Effect of Admixtures in Blast Furnace Slag-fly Ash Based Alkali-activated Paste

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ABSTRACT

Portland cement can be replaced with alkali-activated binders (AABs), a sustainable material. They make use of industrial byproducts rich in aluminosilicates to produce hardened binders under alkaline conditions. The effective utilization of alkali-activated binders in each particular place is greatly influenced by the ease in accessibility of suitable precursors and activators. Slag-based AAB can make a strong and durable mix under ambient curing. But its poor workability and fast setting characteristics limit its scope of wide applications. The purpose of this investigation is to determine the effect of admixtures on the setting time of Alkali-activated Paste with blends of slag and fly ash (BFS-FA AAP). A combination of red gypsum and phosphoric acid is the admixture considered for the study. Setting time of BFS-FA AAP activated with sodium hydroxide and sodium silicate is studied considering different control factors, including Na_2O/b ratio, BFS/binder and activator modulus (SiO_2/Na_2O). The phosphoric acid, when added at 0.45 M along with red gypsum at 2.5% of binder content retarded the setting time of AAP effectively.

Keywords: Alkali-activated Paste, Slag-fly ash based, Rheology, Admixtures

1 Introduction

The global cement industry is booming because of the increasing demand for cement caused by construction of new buildings and other infrastructure. A tonne of CO_2, a major source of global warming due to its greenhouse effect, is released while manufacturing one tonne of Portland cement. The production of Portland cement accounts for more than 7 percent of all manmade carbon dioxide emissions [1]. Furthermore, CO_2 makes up around 65% of greenhouse gases that cause global warming. As a result, the Portland cement business need to match the idealised contemporary vision of a sustainable industry.

Alkali-activated binders (AAB) have received much attention as a part of environmentally friendly binder systems. The primary factor that may influence the adoption and consumption of alkali-activated binders in any given location is the availability of suitable raw materials (precursors and activators) in that area. Alkali-activated materials can minimise CO_2 emissions and industry waste management, using them instead of cement-based products has significant economic and environmental advantages. For selecting alkali-activated materials as a sustainable choice, bulk material transportation must be reduced.

Alkali activation is the process of transforming a solid aluminosilicate (AS), known as the "precursor," into a hardened binder made of hydrous alkali and aluminosilicate under alkaline conditions produced by the "alkali activator". The three most prevalent precursor materials are metakaolin from the calcination of kaolin, fly ash from coal power plants, and slag from iron manufacturing. In addition, there are a wide variety of potential precursors, such as bottom ash from municipal solid waste incineration, red mud, waste from the production of ferronickel, volcanic and synthetic glass, phosphorous slag, copper and zinc slag, and rice husk ash. The activator is the second essential part of AAB. Its function is to facilitate early age strength development and increase the reactivity of aluminosilicate powder. The activators for AABs are

based on an alkaline metal, Na or K, and an anionic group, with hydroxide and silicate being the most popular, but carbonate and sulphate are also relatively common [2].

Compared to alkali-activated fly ash with low-calcium content, which must be heated for curing, activated slag (AAS) or slag-fly ash blended material appear to have broader application prospects. [3] investigated the impact of partially substituting ground granulated blast furnace slag (GGBFS) for fly ash, on compressive strength. The addition of GGBFS as a partial replacement for fly ash enhances the compressive strength of fly ash-based alkali-activated concrete. Additional calcium-based hydration products CSH, CASH and polymerization products NASH make the matrix with fewer cracks and pore spaces, thus more compact.

Alkali-activated slag and fly ash blend provided with ambient curing have proven advantageous in practical applications. Gao et al. [4] studied the effects of two compositional factors: slag/fly ash mass ratios and activator modulus on compressive strength, reaction kinetics and gel characters. The findings demonstrate that, decreasing the ratio of SiO_2 to Na_2O dramatically speeds up the early-age response and increases rate of reaction. At low activator modulus, increasing the slag percentage also speeds up the reaction rate. Greater ratios of mass of slag to fly ash often favour high value of activator moduli, but activator moduli that are either too high or too low have a negative impact on strength.

Fahim et al. [5] investigated how microstructures and mechanical characteristics of blended geopolymer mortars were tailored with the molarity of the solution. The influence of Na_2O content, solution modulus (SiO_2:Na_2O) and water content on the mechanical properties of GPMs was investigated. As the alkali content increases, the setting time and flow capacity of such GPMs were shown to linearly decreasing. Rate of geopolymerization increases while using NaOH with higher molarity, due to the higher concentration of Na_2O, resulting in a shorter setting time. However, as the alkali content rises, the density, split tensile, and flexural strength increases. The highest mechanical properties were achieved by samples made with 12 M NaOH.

The rheology and mechanical properties of AABs are influenced by a variety of factors. The ratios such as SiO_2/Na_2O, water/solid, Na_2O/binder and BFS/binder are the controlling elements on its ability to flow and time for initial and final setting of slag and fly ash-based alkali-activated paste (BFS/FA-AAP). Other factors that can affect the strength of BFS/FA-AAP are the curing condition and duration of curing. Sun et al. [6] conducted systematic quantitative investigation on the impacts of control factors on the early age characteristics of BFS/FA-AAP. According to the results, raising the BFS/binder and Na_2O/binder ratios can result in stronger materials while diminishing workability.

Dai et al. [7] investigated the impact of the water to binder (w/b) ratio on the fresh properties and rheology and of alkali-activated cement (AAC) pastes. A mixture of class F fly ash (FA) and GGBFS at equal proportion, activated using sodium silicate and sodium hydroxide in three distinct w/b ratios. The findings indicated that greater w/b ratios resulted in structural build-up faster than lower w/b ratios. Comparing AAC combinations to regular Portland cement mixtures, it was identified that the w/b ratio had less impact on the setting times. It is discovered that a water content of 0.42 times binder content has shown good workability till 40 minutes.

Athira et al. [8] investigated the effects of water, heat, ambient and other curing procedures on the behaviour of alkali-activated binders. Compared to fly ash-based binders with ambient curing, slag-based alkali-activated binders with ambient curing showed more strength gains. Fly ash promotes to later-age strength, on the other hand slag accelerates strength gain in the initial phases, which emphasizes the importance of slag and fly ash blending.

Tong et al. [9] examined recent developments on utilization of chemical admixtures to increase the setting time and workability of Alkali Activated Slag (AAS) systems. The characteristics of alkaline activators greatly

impact how well the polymers work. Tartaric acid and $CaSO_4$ can greatly slow down the hydration of slag activated with NaOH. Phosphoric acid, phosphate, boric acid, borax are chemicals appropriate for AAS systems as retardants. [10] examined the suitability of phosphoric acid in sodium silicate-activated slag systems as a retardant and discovered that the concentration of phosphoric acid is the key factor on the control of retardation. A minimal impact was observed at a concentration between 0.78 M and 0.84 M.

To minimise AAS shrinkage after drying, Chang et al. [10] employed gypsum. An activator based on sodium silicate was utilised. According to the results of the experiments, adding gypsum sped up the setting process, and at the same age, using more gypsum increased compressive strength. Drying shrinkage decreased with increase in percentage of gypsum. The setting time was sped up by adding additional gypsum and phosphoric acid. At 28 days, the samples with extra gypsum had a lower compressive strength than the control samples.

Lee and Lee [11] studied the time for setting and strength characteristics of blended alkali-activated concrete with slag and fly ash, cured at ambient temperature. In alkali-activated fly ash/slag concrete, adding phosphoric acid (H_3PO_4) solution slowed down the setting time, thus act as a set-retarding additive. A significant increase in the initial setting time and decrease in the time for final setting was observed with the addition of phosphoric acid at a weighted level of 2.0% of the total weight of fly ash and slag (binder). Compared to the specimen without H_3PO_4, the specimen with H_3PO_4 had slightly lower compressive strength, but the inclusion of H_3PO_4 was a successful method retardation in such blended activated concrete.

Wang et al. [12] investigated the hydration kinetics of slag activated with sodium hydroxide and the influence of gypsum on hydration. Even though the initial setting was greatly delayed, gypsum greater than 3% reduced compressive strength. The outcomes demonstrate that $CaSO_4$ can be used as a retarder at a specific dosage, in this case, 4%. At this level, the initial setting had a 1-h delay, and its compressive strength was on par with the sample devoid of $CaSO_4$. Calcium ions were responsible for $CaSO_4$'s retardation mechanism by preventing slag dissolving.

Despite the fact that slag-fly ash-based alkali-activated binders have undergone significant research, their properties vary significantly based on the material's source. The various parameters influencing the properties of BFS/FA-AABs and their optimal values yielding good workability, setting time and strength are still under study. Studies on the effect of gypsum and phosphoric acid on the rheology and strength characteristics on slag-based AABs are available. But their impact on Slag-Fly ash AAB is unknown. The aim of this study is to examine the behaviour of blast furnace slag-fly ash-based alkali-activated binder systems with red gypsum and phosphoric acid as admixtures.

2   Materials and Methods

Fly ash (Class F) and GGBFS were utilised as precursors in the investigation A combination of sodium silicate and sodium hydroxide were used to activate the precursor. Apart from these, admixtures are also added which includes, red gypsum and phosphoric acid. The BFS used is collected from Astra chemicals, Chennai, and it has a specific gravity of 2.9. Due to its strong reactivity, GGBFS is regarded as one of the most desirable precursors for alkali activation. The Class F fly ash is collected from a ready-mix concrete plant in Kollam. The specific gravity of fly ash used is around 2.2. Compared to Class C fly ash, the use of Class F fly ash is more preferable for blended precursor with BFS. The combination of Na_2SiO_3 and NaOH is regarded as an alkaline solution in this study.

The chemical composition of the sodium hydroxide employed in the investigation is given in table 1. The molarity of the NaOH solution employed has a significant impact on the generation of reaction products. Previous research has shown that alkali-activated binders with 12 M NaOH solution produce the best

strength characteristics [5]. Hence 12 M NaOH solution is used for preparing the activator solution for this study. Liquid sodium silicate used for the study was obtained from Minar chemicals, Ernakulam. The chemical composition of sodium silicate is listed in table 2.

Table 1: *Chemical composition of sodium hydroxide*

Assay	Carbonate (Na$_2$CO$_3$)	Chloride (CI)	Sulphate (SO$_4$)	Lead (Pb)	Iron (Fe)	Potassium (K)	Silicate (SiO$_2$)	Zinc (Zn)
97%	2%	0.01%	0.05%	0.001%	0.001%	0.1%	0.05%	0.02%

Table 2: *Chemical composition of the sodium silicate solution*

Product Name	Density	SiO$_2$/Na$_2$O	Solid Content	Water Content	Iron content
54 Bhumi Sodium Silicate	1.55	2.4	59.5%	40.5%	0.02% max

The admixtures used in this study are red gypsum and phosphoric acid. Previous research has shown that gypsum delays the alkali-activated binder's reaction.; red gypsum, a waste material of similar composition, is thus used in this study. Red gypsum (RG), a reddish brown semi-solid mud, is a waste product of the sulphate processing of titanium and iron-rich ilmenite ore to produce titanium dioxide. Red gypsum mainly contains hydrated calcium sulfate (CaSO$_4$.2H$_2$O) and iron hydroxide (Fe(OH)$_2$). The red gypsum collected from Travancore Titanium Products Limited, Trivandrum has a 69% CaSO$_4$ content and 30.94% Fe(OH)$_2$ content. Red gypsum is added at 2.5, 5, 7.5 and 10% of the binder content. Phosphoric acid has shown retarding effect on alkali-activated slag-based binder systems. Previous studies showed that phosphoric acid visibly retard setting beyond 0.78 M and compressive strength gets reduced after 0.87 M. Hence the concentration of Phosphoric acid is taken below 0.8 M for this study. The composition of phosphoric acid used for the study is shown in table 3.

Table 3: *Chemical composition of Phosphoric acid*

Assay	Weight per ml at 20 ˚C	Calcium & Magnesium	Chloride (CI)	Sulphate (SO$_4$)	Nitrate (NO$_3$)
88%	1.75 g	0.01%	0.001%	0.01%	0.002%

The characteristics of the alkali-activated slag-fly ash paste is only considered for the study. The behaviour of AAP depends on certain ratios such as BFS/binder, SiO$_2$/Na$_2$O, water/solid, and Na$_2$O/binder. The values of control factors used in this study are given in table 4. All these ratios are mass ratios. "BFS" represents the mass of GGBFS. "b" represents the sum of the mass of both GGBFS and FA. "Na$_2$O" represents the sum of Na$_2$O in the activators, i.e., sodium silicate and sodium hydroxide. "SiO$_2$" represents the silica equivalent in sodium silicate activator. "w" represents the sum of water content from activators and extra water added. "s" represents the total solid content from precursors, activators, and admixtures if any [6]. The values of control factors are identified based on the optimum values adopted in various literatures.

Table 4: *Control factors of the study*

SiO$_2$/Na$_2$O	Na$_2$O/b	BFS/b	w/s
1.5	6%	0.5, 0.75	0.4

The main objective is to obtain the optimum percentage of red gypsum and the molarity of the phosphoric acid required to cause retardation in setting time of the AAP. Using red gypsum and phosphoric acid in conjunction is the primary focus. The different combinations of red gypsum and phosphoric acid used are shown in table 5.

Table 5: *Different combination mixes with red gypsum and phosphoric acid as admixtures*

	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5	Mix 6
Red Gypsum (% Binder content)	2.5%, 5%, 7.5%, 10%	2.5%	0	1%, 2.5%	2.5%	2.5%
Phosphoric acid (M)	0.8M	0.8M	0.5M, 0.8M	0	0.25M, 0.5M	0.35M, 0.45M
BFS/b	0.5	1	0.5	0.5	0.5	0.5, 0.75

3   Experimental Procedure

3.1   Mixing Protocol

The BFS-FA AAP was prepared according to Table 4. Sodium hydroxide pellets were dissolved in water to make it 12 M concentration. A day before casting, sodium silicate solutions and 12 M NaOH solution were combined according to the mix proportion to create the final activator solution. When the solutions are combined, a significant quantity of heat is released. The solid materials i.e., precursors (BFS and FA) and red gypsum were premixed for 2 min in the Hobart mixer shown in Fig. 1 (a). The alkali-activated solution, along with additional tap water as per the mix proportions were then added and mixed for 1 min. In the case where phosphoric acid is used as an admixture, the solution of required molarity is prepared with the additional tap water and added along with the activator solution. During the 30 s of rest, material stuck to the bowl walls, bottom and mixing arm was scraped off. The mixing process is continued for another 1 min 30 s.

(a)   (b)   (c)
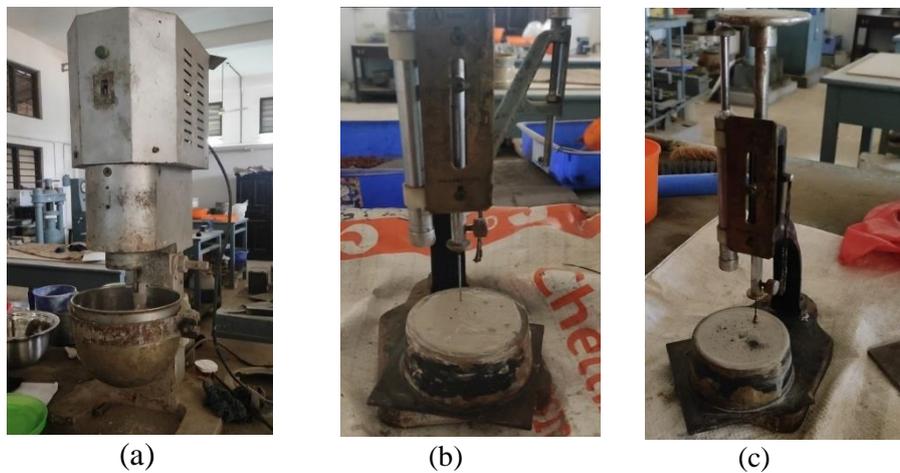
Figure 1: *Apparatus used for the study* **(a)** *Hobart mixer* **(b)** *Vicat apparatus with needle for the test for IST* **(c)** *Vicat apparatus with needle for the test for FST*

3.2   Testing Program

Test for initial and final setting time of BFS-FA AAP was determined using the Vicat apparatus shown in Figure 1 (b), (c). The Vicat mould was filled with a fresh mixture. Periodic penetration tests are carried out by letting a 1-mm Vicat needle penetrate into this paste. The time from when the raw materials first made contact with the activator and when the penetration gauge read 5 mm from the bottom was measured to determine the initial setting time (IST). For final setting time test, needle with an annular collar was used. When the needle leaves an indentation in the paste, but the annular collar doesn't, this is the final setting time (FST), which was measured from the moment the raw ingredients first came into contact with the activator.

4 Results and Discussion

All the samples of AAP were prepared with Na_2O/b ratio of 6%, Ms ratio of 1.5, w/s ratio of 0.4 and tested for setting time. As mentioned in Table 5, mix 1 was prepared with equal proportion of slag and fly ash and varying percentage of red gypsum such as 2.5%, 5%, 7.5% and 10% of the binder content. A phosphoric acid of 0.8 M is also added to the mix. At all the different combinations of admixtures used, the setting time of AAP was retarded for more than 24 hours.

As the setting time was retarded to a great extent in the case of mix 1, the red gypsum of 2.5% of binder content and 0.8 M H_3PO_4 was added to AAP with higher BFS/b ratio of 1 without varying other control factors (mix 2). It was observed that with slag as the only binder, the AAP sets quickly with an IST of 28 minutes. However, when the admixtures were added in the proportion mentioned above, the setting of the AAP was retarded to more than 24 hours. Hence it was figured that the admixtures added have a high retarding effect at the mentioned proportions and combinations, even at high slag content.

The effect of the phosphoric acid alone as an admixture on the setting time was studied using mix 3. Phosphoric acid solution of molarity 0.5 and 0.8 was added to the AAP with 50% BFS in binder and all other control factors same as previous mix. The setting time was retarded at both of the molarity used. At 0.8 M, the AAP didn't set even after 24 hours. Slowing the setting time was noticed as a function of increasing the content of phosphoric acid. A dramatic rise in the setting time was observed after changing the concentration of phosphoric acid from 0.5 M to 0.8 M, inferred that the setting time is highly dependent on the phosphoric acid content. The setting time of more than 24 hours when using 0.8 M phosphoric acid as a retarder is inappropriate in engineering practise. Thus, a molarity of 0.5 and lower may be used for further stages of the study.

The impact of red gypsum alone as admixture on the setting time of BFS-FA AAP was studied using mix 4. It was observed that AAP, with BFS/b ratio 0.5, has an IST of 47 min and a FST of 150 min. Figure 2 shows the effect of various proportion of red gypsum on the setting time of AAP. Initially, red gypsum was added at 2.5% of binder content, and the setting was accelerated, with an IST of 26 min and FST of 115 min. When the quantity of red gypsum was reduced to 1%, the setting time was increased with an IST of 45 min and FST of 125 min, almost equal to the setting time of AAP without admixtures. Even at the low quantities of red gypsum setting time of AAP was accelerated by a small amount. As the quantity of red gypsum increases, its accelerating property is enhanced. Thus, red gypsum acted as an accelerator.

Even though gypsum ($CaSO_4$) is used as a retarder in conventional cement concrete, it behaves differently in the AAB system. The setting time was reduced with the addition of red gypsum, which has a high quantity of $CaSO_4$ (about 69%) and the remaining quantity of iron hydroxide (Fe $(OH)_2$). The reduction in setting time can be attributed to the additional calcium ions entering the system when red gypsum was used. Thus, a higher rate of CASH gel production may be formed with higher concentrations of red gypsum. Consequently, a faster setting was observed when the gypsum was added [10].
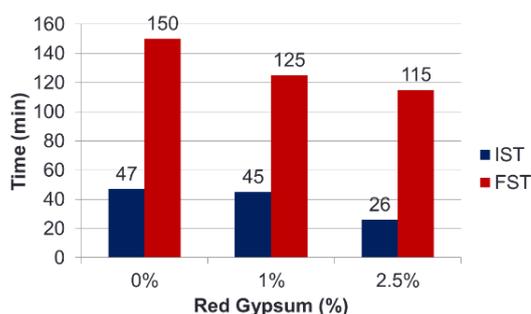
Figure 2: *Influence of red gypsum on initial and final setting time of AAP*

It was observed that the addition of 2.5% red gypsum accelerated the initial setting time by 21 minutes, and phosphoric acid at 0.5M retarded the setting time to more than 24 hours. Thus, red gypsum and phosphoric acid are added together, as mentioned in mix 5, to achieve a reasonable setting time for the BFS-FA AAP. Red gypsum is added at 2.5% of the binder content. The molarity of phosphoric acid used includes 0.25M and 0.5M. Figure 3 shows the setting time test on AAP with phosphoric acid and 2.5% red gypsum. When 2.5% red gypsum and 0.25M phosphoric acid were added to BFS-FA AAP with 50% slag in binder, the IST was accelerated to 38 min and FST to 110 min. When 2.5% red gypsum and 0.5M phosphoric acid were added to BFS-FA AAP the setting time was retarded. When only 0.5M phosphoric acid was added as admixture, the AAP took more than 12 hours to set. Thus, it can conclude that at 2.5% red gypsum content and a molarity of phosphoric acid between 0.25 and 0.5, a reasonable setting time can be achieved.

Using mix 6, the molarity of phosphoric acid at which the BFS-FA AAP has achieved a reasonable setting time in the presence of 2.5% red gypsum is found. Two different molarity of the phosphoric acid, 0.35M and 0.45M, are considered in between 0.25M -0.5M. The BFS/b ratio equal to 0.5 and 0.75 is used, keeping other control factors the same, the setting time is studied. The results are tabulated in table 6. Figure 3(a) shows the variation in the time for setting BFS-FA AAP for the various combination of admixtures used at BFS/b ratio of 0.5. When 0.35M phosphoric acid and 2.5% red gypsum were used as an admixture, the IST was accelerated by 6 min and FST by 20 min compared to AAP without any admixtures. When the molarity of phosphoric acid was increased to 0.45, the IST was retarded by 53 min and FST by 77 min. Thus 0.45M phosphoric acid and red gypsum at 2.5% binder content effectively retarded the setting time of BFS-FA AAP of Na_2O/b ratio 6%, Ms ratio 1.5, w/s ratio 0.4 and BFS/b ratio 0.5.

Table 6: *IST and FST of BFS-FA AAP with various combinations of admixtures*

Sl No	Molarity of phosphoric acid solution (M)	Quantity of red gypsum (%)	BFS/b	IST (min)	FST (min)
1	0	0	0.5	47	150
2	0.25	2.5	0.5	38	110
2	0.35	2.5	0.5	41	120
3	0.45	2.5	0.5	100	227
4	0	0	0.75	32	135
5	0.35	2.5	0.75	35	125
6	0.45	2.5	0.75	80	152

Figure 3 (b) shows the variation in the setting of BFS-FA AAP for the various combination of admixtures used, with a BFS/b ratio of 0.75. When 0.35 M phosphoric acid and 2.5% red gypsum were used as an admixture, the IST was retarded by 3 min and, FST was accelerated by 10 min compared to AAP without any admixtures. When the molarity of phosphoric acid was increased to 0.45, the IST was retarded by 48 min and FST by 18 min.

It was discovered that the extended retarding impact of phosphoric acid might be mitigated by combining it with red gypsum. Even though the retarding effect on setting time was minimised, the setting time was still longer than the AAP without the phosphoric acid and red gypsum. This may be because the concentration of calcium ions increased when gypsum was mixed with phosphoric acid. As a result, the present phosphoric acid concentration is insufficient to continue offering the same retarding impact. Calcium phosphate formed as a result of the action of phosphate acid may cause a retarding mechanism.

| (a) | (b) |
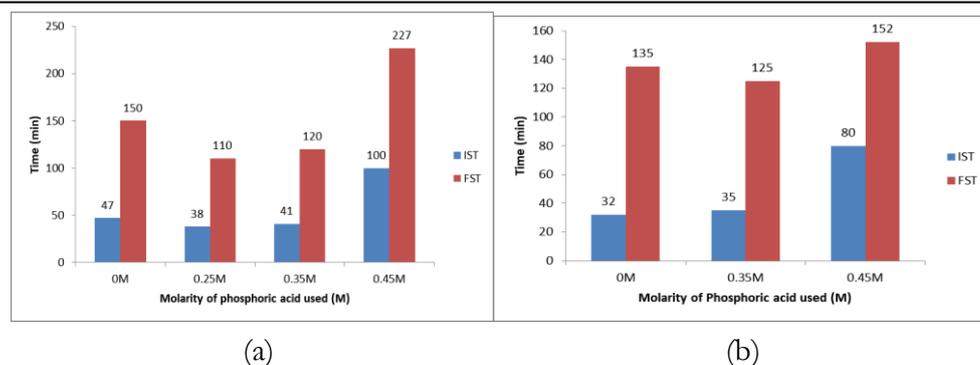
Figure 3: *Variation of setting time with the molarity of phosphoric acid for BFS-FA AAP of at 2.5% of red gypsum* **(a)** *BFS/b ratio= 0.5* **(b)** *BFS/b ratio= 0.75*

In the presence of red gypsum, the solution included more calcium ions, so a more significant concentration of phosphoric acid was needed to achieve the same retarding effect. This indicates that there are still excess calcium ions available for consumption by the hydration reaction of CSH gel, which shortens the setting time. Thus, a desirable setting time in AAP was reported at 0.45 M phosphoric acid and 2.5% red gypsum concentration.

The phosphoric acid, heavily retarded the setting time when added as the only admixture in AAP. Red gypsum acted as an accelerator when added alone in AAP, but when added in combination with phosphoric acid of certain molarity, a retardation in setting time was observed. The combination of red gypsum at 2.5% of binder content and 0.45 M phosphoric acid yielded a favourable setting time for BFS-FA AAP with both 50% and 75% slag content in the binder.

5   Conclusions

This study examined the effects of phosphoric acid and red gypsum on setting time of AAP. The combination of phosphoric acid and red gypsum was the main focus. The phosphoric acid alone increased the time required for the material to set. Red gypsum, on the other hand, sped up the setting process. Phosphoric acid's retarding impact was nullified when it was used in conjunction with gypsum. The phosphoric acid, when added at 0.45 M in combination with red gypsum at 2.5% of binder content retarded the setting time of AAP effectively. The effect of admixtures was found positive to control the setting time of BFS-FA AAP and further study is required to identify the strength characteristics too.

6   Declarations

6.1   Acknowledgements

6.2   Publisher's Note

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How to Cite

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