as M21, M22 and M23 respectively as shown in table -2. Apart from these, the M22 is repeated after 1 month and named as M22 New – M22N. A steel ball mill of 10 Kg capacity was used with 25 kg weighing steel balls as shown in the figure 3.5 (a) and (b). Initially the FA and GGBS were grounded for 90 minutes at which a Blaine’s fineness of $400 \text{ m}^2/\text{kg}$ or greater fineness was attained, after which the chemicals were added and milled for another 12-15 minutes. The mill was operated at the rate of 46 rpm and a 6kg quantity of each mix is derived. The temperature was also checked as shown in figure 3.5(c), in order to ensure that there was no excessive heat of evolution; it was noticed to be at room temperature around 23°C . The mixes thus obtained were packed in zip lock bags as shown in the figure 3.5 (d). The whole ball milling process was performed at NCCBM, Gachibowli.



(a) Steel Ball mill with the balls

(b) Inside the ball mill



(c) Temperature check

(d) Attained mixes packed

Figure 3.5. Ball milling process

Table - 3.2 Mixes arrived with their composition.

Cement ID	Mix combination	Activator (% of FA replaced)	FA % (GGBS =30% Fixed)
M21	Na ₂ SiO ₃ & MgO (S-M)	5&10	55
M22		10 &5	55
M22N (To study age effect)		10 &5	55
M23		10 &10	50

The obtained mixes, as shown in table -3.2 were studied in detail through several chemical and physical tests as given in table -3.3.

Table - 3.3 Tests conducted

❖ Chemical Analysis
X-Ray Fluorescence (XRF)
❖ Proposed (Physical analysis) as per IS 4031 (Parts)
Part -1 Fineness (dry sieving)
Part -2 Fineness (Blaine's Permeability)
Part -3 Soundness
Part -4 Consistency
Part -5 IST & FST
Part -6 Compressive strength
Part -9 Heat Of Hydration (HOH)
Part -11 Density

Table – 3.3 highlights all the tests that were carried out on mixes M21, M22 and M33. The XRF and HOH were the tests performed on the M22N mix to compare the results with M22 mix case.

IV. RESULTS AND DISCUSSION

The results attained through the different phases are given below with appropriate explanation for each test performed on the mix cases obtained as part of the study carried out.

4.1 Phase – 1 Investigation

Table- 4.1 Mixes composition and tests conducted

S.No	GGBS in %	Na ₂ SiO ₃ in %	NaOH in %	Na ₂ CO ₃ in %	MgO in %	Lime in %	Standard Consistency (%)	IST (min)	FST (min)	SSA (m ² /g)	sieve residue %	CS (N/mm ²)
I	10.0	6.0	6.0	-	-	-	24	-	-	-	-	0
II	10.0	6.0	6.0	-	-	10	28.5	-	-	-	-	<2
III	10.0	6.0	4.0	-	-	-	23.5	58	-	-	-	<2
IV	10.0	6.0	4.0	-	-	10	30	1	-	-	-	<2
V	30	-	-	8	-	-	27	98	174	118.6	68	-
VI	30	5	-	-	5	-	24.5	216	-	18.96	93	17.05
VII	30	-	-	8	-	5	31	1	~220	77.96	1.677	4.01
VIII	30	-	-	8	-	-	25.25	48	170	79.2	11.11	2.01
IX	30	-	-	6	5	-	29	18	119	57.8	13.33	-

Table 4.1 displayed the mix combinations, their activator dosages along with the test results of each mix case. Through this phase, it was evident that at 5% dosage of both Na₂SiO₃ & MgO was noticed to give 17.05 MPa at 28 days by mere use of a mixer. 30% replacement of FA by GGBS was also proved to be beneficial compared to 10% GGBS. Hence, the same combination is focussed on in the further study.

4.2 Phase – 2 Investigation

As part of this phase, a discussion on the all the different physical and chemical tests performed on each mix derived is carried out comprehensively. Physical tests were performed as per IS 4031 on the three mix cases. To unlock the role of MgO, the chemical analysis has helped to understand the behaviour and performance of the mixes in case of the physical tests performed.

4.2.1 X-Ray Fluorescence (XRF)

Table- 4.2 XRF Analysis

Mix ID	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	Ka	Kb	HM	Ca/Al	Ca/Si	Na/Al	Si/Al	SiO ₂ /Na ₂ O
M-21	47.3	17.9	3.1	11.5	12.1	0.87	1.65	0.32	0.38	0.36	0.88	0.6	0.2	0.1	2.6	28.6
M-22	51.1	18.7	3.2	11.5	7.4	0.88	3.41	0.31	0.37	0.27	0.74	0.6	0.2	0.2	2.7	15.0
M22N	52.3	18.9	3.7	10.6	7.2	0.88	3.30	0.28	0.36	0.25	0.70	0.6	0.2	0.2	2.8	15.8
M-23	50.3	19.6	4.0	11.5	2.0	0.91	8.24	0.30	0.39	0.19	0.66	0.6	0.2	0.4	2.6	6.1

Table 4.2 represents the XRF analysis carried out on the mix cases with respect to their chemical composition. For the geopolymer structure to form the Si/Al ratio must be maintained in the range 1-3 ideally (Cheng, 2013 and Annapurna, 2019) this was satisfied for all mix cases. The Ka value was very close to the ideal values given by Maxim, 2015 and Ahmed, 2015. Where Ka is the coefficient of activity = (Al_2O_3 / SiO_2) , Kb being the coefficient of basicity = $((CaO + MgO) / (SiO_2 + Al_2O_3))$ and HM being the Hydration modulus, given as $((CaO + MgO + Al_2O_3) / SiO_2)$. In addition, the ratio of Calcium to Silica becomes a deciding factor for the generation of Calcium silicate Hydrate gel (C-S-H). All the mix cases yielded a low value of Ca/ Si ratios, where Abdel 2018, also suggested low values.

However, the mix case M23 was noticed to have least MgO content in the chemical composition compared to the other three cases including M22N. The SiO₂/ Na₂O values was also observed to be very low for this mix case. Simply by contrasting M23 (MgO least) and M21(MgO highest), the role of MgO and ideal content can be found out.

Except for Fe₂O₃ and CaO, all the other values varied by 2-10% only in the chemical content as well as the ratios. The ferric oxide content was slightly higher for M22N and lime content was slightly lower for M22N. This slight variation might be because of the ball milling done, which cannot be monitored or hampered beyond a certain extent.

4.2.2 Fineness by Blaine's Air Permeability Method

The greater fineness of M23 explains the strength attained. As shown in the figure 4.1, M22 and M23 have same amounts of silicate content, but MgO increment increased the fineness, simply because MgO is finer. Also, though M21 and M23 had the same amount of MgO, they had different fineness' because silicate content was also increased in M23.

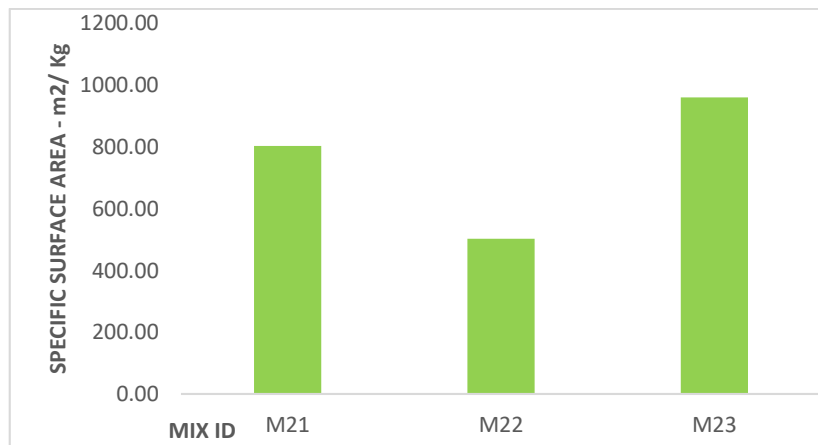


Figure 4.1 Blaine’s Fineness values

4.2.3 Soundness by Li-Chatelier’s Method

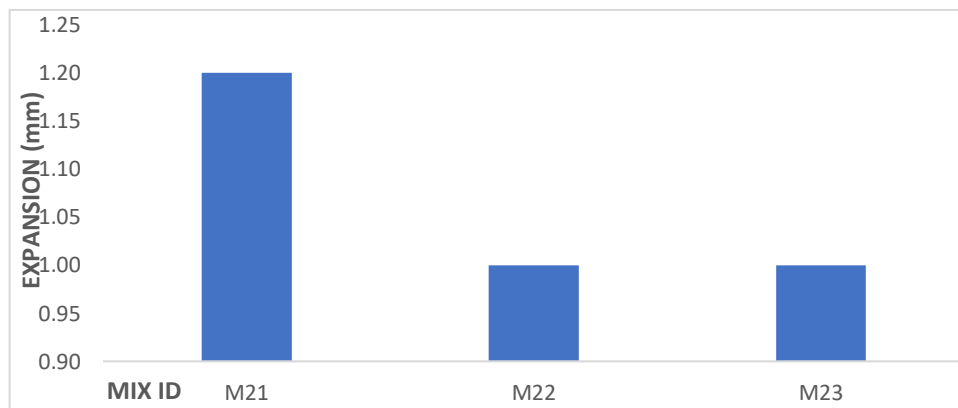


Figure 4.2 Expansion values in Soundness

Usually, Alkali activated Cements have very less unhydrated amount of Cement left, which leaves very less chances of expansion. As a result, the negative values attained, usually would represent the shrinkage, with Faris, 2018. Also, while the ideal amount of lime in Cement being about 40% in PPC and 60% in OPC, for all these mixes the lime content was around 12% only, hence all the values were noticed to be very less (<10 mm), though not negative. M21 showed higher expansion over the other mix cases, as shown in the figure 4.2 as the MgO was kept 10% while Na₂SiO₃ was maintained 5%. On contrary, from Phase -1 investigation also, it was seen that when MgO- Na₂SiO₃ were taken in the ratio 1:1 (in terms of their weight) i.e. 5-5% dosage and additionally in Phase -2 investigation, the combination with 10% dosage each (M23) gave good strength. The shrinkage would also be less automatically because usually concrete or mortar suffers shrinkage due to water curing, here the ambient curing compliments for the alkali cements.

4.2.4 Standard Consistency

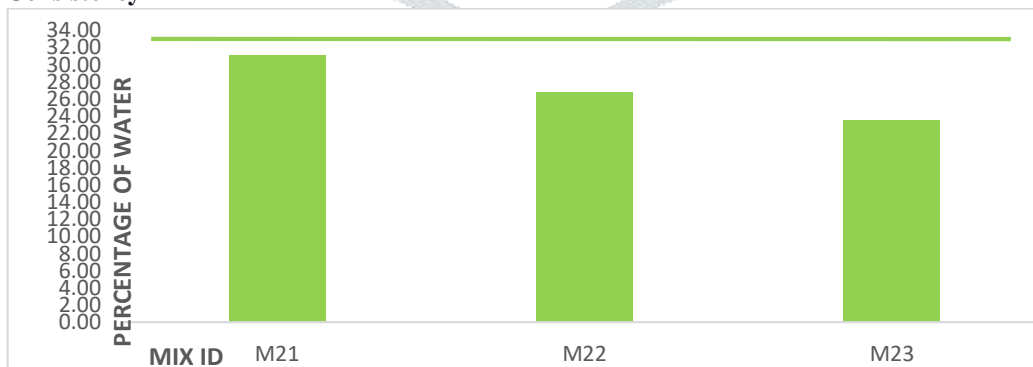


Figure 4.3 Standard Consistency

All the mixes followed a declining path as the Na₂SiO₃ and MgO contents were increased because of the absorptive and in soluble (Lee, 2002) nature of both respectively. The water required for Alkali cements is less compared to OPC (which is 33%) as shown in figure 4.3. This adds as an advantage in case of Alkali cements. Given the ambient curing condition, the total amount of water required would be still lesser.

4.2.5 IST & FST

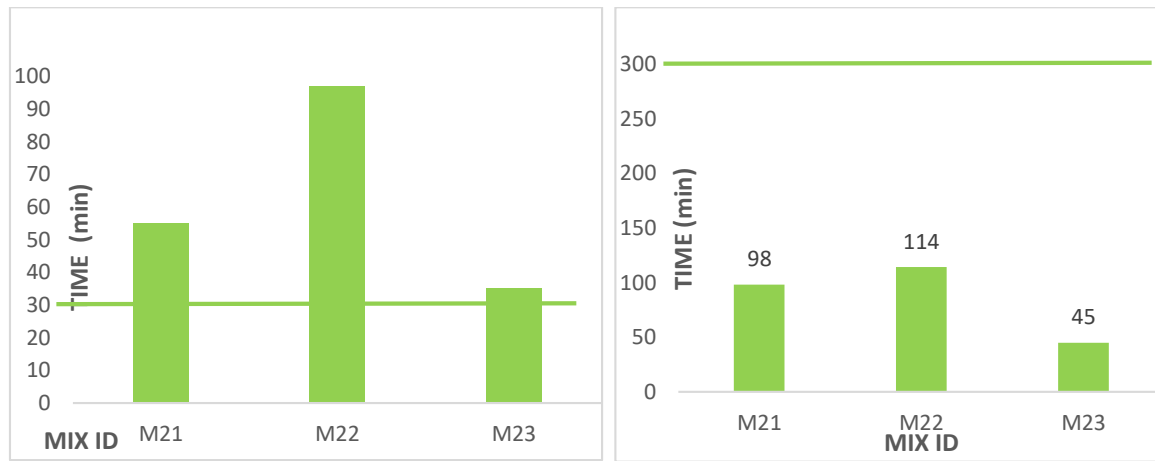


Figure 4.4 IST & FST

Na₂SiO₃ is known for its prolonged setting action, where Na₂SiO₃ when present along with FA and GGBS, increases the silica content even more, which is known to prolong the setting time as the figure 4.4 indicates. In case of 10% Na₂SiO₃ in both M22 and M23 cases, in M22 the MgO content was 5%, whereas the MgO content was 10% in M23. When same amount of Na₂SiO₃ was used while increasing the MgO content will increase the rapid setting as the fineness and Rate of HOH was higher for M23. Hence, M22 shows good agreement with the IST times as that of Faris, 2017. It holds good for FST too.

4.2.6 Compressive Strength

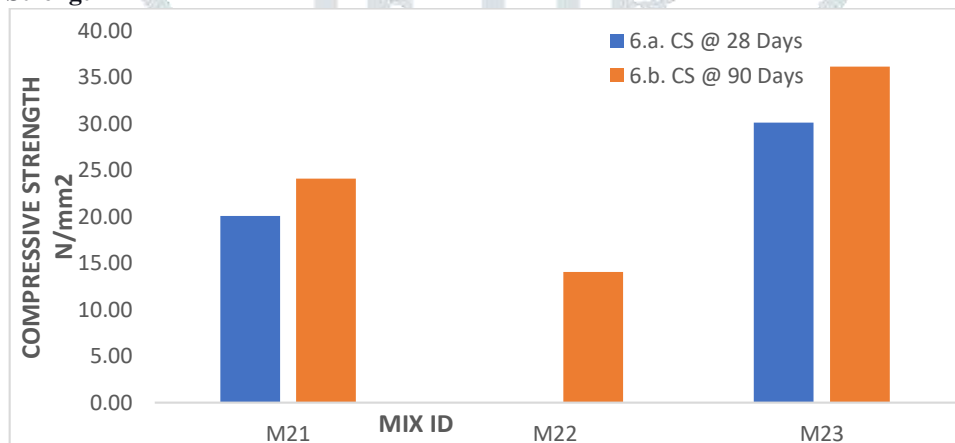


Figure 4.5 Compressive strength at 28 days and 90 days

Though all the mixes showed increment, M22 is noticed to show significant variation from the compressive strength rendered at 28 days to the 90 days compressive strength as shown in the figure 4.5, which is in agreement with Kamal,2016. The mix cases with higher fineness explains the strength for M23 but though the fineness of M22 is higher than that of M21, it didn't attain strength as the MgO content was only 5%. This shows that MgO content increment can increase the strength and the Na₂SiO₃ increment compliments even more. Most importantly, as the curing condition involved was ambient curing, this implies its edge in case of plastering works.

4.2.7 Heat of Hydration (HOH)

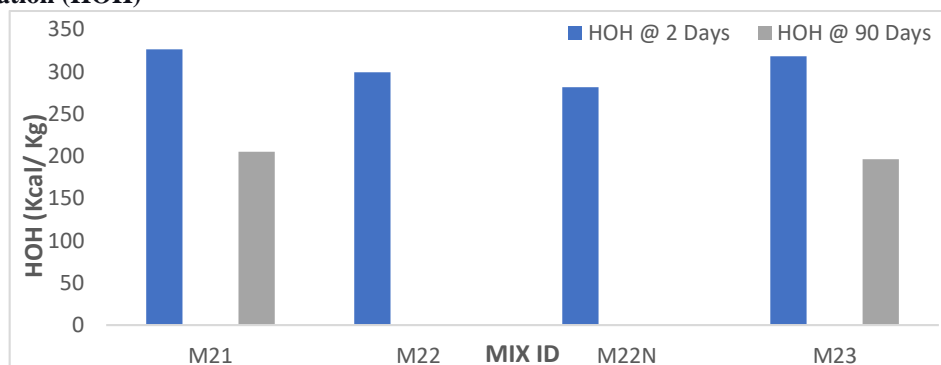


Figure 4.6 HOH after 28 and 90 days

These values were lower compared to the PPC considered, i.e. 336 at 2 days and 249 at 28 days as seen in the figure 4.6. Hence advantageous. Moreover as discussed earlier, the temperature was not high primarily, while mixing the contents in the mill. At 2 days, the sample remained unhydrated and the values projected were also the heat of hydration of unhydrated samples, whereas at 90 days the figure 4.8 shows the hydrated samples' heat of hydration.

Evidently, for both M22 and M22N the HOH at 28 days is nil. In addition, the slight variation of the HOH value at 2 days can be because of the variation in the attained fineness of the particles, while mixing in the steel mill.

4.2.8 Density

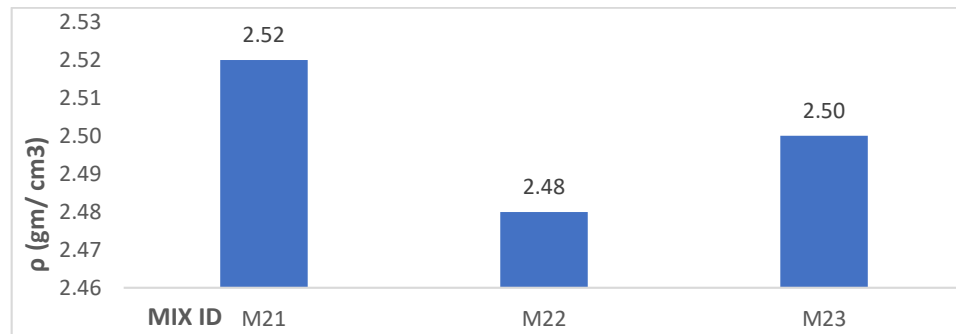


Figure 4.7 Density of the mixes

The density of the Alkali Activated Cement was noticed to be on the higher side compared to OPC i.e., 1.44 gm/cm³ as displayed in the figure 4.7. Hence, through this test the long-term properties such as durability and permeability can be related and understood that the negative effect would be lesser.

4.2.9 Service life of mix

In overall, for M22 & M22N cases, the composition of most of the chemicals varied only by 3-8%, and that the fresh mix; evidently, from the table- 4.2, mixes M22 and M22N, all the values of Ka, Kb, HM, Ca/ Al, Ca/ Si, Na/ Al, Si/ Al and SiO₂/ Na₂O vary by 3-5% only. In addition, did neither M22N nor M22 evolve any heat of hydration at 28 days.

4.2.10 Role of MgO

MgO in combination with Na₂SiO₃, when used at equal weight percentages, observed to give good strength. The good compatibility of this activator combination was proved through the standard consistency test, where the diverse natures of MgO and Na₂SiO₃ (insoluble and absorptive) balanced out for good. When the MgO content is increased for same amount of Na₂SiO₃, the setting time may be reduced. However, these aspects comply to only the MgO- Na₂SiO₃ combination of activators.

V. CONCLUSIONS

1. The replacement of Fly ash by 30 % of GGBS when paired with the unique activator combination of Na₂SiO₃ and MgO gave reasonable strength of 17.05 N/mm² by mere manual mixing, at 5% dosage of each.
2. XRF reported that Ca/Si and Si/Al ratios lied in the range 0.2-0.3 and 2.6-2.8 respectively; helping the C-Mg-S-H gel formation, which promoted the formation of structures of geopolymer, hence, aided the Alkali cement.
3. As part of the physical tests, it was noticed from the standard consistency test, that water required was very less compared to OPC; about 23.5% only, for Na₂SiO₃ – MgO mix both at 10% dosage.
4. The best combination was obtained with the Na₂SiO₃ - MgO with 10% dosage of each. This mix was able to render a compressive strength of 33.58 N/mm² at 28 days and 39.45 N/mm² at 90 days with just the ambient curing.
5. As per the two mixes observed with the age gap, had its chemical composition varying only by 3-10%. Hence, through the study a service life of minimum 30days can be guaranteed, when the Alkali cement is stored in an air-tight condition.
6. The optimum use of MgO can be when added in same amount, as that of the Na₂SiO₃ in terms of fineness, soundness, standard consistency and strength. However, the increase or decrease in the MgO content than ideal content can have negative outcomes too.

VI. ACKNOWLEDGEMENT

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