Performance of alkali activated slag concrete under aggressive environment

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Abstract

The environmental effects of production of Portland cement (PC) have provoked to examine the growth of concrete with 100% replacement of cement with industrial byproducts containing high amount of Si and Al, which are activated by alkali solutions termed as geopolymer concrete. Concrete made with PC can be durable under mild exposure condition when properly designed whereas undergo deterioration under severe exposure condition. Since very few works have been performed on the ambient cured alkali activated slag concrete (AASC) under aggressive environmental condition, this work was intended to study the effect of binder content and sodium hydroxide concentration on AASC subjected to aggressive environment. In this regard, an experimental investigation was carried out to study the influence of AASC under chloride, acid and sulphate environment on its physical and mechanical properties. The results infer that the AASC mixes perform well under aggressive environment condition.

Keywords: geopolymer concrete, alkali activated slag, aggressive environment, mechanical properties, sulphate, chloride, ambient temperature.

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1 Introduction

The manufacture of PC needs huge amount of energy in the meantime transmits immense sum of CO_2 to the atmosphere due to the calcination reaction during its manufacturing process. In the manufacture of 1 T of PC, 0.53 T of CO_2 is released due to the calcination and may develop as much as 1T if carbon gasoline is used as energy source. Conversely, the creation of commercial through-products releases fewer amounts of green house gases (GHG) comparing to PC. Fly ash produces 80-90 % and slag produces 80 % less GHG emission to the atmosphere compared with PC. Therefore 100 % substitute of PC with fly ash or slag would significantly reduce the impact on environment. In cement concrete, calcium silicate hydrate (C-S-H) and the portlandite ($Ca(OH)_2$) are the chief hydration products which governs the strength and the binding characteristics as well as highly susceptible to chemical degradation when exposed to severe environmental conditions. This may also in some cases; that the configuration must be revamped or even reinstated due to this effect. The dispersal of dissolved species inside interstitial fluid and dissolution of diverse hydrate phases were the two processes suggested by Xie [1] to control the degradation of cement-based materials under chemical exposure. Yang and Cho [2] expressed that the accelerated chloride migration test showed a decent relationship linking the charge passed and the steady state chloride flux. However, a new method based on Neumann's theory was proposed by Chih-Hsing Wang [3] to determine the chloride diffusion coefficient without the aid of electric current contrast to rapid chloride permeability test or rapid chloride migration test. Cement concrete subjected to sulphate exposure is depicted by the chemical response of sulphate particles as the forceful agent and the aluminate segment of hardened cement paste [4-5]. The formation of ettringite due to high amount of C_3A is the main reason for the deterioration of PC [6]. Among the properties that control the resistance to sulphate exposure, permeability

and chemical composition of cement paste are the most important. Due to the difference in the chemical and phase composition, the concrete made with PC is more susceptible than made with slag to acid attack. As a result of the formation of C-S-H gel and silica gel, the surface of the cement concrete specimens becomes soft and could be delaminated resulting in concrete layers to deterioration [7]. Alkali activated slag (AAS) mortar exposed to 5% sulphuric acid shows severe degradation than citric acid, hydrochloric acid and nitric acid solutions over a period of 6 month [8]. The concrete made with AAS had superior resistance than concrete made with OPC of similar grade when reacted with acetic acid solution of pH=4 [7].

Most of the previous studies have been done to study the behavior of geopolymer in the form of mortar under aggressive environment when cured under elevated temperature. Since very few researches were carried on geopolymer concrete under ambient temperature curing, this experimental investigation was carried out to study the effect of binder content and NaOH concentration on the physical and mechanical properties of AASC under aggressive environment.

Hence, this paper at the inception provides an outline of the experimental investigation integrating materials & their characteristics, their proportioning and experimental mechanism. It was then pursued by an elaborated discussion on the obtained results, where the effect of addition of binder content (350, 400 and 450kg/m^3) and NaOH concentration (10, 12 and 14M) on the physical and mechanical properties of the AASC under aggressive environment (acid, sulphate and chloride) condition. The properties are offered in conjunction with added significant elucidations.

2 Materials and Methods

2.1 Constituent materials and mix proportion

Ground granulated blast furnace slag (GGBFS) was used as geopolymer source material (GSM), as GGBFS is the most common cementitious material for AAS binder [9] and its chemical composition is given in Table 1.

From Table 1, the slag used was categorized as acidic, as the ratio between the total basic to acidic components was found to be 0.98 which makes it best suited as a starting material for AAS binder. The past research works suggested that the blend of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) solution can be used as alkali activators resulting in higher strength [10]. Sodium hydroxide was acquired in the form of flakes and sodium silicate in liquid form with silica modulus of 2.5. The fine and the coarse aggregates were equipped in accordance with ASTM C33/C33M (*Standard Specification for Concrete Aggregates, ASTM International, 2016*) and their moisture condition was found to be in saturated surface dry (SSD) condition. Graded river sand with a fineness modulus of 2.56 and specific gravity of 2.58 was used as fine aggregates along with the coarse aggregate of 16mm maximum size of crushed granite type available in saturated surface dry condition with a fineness modulus of 6.70 and specific gravity of 2.61. To evaluate the performance of the mixes under aggressive environment condition, the AASC mixes were prepared with variation in the volume of GGBFS as 350, 400 & 450 kg/m^3 and the NaOH concentration as 10, 12 and 14 molar, keeping the molar ratio of SiO_2/Al_2O_3 and liquid to binder ratio constant and the mix proportions are shown in Table 2.

2.2 Methodology

The workability of the mixes was determined in the form of slump cone test following ASTM C143/C143M (*Standard Test Method for Slump of Hydraulic-Cement Concrete, ASTM International, 2015*). The compressive strength was determined using cylindrical specimens of size 100mm dia. and 200mm height as per ASTM C39/C39M (*Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens, ASTM International, 2015*). The moisture absorption and volume of voids were ascertained using 100mm size cubes as per ASTM C642 (*Standard Test Method for Density, Absorption and Voids in Hardened Concrete, ASTM International, 2013*). Exposure to acid attack was chosen to study its durability characteristics as the acids are normally considered to be highly aggressive to concretes. The sulphuric acid is taken into the account as this combines both acidic and sulphate environment simultaneously. The percentage variation in mass and compressive strength of the mixes made with 100 mm cubes exposed to 2% sulphuric acid over a period of 90 days were assessed as considered by Li and Zhao [11]. The chloride ion diffusion was measured using salt ponding test similar to the test prescribed in AASHTO T259 (*Standard Method of Test for Resistance of Concrete to Chloride Ion Penetration (Salt Ponding Test), American Association of State Highway and Transportation Officials, 2002*) and AASHTO T260 (*Standard Method of Test for Sampling and Testing for Chloride Ion in Concrete and Concrete Raw Materials, American Association of State Highway and Transportation Officials, 2009*) using cylindrical specimens of 100mm dia. and 200mm height and the potentiometric titration was carried out as per NT BUILD 208 (*Concrete, hardened: Chloride content by Titration, Nord Test Method, 3rd Edn., 2011*) to determine the total chloride content (%) by weight of concrete using Eq. (1).

$$\textit{Total Chloride Content } (Cl^-) = 3.545 \times \frac{V_1 N_1 - V_2 N_2}{m} \quad (1)$$

where, V_1 and N_1 are the volume and normality of $AgNO_3$ solution added, V_2 and N_2 are the volume and normality of ammonium thiocyanate added.

The resistance to sulphate attack was evaluated using cylindrical specimens of size 100mm dia. and 200mm height following ASTM C88 (*Standard Test Method for Soundness of Aggregates by use of Sodium Sulfate or Magnesium Sulfate, ASTM International, 2013*). The specimens were immersed in 5% sodium and magnesium sulphate solutions over a period of 90 days and the reduction in mass and strength were evaluated. The core samples of 50mm height were taken for visual examination. The solutions were changed periodically at an interval of 10 days in order to maintain the concentration throughout the exposure period. The loss in the strength was measured by sulphate deterioration factor (SDF) which is the percentage variation in strength of the specimens after immersion in sulphate solution with that of immersed in water at age "t".

3. Results and discussion

3.1. Fresh concrete properties

The workability of concrete is mainly influenced by the water requirements at the time of mixing. For conventional concrete, it was decided mainly on the basis of the maximum size of aggregate used. The test results of the workability of the AASC mixes are presented in Fig 1. As can be seen, the workability of AASC mixes decreased when NaOH concentration increased. A clarification for this pattern can be because of the measure of water and alkaline in the solution prepared. More NaOH solids were allowed with the increase in the activator concentration and

less free water for a given volume, bringing about the reduction in the slump values. Similarly, the slump values were observed to reduce with the increase in the GGBFS volume similar to OPC concrete.

3.2 Compressive strength

The compressive strength results of the AASC mixes at the age of 7 and 28 days curing are shown in Fig 2 and 3 respectively. The use of high calcined source material as binder was stated to improve the microstructure of the geopolymer matrix resulting in high strength [12] and the reaction between GGBFS and alkaline solution is an exothermal process in which the geopolymerization process will be promoted by the generated heat [13]. GGBFS contains higher CaO content and consequently, it is a good impending resource of soluble Ca in the mixture. The amount of soluble calcium relies on the GGBFS quantity available in the mix and the compressive strength has a direct consequence on it. An increase in the compressive strength results was found to be averagely 12.9 % for 7 days and 11.8 % for 28 days curing with an increase in the GGBFS content of 350 to 400 kg/m^3 and drops averagely by 3.8 % for 7 days and 3.4 % for 28 days curing when increase to 450 kg/m^3.

The activator concentration has been known as the most critical component influencing the properties of alkali-activated binders. The impacts of activator concentration, in any case, have not generally been that basic. While a few studies proposed that a base minimum concentration of activators are required for the activation to be successful, there are distinctive studies demonstrating adverse impacts of high concentration on the strength properties of alkali-activated binders. The utilization of high concentration NaOH solution increases the dissolution

of the solid materials and the geopolymerization process resulting in higher compressive strength and mostly because of the higher level of leaching of Si and Al. NaOH concentration on aqueous phases increases the compressive strength averagely by 13.0% and 12.9% for 7 and 28 days curing respectively for an increase in the NaOH concentration of 10M to 12M; and further increment in the strength averagely by 11.5% and 12.1% for 7 and 28 days curing respectively from 12M to 14M. Since one of the fundamental strides of geopolymerization procedure is the breakdown of aluminosilicate bonds (Si-O-Si and Al-O-Si) in alkaline environment, higher concentration prompted enhanced breakdowns. As more breakdown of aluminosilicate bonds, there were further prospects for hydrates to be shaped, thus increasing the compressive strength. The increase in the compressive strength with the increase in the NaOH concentration can be related to improved solvency of aluminosilicate at higher concentration [14-15].

3.3 Saturated water absorption and volume of voids

The variation in the water absorption and the volume of voids of the AASC mixes are shown in Fig 4 and 5 respectively. The amount of binder content in concrete has an especially strong impact on the water retention properties of AASC mixes, despite the fact that the absorption characteristics are, in all cases, lower than the equivalent OPC mixes even where porosities are comparative. This is most likely a consequence of the presence of exceptionally refined, convoluted and confined porosity in the AASC samples, which water does not promptly infiltrate, furthermore the continuous arrangement of reaction products at advanced ages of curing [16].

The superior property of the volume of voids is mainly because of the structure of the C-A-S-H ($CaO-Al_2O_3-SiO_2-H_2O$) binding gel type which dominates the microstructure of the geopolymer concrete is highly dense [17] comparing to N-A-S-H ($Na_2O-Al_2O_3-SiO_2-H_2O$) gel formed in fly ash based GPC and C-S-H gel [18] formed in conventional cement concrete. The existence of added bound water, persuaded by the availability of Calcium ion in the network, offers superior pore-filling ability to C-A-S-H gel than N-A-S-H type gels in GPC [19].

3.4 Resistance to sulphate attack

3.4.1 Sulphuric acid attack

The percentage reductions in mass and compressive strength results are shown in Fig 6 and 7 respectively. The excellent acid resistance performance is mainly owing to the absence of $Ca(OH)_2$. Unlike concrete made with PC which relies on C-S-H bonds for structural integrity, geopolymers are novel binders that depend on aluminosilicate for structural integrity and hence reported as acid resistant. High content of binder leads to reduction in deterioration of specimens and found structurally intact with no appreciable change in colour. This excellent performance may also be associated to the formation of alumino-silicate gel as the main binder in AASC mixes which generally does not have free lime [7]. Greater resistance to sulphuric acid of GPC in general has been stated by Thokchom et al., [20].

The AASC specimens were found to remain structurally intact without any significant variation in mass and compressive strength. Even after 90 days, the AASC specimens were structurally sound without any surface deterioration.

3.4.2 Sodium and Magnesium sulphate attack

The percentage variation in mass loss and strength loss of the AASC mixes after exposure to 5% Na_2SO_4 solutions are shown in Fig 8 and 9 respectively and that of under $MgSO_4$ exposure are shown in Fig 10 and 11 respectively. The maximum reduction in the mass of the AASC mixes was found to be 2.54% and 4.57% under Na and Mg sulphate solutions respectively and that of the compressive strength was observed to be 8.06% and 13.15% respectively. Some of the past researches [21-23] showed that there is a gain in the mass of the specimens under Na sulphate solution, whereas the present investigation shows a slight reduction in the mass under Na sulphate environment. This may be due to the ambient curing condition followed in this work unlike elevated curing techniques adopted in the past researches, where the liquid present in the pores will be evaporated leaving the pores to be filled by the sulphate attacking agents resulting in mass gain [23]. The gain in the mass is also associated with the expansion of the specimens, when the pores are filled with sulphate attacking agents like gypsum and ettringite [24].

It has been noted that the percentage reduction in mass and compressive strength decreases with the increase in the NaOH concentration. High concentration of NaOH might be the reason for the arrangement of increasing amount of dense sodium-alumino-silicate gel in the network and rate of geopolymerization thereby improved performance. The absence of Gypsum & Ettringite results in no significant reduction in mass loss and strength degradation of the AASC mixes. The specimens were found to maintain their integrity with no distress observed on the surface when examined visually. The specimens were subjected to increased amount of distress in $MgSO_4$ solution comparing to Na_2SO_4 solution. After 90 days of exposure, there were some cracks on the corners were observed and the degradation might be due to the presence of Gypsum and the

248 release of alkalis from geopolymers into the solution in AASC. No formation of ettringite was
249 observed as the slag is rich in aluminum that is not in the form available for the reaction.
250
251 Fig 12 shows the exposed specimens under Na and Mg sulphate environment after 90 days. As
252 the percentage variation in mass of the AASC specimens exposed to sodium and magnesium
253 sulphate solutions are negligible, this cannot be taken as a reliable sign of deterioration by
254 sulphate attack. Conversely, there is a considerable reduction in the compressive strength was
255 observed with the sulphate exposure which can be taken as a measurable indicator of
256 deterioration. It was observed that the specimens immersed in Na_2SO_4 solution shows exposition
257 of grains alone whereas the specimens in $MgSO_4$ solutions shows both exposition and dissolved
258 aluminosilicate matrix, which implies the rigorousness of Mg ions [25].
259
260 **3.5 Chloride penetration**
261 **3.5.1 Chloride content profile**
262 Various strategies are available to assess the chloride ion diffusion in OPC concretes, frequently
263 offers an electrical field to drive the chloride ion transportation in the samples. At the point when
264 these techniques are utilized to assess the geopolymer frameworks, the electrical potential is
265 required to accelerate the ion transportation through the pore system because of increased Na^+
266 concentration present in the pore arrangement [26]. Therefore, the chloride diffusion mechanism
267 can be affected altogether. In this way, these accelerated tests are viewed as temperamental,
268 when contrasting frameworks and generously diverse pore arrangements [27]. The long term
269 investigation of chloride ion penetration in AASC mixes was attempted as the accelerated
270 methods were proved to be futile, since a rise in temperature during test was exhibited in

geopolymer specimens is against the Ohm's law [28]. Salt ponding technique can be used to assess the chloride ion penetration under non-accelerated conditions as per ASTM C1202 (*Standard test method for electrical indication of concrete's ability to resist chloride ion penetration, ASTM International, 2008*). The chloride content profiles of the AASC mixes are shown in Fig 13 to 15.

From the Figs, it has been observed that the reduced absorption capacity of the AASC mixes was mainly due to the high tortuous nature of the aluminosilicate phase of AASC than the porous microstructure of fly ash based geopolymer mixes [19]. The reason that related to the reduced porosity and pore sizes and improved tortuosity has been discussed at the above. The chloride ingression can also be further reduced with the extended age of curing [27] due to the reduced permeability and sorptivity. The results reveal that the increase in the NaOH concentration reduced the chloride ion diffusion. The increase in the NaOH concentration leads to the increase in the compressive strength of geopolymer mixes due to dense matrix, thereby reduction in the porosity resulting in less chloride ingression. Generally, alkali with high concentration dissolves high proportion of source material due to higher degree of geopolymerization resulting in dense microstructure in the system, thereby reducing the chloride ion ingression in AASC mixes with higher NaOH concentration. In general, the mixes made with AAS has almost zero $Ca(OH)_2$ [29] and pores of lesser size than OPC results in lower permeability, results in protection from the ingress of deleterious ions.

3.5.2 Chloride Diffusion Coefficient (D_c)

The chloride profile from the tested specimens was used to compute the chloride diffusion coefficient (CDC) and the results are detailed in Table 3. In the present study, the chloride diffusion coefficient (D_c) has been computed using Fick's second law of diffusion which is given by Eq. (2).

$$\frac{\partial C}{\partial t} = D_c \frac{\partial^2 C}{\partial x^2} \qquad (2)$$

where, C is the concentration of chloride ions as a function of distance x, at any time t, and D_c is the diffusion coefficient.

This is helpful in predicting the time required for protecting the concrete from corrosion presuming a threshold rate in depassivating the steel reinforcement and thereby the initiation of corrosion. The CDC values were found to reduce with the increase in the NaOH concentration and slag content. The improved resistance to chloride penetration is mainly due to the refined pore structure, which confines the diffusion of chloride ions into the samples [30]. The CDC values were also observed to get reduced with the increase in the slag content. This is due to the high refinement of pore microstructure with increased amount of slag content thereby reducing the ingress of aggressive agents. Due to the very low CDC values for AASC mixes, it can be accepted that the time required for depassivation of steel in AASC is higher than cement concrete.

4 Conclusions

By analyzing the concrete mixes prepared at a range of sodium hydroxide concentration and binder content, using alkali activated slag as binder, the mechanical properties and behavior

under aggressive environment have been determined. From the experimental results obtained, the following conclusions can be made:

1. The compressive strength of the AASC mixes increases with the increase in the NaOH concentration.
2. The AASC mixes shows superior mechanical and durability properties under aggressive environment.
3. There is less evident of microcracking was observed in AASC mixes even at higher binder content, which is mainly due to the lower amount of heat release in AASC mixes comparing to OPC mixes.
4. The performance of the AASC mixes was also improved with the increase in the NaOH concentration which is mainly due to the improved polymerization products at higher concentration.
5. The excellent resistance of the AASC mixes under acid environment is mainly due to the very low calcium oxide content and that of the sulphate environment is mainly due to the absence of sulphate deteriorating factors.
6. The reduced pore size and improved tortuous nature of AAS confines the diffusion of chloride ions into the samples.
7. In general, the mixes made with AAS has almost zero calcium hydroxide and pores of lesser size than OPC results in lower permeability, results in protecting the concrete from the ingress of deleterious ions.

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Table 1. Chemical composition of GGBFS

Oxide	CaO	SiO_2	Al_2O_3	MgO	SO_3	Fe_2O_3	Na_2O	K_2O
GGBFS (%)	36.77	30.97	17.41	9.01	1.82	1.03	0.69	0.46

485 Table 2. Mix proportioning of the AASC mixes

Mix label	Slag content	Fine aggregate	Coarse aggregate	NaOH solution	Na_2SiO_3 solution
	Mix proportions (kg/m^3)				
AAS350	350	450	1422	58.33	116.67
AAS400	400	432	1368	66.67	133.33
AAS450	450	415	1313	75	150

486

487

Table 3. Chloride Diffusion Coefficient (CDC) results of the AASC mixes

Slag content (kg/m^3)	Chloride Diffusion Coefficient in m^2/s (x 10^{-12})		
	AAS10M	AAS12M	AAS14M
350	2.64	1.84	1.61
400	1.81	1.57	0.88
450	1.56	0.91	0.80

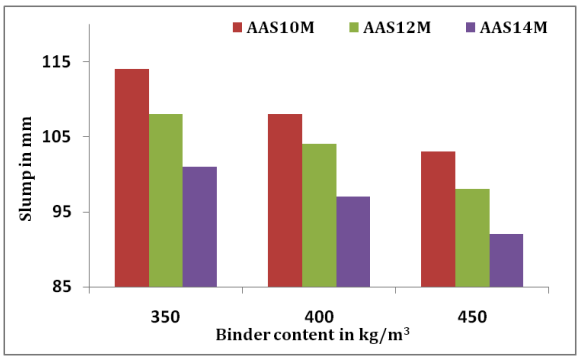


Fig 1. Workability results of the AASC mixes

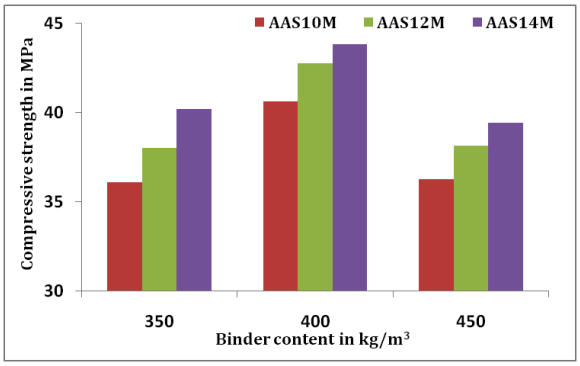


Fig 2. 7 days compressive strength results of the AASC mixes

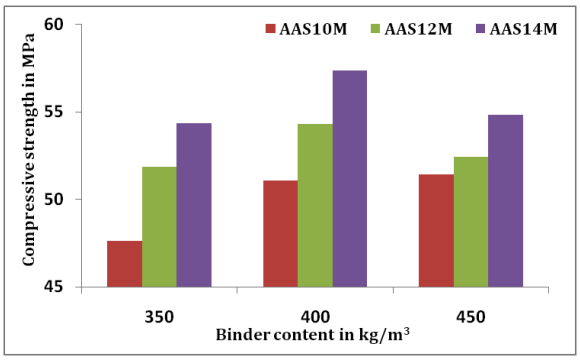
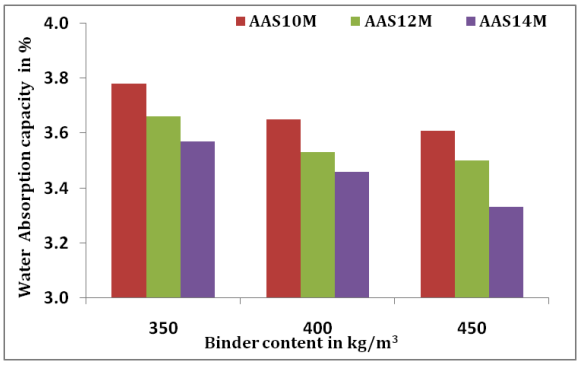


Fig 3. 28 days compressive strength results of the AASC mixes

500 

501 Fig 4. Water absorption capacity results of the AASC mixes

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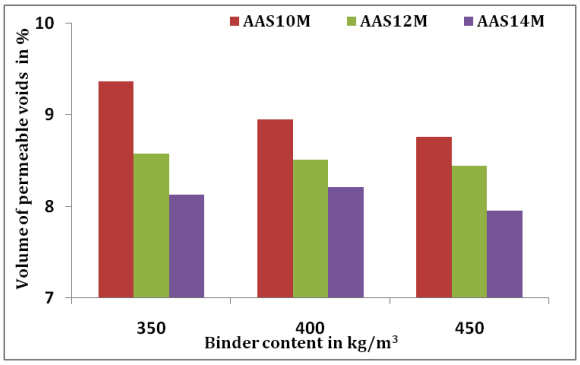


Fig 5. Volume of permeable voids results of the AASC mixes

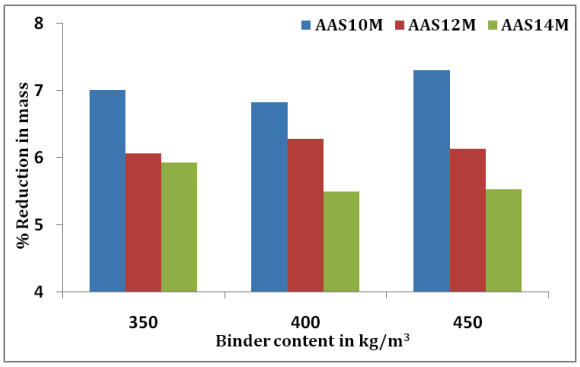


Fig 6. Variation in mass loss of the AASC mixes under H_2SO_4 attack

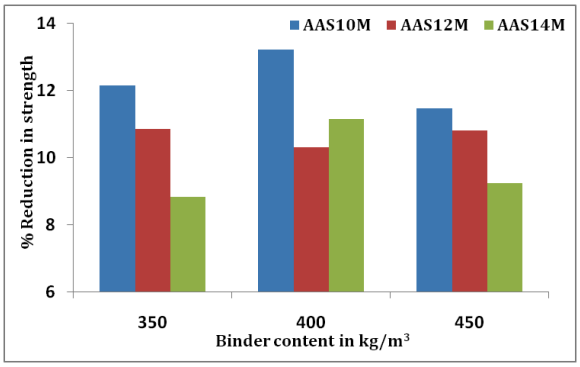


Fig 7. Variation in strength loss of the AASC mixes under H_2SO_4 attack

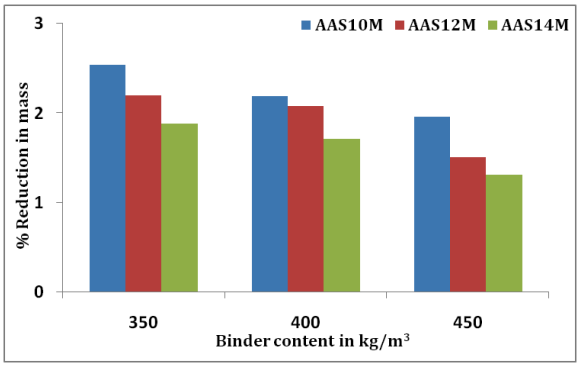


Fig 8. Variation in mass loss of the AASC mixes under Na_2SO_4 attack

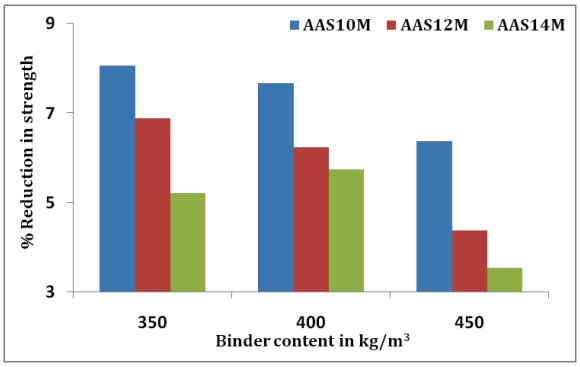


Fig 9. Variation in strength loss of the AASC mixes under Na_2SO_4 attack

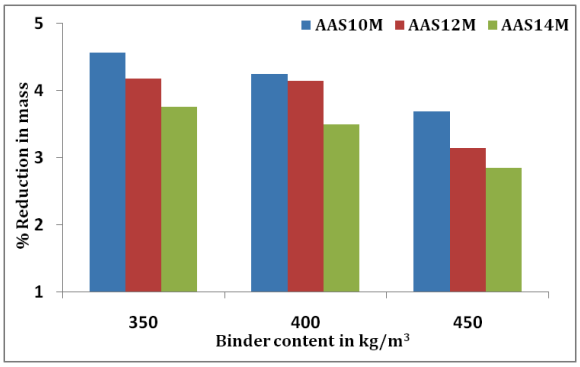


Fig 10. Variation in mass loss of the AASC mixes under $MgSO_4$ attack

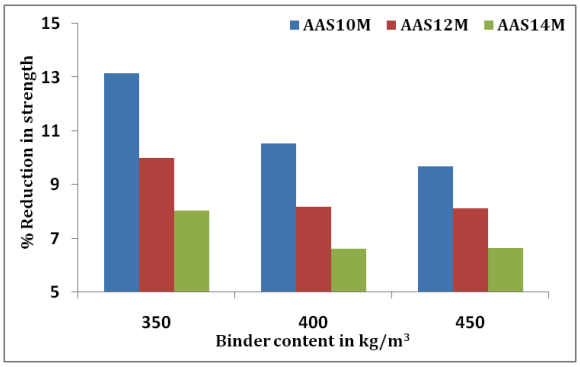
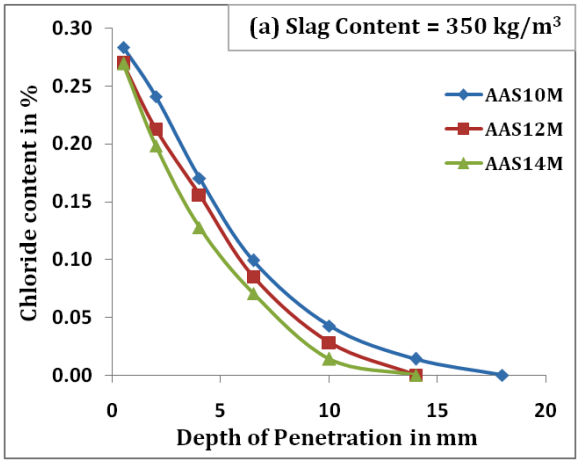


Fig 11. Variation in strength loss of the AASC mixes under $MgSO_4$ attack

Fig 12a. Specimens subjected to Na_2SO_4 exposure

Fig 12b. Specimens subjected to $MgSO_4$ exposure



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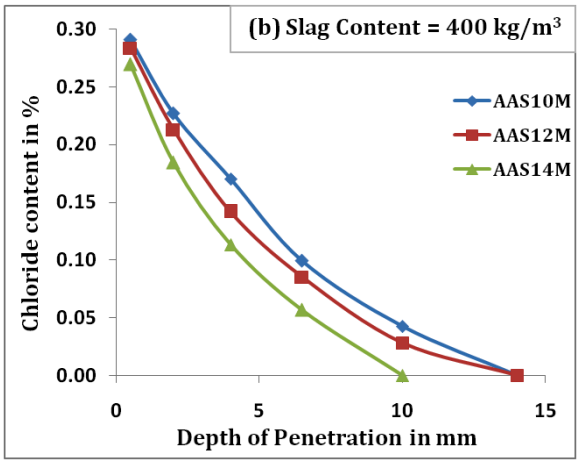

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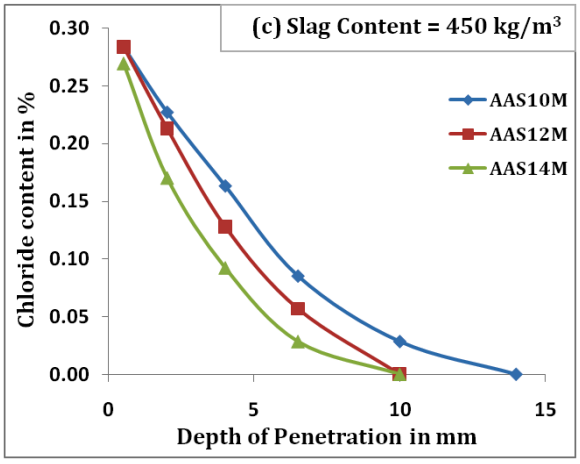

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535 Fig 13. Chloride content profile for the AASC mixes with L/B = 0.45

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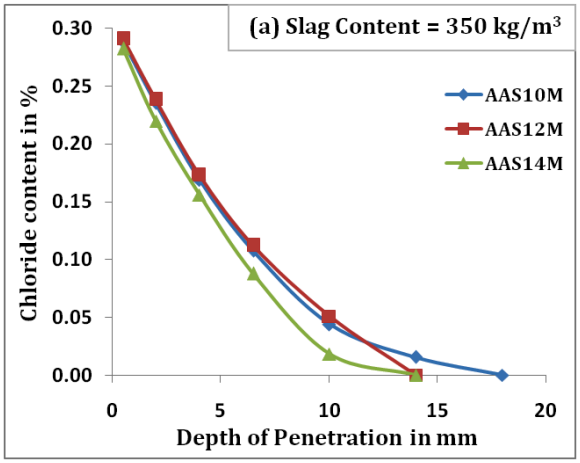

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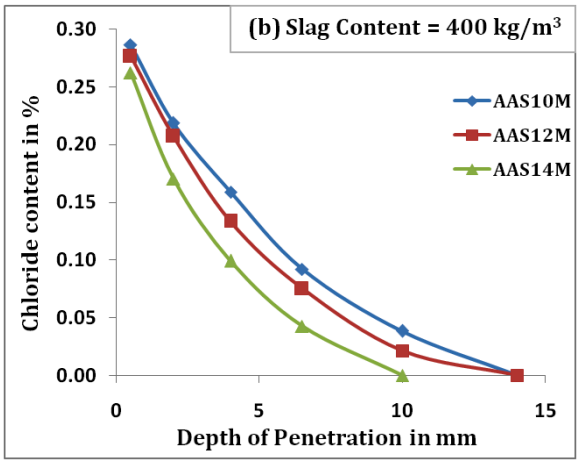

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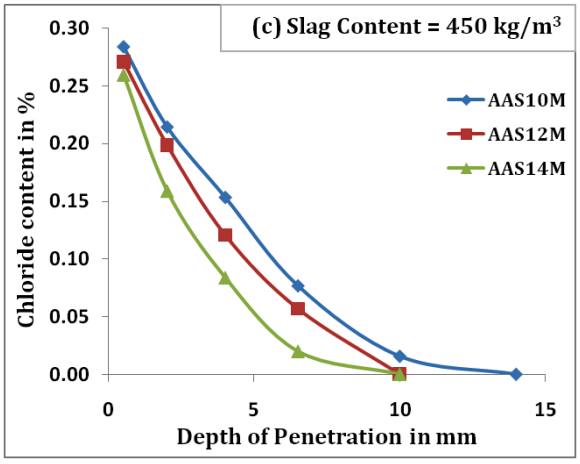

541

542 Fig 14. Chloride content profile for the AASC mixes with L/B = 0.50

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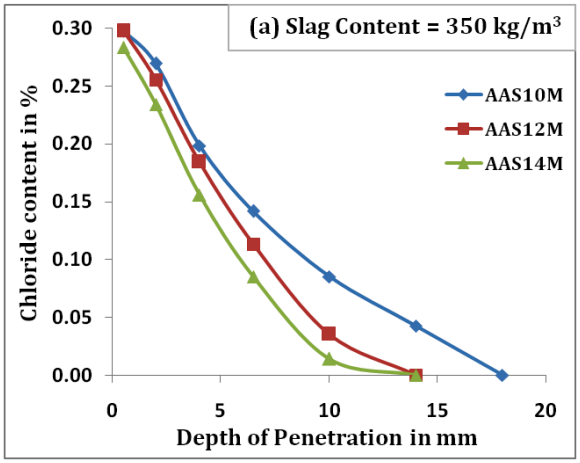

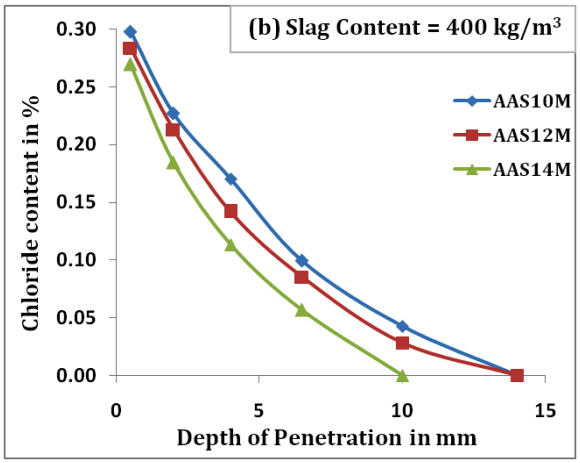

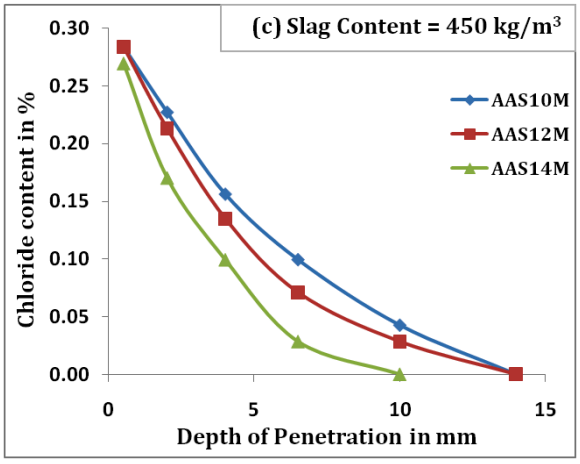

Fig 15. Chloride content profile for the AASC mixes with L/B = 0.55