

ENGINEERING PROPERTIES OF ALKALI ACTIVATED MORTAR

Thesis Submitted For Partial Fulfillment of

Master Degree In Civil Engineering

By

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CERTIFICATE

This is to certify that this thesis entitled “**Engineering properties of Alkali Activated Mortar**” has been prepared by **Sougata Das (Class Roll No. –001410402024)** for partial fulfillment of the requirements for the award of Master Degree in Civil Engineering (MCE), is a record of research work carried out under my supervision and guidance.

Neither this thesis nor any part of the thesis has been submitted for any degree/diploma or any other academic award anywhere before.

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List of Tables

Table No.	Description	Page No.
Table 3.3.12.	Summary of Experimental programme.	36
Table 3.4.1.	Details of mixes for AAGGBS(Alkali activated slag) test specimens	37
Table 3.5.2.	Details of mixes for AAGGBSA (Alkali activated slag+ admixtures) test specimens	39
Table 3.4.3.	Details of mixes for AAGGBSA test specimens for temperature study	41
Table 3.4.4.	Details of mixes for AAGGBSFA (Alkali activated slag+Fly ash+ Admixture) test specimens	43
Table 3.4.5.	Details of mixes for AASFA (Alkali activated slag+Fly ash+ Admixture) test specimens for temperature study	44
Table 3.4.6.	Details of mixes for CSC (portland slag cement + admixture) test specimens	45
Table 3.4.7.	Details of mixes for CSC (Portland slag cement + Admixture) test specimens exposed to high temperature	46
Table 4.2.1.1.	Flow values for 4%, 6% & 8% Na ₂ O in presence and absence of Admixtures	48
Table 4.2.2.1.	Water Absorption vs. Percentage Na ₂ O content for varying Water/GGBS ratio in presence and absence of admixture	52
Table 4.2.3.1.	Apparent Porosity vs. Percentage Na ₂ O content for varying Water/GGBS ratio in presence and absence of admixture	54
Table 4.2.4.1.	Sorptivity vs. Percentage Na ₂ O content for varying Water/GGBS ratio in presence and absence of admixture	64
Table 4.2.5.1.	Compressive Strength vs. Percentage Na ₂ O content for varying Water/GGBS ratio in presence and absence of admixture	64
Table 4.2.6.1	Split Tensile Strength vs. Percentage Na ₂ O content for varying Water/GGBS ratio in presence and absence of admixture	67
Table 4.2.7.1.	UPV vs. Percentage Na ₂ O content for varying Water/GGBS ratio in presence and absence of admixture	68

List of Tables Contd...

Table No.	Description	Page No.
Table 4.3.1.1 (a).	Percentage of weight loss vs. Temperature levels(2 hrs duration) for different mortar types	73
Table 4.3.1.1 (b).	Percentage of weight loss vs. Temperature levels(4 hrs duration) for different mortar types	74
Table. 4.3.2.1 (a).	Water Absorption vs. Temperature levels(2 hrs duration) for different mortar types	78
Table 4.3.2.1 (b).	Water Absorption vs. Temperature levels(4 hrs duration) for different mortar types	78
Table 4.3.3.1(a).	Apparent porosity vs. Temperature levels(2 hrs duration) for different mortar types	82
Table 4.3.3.1(b).	Apparent porosity vs. Temperature levels(4 hrs duration) for different mortar types	82
Table 4.3.4.1.	Sorptivity vs. Temperature levels(2 hrs duration) for different mortar types	84
Table 4.3.4.1.	Sorptivity vs. Temperature levels(4 hrs duration) for different mortar types	85
Table 4.3.5.1.	Compressive Strength vs. Temperature levels(2 and 4 hrs duration) for different mortar types	89
Table 4.3.6.1.	Split Tensile Strength vs. Temperature levels(2 and 4 hrs duration) for different mortar types	92
Table 4.3.7.1.	UPV values vs. Temperature levels (2 and 4 hrs duration) for different mortar types	96

List of Figures

Table No.	Description	Page No.
Fig 1.2.2.1.	Theoretical model for the reaction mechanism in alkali-activated slag	7
Fig. 1.2.3.1.	TEM micrographs of (a) anhydrous slag (b) slag hydrated with NaOH for 7 days (c) slag hydrated with waterglass for 7 days	8
Fig 1.2.3.2.	Structural model for an Al-containing C S H gel (a) linear chains; (b) linear chains with occasional cross-linking, forming planes	9
Fig.1.4.1	Application of Alkali Activated Composites	10
Fig.2.8.1.	Relationship between residual compressive strength and water absorption	17
Fig.2.8.2.	Relationship between residual compressive strength and porosity	17
Fig2.8.3.	Relationship between residual compressive strength and sorptivity	17
Fig 2.10.1	SEM micrograph of alkali-activated specimens using 3:3 sodium hydroxide to sodium silicate	19
Fig.2.11.1.	Strength development of AASF mortars at 28 days age	20
Fig 3.2.1.	Ground Granulated Blast furnace Slag	24
Fig 3.2.2.	Sodium Silicate solution, Sodium Hydroxide Platelets and their solution	24
Fig 3.2.3.	Hobart Mixer	26
Fig 3.2.4.	Preparation of PSC Mortar	26
Fig3.3.1(a).	Mini Slump Cone with Flow Table	27
Fig3.3.1(b).	Flow measurement	27
Fig.3.3.1(c).	Vicat Apparatus	28

List of Figures contd...

Table No.	Description	Page No.
Fig.3.3.2.	Arrangement to measure Apparent Porosity	28
Fig-3.3.3.	Digital compression testing machine	29
Fig-3.3.4.	Split tensile strength testing	29
Fig. 3.3.5.	Setup for water sorptivity test	30
Fig 3.3.6.	Setup for water sorptivity test	30
Fig 3.3.7.	Ultrasonic Pulse Velocity (UPV) Test	31
Fig 3.4.8.	Muffle furnace	32
Fig.3.4.9.	MIP testing machine	32
Fig 3.10.9.	A photographs of X-ray Diffractometer	33
Fig 3.10.11(a).	JEOL JSM 5200 Scanning electron microscope	34
Fig 3.10.11(b).	Sample preparation in Auto fine coater	35
Fig.4.2.1.1 (a).	Flow values for 4% Na₂O in presence and absence of Admixtures	49
Fig 4.2.1.1(b).	Flow values for 6% Na₂O in presence and absence of Admixtures	49
Fig.4.2.1.1(c).	Flow values for 8% Na₂O in presence and absence of Admixtures	50
Fig. 4.2.1.1(d).	Comparison of Flow values for 4%,6% & 8% Na₂O in presence and absence of Admixtures	51
Fig. 4.2.2.1(a).	Variation of water absorption using 4%, 6% & 8% Na₂O with and without Admixture for Water/GGBS=0.33	52

List of Figures contd...

Table No.	Description	Page No.
Fig. 4.2.2.1(b).	Variation of water absorption for 4%, 6% & 8% Na ₂ O with and without Admixture for Water/GGBS ratio = 0.35	52
Fig. 4.2.2.1(c).	Variation of water absorption for 4% ,6% & 8% Na ₂ O with and without Admixture for Water/GGBS ratio= 0.38	53
Fig. 4.2.2.1(d).	Variation of Water Absorption for different Na ₂ O % and Water/GGBS ratio with and without Admixture.	53
Fig.4.2.3.1 (a).	Variation of apparent porosity for 4% 6% & 8% Na ₂ O with and without Admixture for Water/GGBS= 0.33	55
Fig.4.2.3.1 (b).	Variation of apparent porosity for 4% 6% & 8% Na ₂ O with and without Admixture for Water/GGBS = 0.35	55
Fig.4.2.3.1(c).	Variation of apparent porosity using 4%, 6% & 8% Na ₂ O with and without Admixture for Water/GGBS = 0.38	56
Fig. 4.2.3.1(d).	Variation of App. Porosity for different Na ₂ O % and Water/GGBS ratio with and without Admixture	56
Fig 4.2.4.1(a).	Sorptivity for 4% Na ₂ O at Water/GGBS ratio=0.33	57
Fig 4.2.4.1(b).	Sorptivity for 6% Na ₂ O at Water/GGBS ratio=0.33	58
Fig 4.2.4.1(c).	Sorptivity for 8% Na ₂ O at Water/GGBS ratio =0.33	58
Fig 4.2.4.1(d).	Sorptivity for Water/GGBS ratio = 0.33 in presence and absence of Admixture	59
Fig 4.2.4.1(e).	Sorptivity for 4% Na ₂ O at Water/GGBS ratio = 0.35	59
Fig 4.2.4.1(f).	Sorptivity for 6% Na ₂ O at Water/GGBS ratio = 0.35	59
Fig 4.2.4.1(g).	Sorptivity for 8% Na ₂ O at Water/GGBS ratio = 0.35	60
Fig 4.2.4.1(h).	Sorptivity for Water/GGBS ratio = 0.35 with and without Admixture	60
Fig 4.2.4.1(i).	Sorptivity for 4% Na ₂ O at Water/GGBS ratio = 0.38	60

List of Figures contd...

Table No.	Description	Page No.
Fig 4.2.4.1(j).	Sorptivity for 6% Na ₂ O at Water/GGBS ratio = 0.38	61
Fig 4.2.4.1(k).	Sorptivity for 8% Na ₂ O at Water/GGBS ratio = 0.38	61
Fig 4.2.4.1(l).	Sorptivity for Water/GGBS ratio =0.38 with and without Admixture	61
Fig.4.2.4.1(m).	Sorptivity at Water/GGBS ratio of 0.33 in presence and absence of admixtures for varying Na ₂ O content	62
Fig. 4.2.4.1(n).	Sorptivity at Water/GGBS ratio of 0.35 in presence and absence of admixtures for varying Na ₂ O content	62
Fig. 4.2.4.1(c).	Sorptivity at Water/GGBS ratio of 0.38 in presence and absence of admixtures for varying Na ₂ O content.	62
Fig. 4.2.5.1(a)	Compressive strength vs. Water/GGBS ratio for 4% Na ₂ O content in presence and absence of admixture	65
Fig. 4.2.5.1(b)	Compressive strength vs. Water/GGBS ratio for 6% Na ₂ O content in presence and absence of admixture	65
Fig. 4.2.5.1(c)	Compressive strength vs. Water/GGBS ratio for 8% Na ₂ O content in presence and absence of admixture	65
Fig. 4.2.5.1(d).	Compressive strength vs. Water/GGBS ratio at varying Na ₂ O content in presence and absence of Admixtures.	66
Fig.4.2.6.1.	Split Tensile Strength vs. Percentage Na ₂ O content for different Water/GGBS ratio in presence and absence of Admixture	67
Fig.4.2.7.1(a).	UPV vs. % Na ₂ O(in presence and absence of admixture for Water/GGBS ratio =0.33)	68
Fig.4.2.7.1(b).	UPV vs. % Na ₂ O (in presence and absence of admixture for Water/GGBS ratio = 0.35)	69
Fig.4.2.7.1(c).	UPV vs. % Na ₂ O (in presence and absence of admixture for Water/GGBS ratio =0.38)	69
Fig.4.2.8.1(a).	Apparent Porosity & Water Absorption for different mortar types	70

List of Figures contd...

Table No.	Description	Page No.
Fig. 4.2.8.1(b).	Sorptivity of different mortar types	70
Fig.4.2.8.2.1(a).	28-days Compressive Strength for different mortar types	71
Fig.4.2.8.2.1(a).	28-days Compressive Strength for different mortar types	72
Fig.4.3.1.1(a) .	Percentage loss of weight vs. Temperature levels (Heating duration=2hrs) for different mortar types	73
Fig.4.3.1.1(a) .	Percentage loss of weight vs. Temperature levels (Heating duration=4hrs) for different mortar types	74
Fig.4.3.1.2(a).	Percentage loss of weight vs. Duration of heating for AAGGBS mortar	75
Fig.4.3.1.2(a).	Percentage loss of weight vs. Duration of heating for AASF(Slag: Fly ash=80:20) mortar	75
Fig.4.3.1.2(a).	Percentage loss of weight vs. Duration of heating for AASF(Slag: Fly ash=70:30) mortar	75
Fig.4.3.1.2(a).	Percentage loss of weight vs. Duration of heating for AASF(Slag: Fly ash=60:40) mortar	76
Fig.4.3.1.2(a).	Percentage loss of weight vs. Duration of heating for SC(60:40) mortar	76
Fig.4.3.2.1(a).	Water absorption vs. Temperature Levels (2 hrs duration of heating) for different types of mortars	77
Fig.4.3.2.1(b).	Water absorption vs. Temperature Levels (4 hrs duration of heating) for different types of mortars	77
Fig. 4.3.2.2(a).	Water Absorption vs. Duration of heating for AAGGBS mortar	78

List of Figures contd...

Table No.	Description	Page No.
Fig. 4.3.2.2(b).	Water absorption vs. Duration of heating for AASF(Slag: Fly Ash=80:20) mortar	79
Fig. 4.3.2.2(c).	Water absorption vs. Duration of heating for AASF(Slag: Fly Ash=70:30) mortar	79
Fig. 4.3.2.2(d).	Water absorption vs. Duration of heating for AASF(Slag: Fly Ash=60:40) mortar	79
Fig. 4.3.2.2(e).	Water absorption vs. duration of heating for CSC mortar	80
Fig.4.3.3.1(a).	Apparent porosity vs. Temperature Levels (2 hrs duration of heating) for different types of mortars	81
Fig.4.3.3.1(b).	Apparent porosity vs. Temperature Levels (4 hrs duration of heating) for different types of mortars	82
Fig. 4.3.3.2(a).	Apparent Porosity vs. Duration of heating for AAGGBS mortar	82
Fig. 4.3.3.2(b).	Apparent Porosity vs. Duration of heating for AASF (Slag: Fly Ash=80:20) mortar	83
Fig. 4.3.3.2(c).	Apparent Porosity vs. Duration of heating for AASF(Slag: Fly Ash=70:30) mortar	83
Fig. 4.3.3.2(d).	Apparent Porosity vs. Duration of heating for AASF (Slag: Fly Ash=60:40) mortar	83
Fig. 4.3.3.2(e).	Apparent Porosity vs. Duration of heating for CSC mortar	84
Fig.4.3.4.1(a).	Sorptivity vs. Temperature Levels (2 hrs duration of heating) for different types of mortars	85
Fig.4.3.4.1(b).	Sorptivity vs. Temperature Levels (4 hrs duration of heating) for different types of mortars	85
Fig.4.3.4.2(a)	Sorptivity vs. Duration of heating for AAGGBS mortar	86

List of Figures contd...

Table No.	Description	Page No.
Fig.4.3.4.2(b)	Sorptivity vs. Duration of heating for AASF(Slag: Fly Ash=80:20) mortar	86
Fig.4.3.4.2(c)	Sorptivity vs. Duration of heating for AASF(Slag: Fly Ash=70:30) mortar	87
Fig.4.3.4.2(b)	Sorptivity vs. Duration of heating for AASF(Slag: Fly Ash=60:40) mortar	87
Fig.4.3.4.2(b)	Sorptivity vs. Duration of heating for CSC mortar	88
Fig. 4.3.5.1(a).	28 days Compressive Strength vs. Temperature Levels (2hr duration of heating) for different mortar types	89
Fig. 4.3.5.1(b).	28 days Compressive Strength vs. Temperature Levels (4hr duration of heating) for different mortar types	90
Fig. 4.3.5.2(a).	Compressive Strength vs. Duration of heating for AAGBS mortar	90
Fig. 4.3.5.2(b).	Compressive Strength vs. Duration of heating. for AASF(Slag: Fly Ash=80:20) mortar	91
Fig. 4.3.5.2(c).	Compressive Strength vs. Duration of heating. for AASF(Slag: Fly Ash=70:30) mortar	91
Fig. 4.3.5.2(d).	Compressive Strength vs. Duration of heating. for AASF(Slag: Fly Ash=60:40) mortar	91
Fig. 4.3.5.2(e).	Compressive Strength vs. Duration of heating. for CSC mortar	92
Fig. 4.3.6.1(a).	Split Tensile Strength vs. Temperature Levels (2hr duration of heating) for different mortar types	93
Fig. 4.3.6.1(b).	Split Tensile Strength vs. Temperature Levels (4hr duration of heating) for different mortar types	93
Fig. 4.3.6.2(a)	Split Tensile Strength vs. Duration of heating for AAGGBS mortar	94
Fig. 4.3.6.2(b)	Split Tensile Strength vs. Duration of heating for AASF(Slag: Fly Ash=80:20) mortar	95

List of Figures contd...

Table No.	Description	Page No.
Fig. 4.3.6.2(c)	Split Tensile Strength vs. Duration of heating for AASF(Slag: Fly Ash=70:30) mortar	95
Fig. 4.3.6.2(d)	Split Tensile Strength vs. Duration of heating for AASF(Slag: Fly Ash=60:40) mortar	95
Fig. 4.3.6.2(e)	Split Tensile Strength vs. Duration of heating for SC mortar	95
Fig..4.3.7.1(a).	UPV vs. Temperature Levels (2 hrs duration of heating) for different mortars types	96
Fig..4.3.7.1(b).	UPV vs. Temperature Levels (4 hrs duration of heating) for different mortars types	97
Fig.4.3.7.2.(a).	UPV vs. Duration of heating for AAGGBS mortar.	97
Fig.4.3.7.2.(b).	UPV vs. Duration of heating for AASF (Slag: Fly Ash=80:20) mortar	98
Fig.4.3.7.2.(a).	UPV vs. Duration of heating for AASF (Slag: Fly Ash=70:30) mortar.	98
Fig.4.3.7.2.(a).	UPV vs. Duration of heating for AASF(Slag: Fly Ash=60:40) mortar.	99
Fig.4.3.7.2.(a).	UPV vs. Duration of heating for CSC mortar.	99
Fig.4.4.1.1(a).	SEM image (500 X) of Alkali activated slag with Admixture	100
Fig.4.4.1.1(b).	SEM image (2000 X) of Alkali activated slag with Admixture	100
Fig.4.4.1.1(c).	SEM image (500 X) of Alkali activated slag without Admixture	101
Fig.4.4.1.1(d).	SEM image (2000 X)of Alkali activated slag without Admixture	102
Fig.4.4.2.1(a).	SEM image (500 X) of Normal Slag Cement with Admixture	103
Fig.4.4.2.1(b).	SEM image (1000 X) of Normal Slag Cement with Admixture	102
Fig.4.4.2.1 (a).	SEM image (250X) of Alkali Activated GGBS mortar exposed to 800°C for 4hrs.	103

List of Figures contd...

Table No.	Description	Page No.
Fig.4.4.2.1 (b).	SEM image(500X) of Alkali Activated GGBS mortar exposed to 800°C for 4hrs	103
Fig.4.4.2.1(c).	SEM image (1000X) of Alkali Activated GGBS mortar exposed to 800°C for 4hrs	103
Fig.4.4.2.1(d).	SEM image (3000X) of Alkali Activated GGBS mortar exposed to 800°C for 4hrs	104
Fig.4.4.2.1(c).	SEM image (5000X) of Alkali Activated GGBS mortar exposed to 800°C for 4hrs	104

Abbreviations used:

AAC	Alkali Activated Composites
AAS	Alkali Activated Slag
AAGGBS	Alkali Activated Ground Granulated Blast Furnace Slag
AASF	Alkali Activated Slag & Fly-ash
GGBS	Ground Granulated Blast furnace Slag
PSC	Portland Slag Cement
CSC	Conventional Slag Cement
UPV	Ultrasonic Pulse Velocity
SEM	Scanning Electron Microscope
w/c	Water cement ratio
w/b	Water binder ratio
MIP	Mercury Intrusion Porosimetry
XRD	X-ray diffraction
Admix.	Admixture
AAGGBSF	Alkali Activated Ground Granulated Blast Furnace Slag and Fly Ash
AAGGBSA	Alkali Activated Ground Granulated Blast Furnace Slag with Admixture
AAGGBSFA	Alkali Activated Ground Granulated Blast Furnace Slag and Fly Ash with Admixture
TEM	Transmission Electron Microscopy
XRF	X-Ray Fluorescence

Content

	Page no.
Certificates	
Acknowledgement	
List of Tables	
List of Figures	
Abbreviations used	
Synopsis	
1. Introduction	1-12
1.1. Preamble	1
1.2. Alkali Activated Composites(AAC) terminology and chemistry	3
1.3. Role of Constitutive material in Alkali Activated Composites (AAC)	9
1.4. Application	10
1.5. Objective of present research	11
2. Review of Existing literature	13-22
2.1. Preamble	13
2.2. Literature related to Alkali activated composites (AAC)	
2.2.1. Environmental factors.	13
2.2.2. Chemistry of AAC	14
2.2.3. Properties of AAGGBS	16
2.2.4. Effect of Admixtures on AAC	16
2.2.5. Effect of High Temperature on AAC	18
2.2.6. Properties of GGBS/Fly-Ash AAC blends	20
2.3. Summary on literature review	22
3. Experimental investigation	23-45
3.1. Preamble	23
3.2. Manufacturing process of AAC mix	23
3.3. Testing and characterizing AAC	
3.3.1. Work ability and Setting Time	27
3.3.2. Bulk density and Apparent porosity	28
3.3.3. Compressive strength	29

3.3.4. Split tensile strength	29
3.3.5. Water Absorption	30
3.3.6. Water Sorptivity	30
3.3.7. Ultrasonic Pulse velocity	31
3.3.8. Heating of samples in maple furnace	32
3.3.9. MIP Analysis	32
3.3.10.X-ray diffraction (XRD)	33
3.3.11. Scanning Electron microscopy	34
3.3.12. Summary of Experimental Programme	36
3.4. Alkali Activated Composite mortars(AAS & AASF) and Normal Slag Cement(SC) mortars mixes	37
4. Results and Discussion	47-104
4.1. Preamble.	47
4.2. Physico-Mechanical properties of Alkali Activated mortar	
4.2.1. Workability	48
4.2.1.1. Effect of Na ₂ O content in presence and absence of Admixtures for different Water/GGBS ratio	48
4.2.2. Water Absorption	
4.2.2.1. Effect of Na ₂ O content in presence and absence of Admixtures for different Water/GGBS ratio	51
4.2.3. Apparent porosity.	
4.2.3.1. Effect of Na ₂ O content in presence and absence of Admixtures for different Water/GGBS ratio	54
4.2.4. Sorptivity	
4.2.4.1. Effect of Na ₂ O content in presence and absence of Admixtures for different Water/GGBS ratio	57
4.2.5. Compressive Strength	
4.2.5.1. Effect of Na ₂ O content in presence and absence of Admixtures for different Water/GGBS ratio	64
4.2.6. Split-Tensile Strength	
4.2.6.1. Effect of Na ₂ O content in presence and absence of Admixtures for different Water/GGBS ratio	67
4.2.7. Ultrasonic Pulse Velocity Test.	
4.2.7.1. Effect of Na ₂ O content in presence and absence of Admixtures for different Water/GGBS ratio	68
4.2.8. Comparison of properties of AAGGBS mortar with Alkali Activated Slag-Fly Ash mortar & Normal Slag Cement (PSC) mortar	69
4.2.8.1. Apparent Porosity ,Water Absorption & Sorptivity	69
4.2.8.2. Compressive strength & Split Tensile Strength	71

4.3.	Properties of Alkali Activated Mortars specimens Exposed to High Temperature	
4.3.1.	Loss of weight	
4.3.1.1.	Percentage loss of weight vs. Temperature Levels	73
4.3.1.2.	Percentage loss of weight vs. Duration of heating	75
4.3.2.	Water absorption	
4.3.2.1.	Water absorption vs. Temperature Levels	77
4.3.2.2.	Water absorption vs. Duration of heating	78
4.3.3.	Apparent Porosity	
4.3.3.1.	Apparent Porosity vs. Temperature Levels	81
4.3.3.2.	Apparent Porosity vs. Duration of heating	83
4.3.4.	Sorptivity	
4.3.4.1.	Sorptivity vs. Temperature Levels	84
4.3.4.2.	Sorptivity vs. Duration of heating.	86
4.3.5.	Compressive strength	
4.3.5.1.	Compressive strength vs. Temperature Levels	89
4.3.5.2.	Compressive strength vs. Duration of heating.	90
4.3.6.	Split-tensile strength.	
4.3.6.1.	Split tensile strength vs. Temperature Levels	92
4.3.6.2.	Split tensile strength vs. Duration of heating	94
4.3.7.	Ultra Sonic Pulse Velocity	
4.3.7.1.	UPV against Temperature Levels	96
4.3.7.2.	UPV against Duration of heating	97

4.4. Micro structural study (SEM)	
4.4.1. SEM of Alkali Activated GGBS	100
4.4.2. SEM of Alkali Activated GGBS exposed to high temperature	103
5. Conclusion	105-106
References	107-108
Appendix.	109-113
Appendix-I	
Composition of materials	109
Appendix-II	
Quantity calculation of Alkali activators	111
Appendix-III	
Sample calculations to get engineering properties from test results	112

Synopsis

The developing countries like India and China are one of the major cement producing countries in the world and the concrete industry is increasing rapidly to meet their demand for buildings and infrastructure. Cement industry is responsible for generating nearly 1460 million tons of carbon dioxide per year^[1]. Moreover, Portland cement consumption has grown to a great extent in the last 20 years. By 2020, overall demand is estimated to reach 3.6×10^9 tones,^[2]. All these factors encourage the study and development of new alternate binders having lower CO₂ emissions using industrial by-products. In this context, Alkali activated binders or Composites are becoming extremely popular for better engineering properties and drastic reduction of CO₂ emission.

Both the quantitative and the qualitative relations between strength and processing parameters for Alkali activated binders have not yet been properly understood. Research has been done and manufacturing technologies of Alkali activated composites (AAC) are implemented but these technologies still do not provide consistent properties. To understand exact reaction mechanism of AAC, further research is still required. Workability, Hardened property, Strength development and Durability characteristics are dependent on the composition of the ingredients used, their relative proportions, the composition and molarity of the alkali activator and the curing conditions.

The main objective of the present experimental investigation is to study the engineering properties of Alkali activated slag mortar of fresh and hardened state. Performance of admixed AAC, when exposed to high temperature has also been studied. Alkali activated mortar have been prepared by using Ground Granulated Blast furnace Slag (GGBS) as base material which was mixed with sand (Zone-II, Cement:Sand=1:1). Sodium Hydroxide (NaOH) pellets with Sodium Silicate (Na₂SiO₃-Water Glass) solution was mixed and activator solution was prepared. Workability of Alkali activated composite mix depends on amount of Na₂O content, & SiO₂ content and also on Na₂O/SiO₂ ratio, extra water content and also on dosage and type of admixtures/plasticizers added. The quantity of Na₂O content was considered as 4%, 6% and 8% by weight of GGBS. SiO₂ content of 6% by weight of GGBS was kept constant. The engineering properties have been studied in absence and presence of admixtures and an improved AAS mortar in terms of workability and strength has been achieved. A study on the effect of high temperature exposure on strength and durability of Alkali activated GGBS mortar was also conducted systematically. The mortar test specimens have been exposed to high temperature (400°C, 600°C and 800°C) for 2-hrs and 4hrs, in a Muffle furnace. The

engineering properties have been compared with the properties of specimens unexposed to high temperature. GGBS was partially replaced by Fly Ash in different proportion to get an improved performance of the composite on exposure to elevated temperature. A comparative study with conventional blast furnace slag cement based mortar, has also been made. Scanning Electron microscopy (SEM), have also been conducted to examine the microstructure of Alkali activated slag mortar and conventional cement mortar out of Portland Slag Cement. The test results are presented in tabular and graphical form and proper scientific interpretation are made. Based on test results some broad conclusions are also made and areas of future research have been indicated.

1. Introduction

1.1.Preamble

Different industrial by-products activated with a high pH alkali metal hydroxide and metal silicates solution (generally sodium silicate-based) and then cured at a certain temperatures to get a solid product of reasonably high strength and durability. These industrial by-products are generally, Fly ash, Ground granulated blast furnace slag (GGBS), Metakaolin, Sewage waste, Paper processing residues, etc. The high alkali environment breaks down Si-O chemical bonds in the glassy phase of the material. These phases then become available for reaction with the alkaline species in the liquid. Reaction products are zeolitic in nature and the activator is consumed in the reaction. The chemistry is dependent on the composition of the source material and the type of activator used.

Alkali activated material has attracted great attention in recent years due to its excellent performances such as mechanical properties, durability, thermal stability and low environmental impacts. It is usually produced by mixing alkaline activator solutions with solid raw materials. Alkali activated systems can be classified into two types: calcium and silica enriched (Ca + Si) system and alumino-silicates dominated (Si + Al) system. The represented precursor of calcium enriched system is ground granulated blast furnace slag (GGBS), and the reaction product is a C-A-S-H (I) type gel with a lower Ca/Si ratio and a high Al incorporation. While the typical starting materials of Si + Al system are class F fly ash or metakaolin, having N-A-S-H type gels with three-dimensional network as the major final product.

The characteristics of the materials used and their alkaline activators play a significant role in the hydration and micro structural development of the binders obtained, with the concomitant variations in their mechanical strength, suitably alkali-activated cements and concretes can exhibit even higher performance than Portland cement concretes. Alkaline cements are cementitious materials formed as the result of an alkaline attack on the amorphous or vitreous aluminosilicates. When mixed with alkaline activators, these materials set and harden, yielding a material with good binding properties. The strength development in Alkali activated composites depends on the chemical composition, morphology and reactivity of source materials, chemical composition of activating solution and curing process.

Alkali-activated composites have special heat resisting properties due to the following physicochemical characteristics:

- They are totally inorganic polymeric materials and therefore are incombustible.
- They have ceramic-like properties that render them resistant upon heating at high temperatures due to formation of several crystalline aluminosilicate phases with potentially high melting points ($>1100^{\circ}\text{C}$)^[3]. Therefore, they retain their structural integrity at elevated temperatures.

They have thermal conductivity values in the region of 0.1–0.3 W/m-K, ^[4] which is relatively low in comparison to the other commonly used structural building materials and almost identical or comparable to the marketable fire resistant materials.

Alkali activated fly ash composites give better heat resistant properties than alkali activated slag composite because presence of CaO decreases the refractoriness of the binary $\text{Al}_2\text{O}_3\text{-SiO}_2$. However our aim will be check its effectiveness over Blast Furnace slag system. And improve its performance by adding different additives.

1.2. Alkali Activated Composite Terminology and Chemistry

A wide variety of alkali-activated cements has been developed in the last few decades. Based on the nature of their cementitious components ($\text{CaO-SiO}_2\text{-Al}_2\text{O}_3$ system), alkaline cements may be grouped under two main categories: (1) high calcium and (2) low calcium cements. The activation pattern differs in each.

- **(Na, K)₂O-CaO-Al₂O₃-SiO₂-H₂O system, Model 1:** in this model calcium- and silicon-rich materials such as blast furnace slag ($\text{SiO}_2 + \text{CaO} > 70\%$) are activated under relatively moderate alkaline conditions. In this case the main reaction product is a C-A-S-H (calcium silicate hydrate) gel, similar to the gel obtained during Portland cement hydration, which takes up Al in its structure.

- **(Na, K)₂O-Al₂O₃-SiO₂-H₂O system, Model 2:** in this model the materials activated comprise primarily aluminum and silicon. The materials used in this second alkali activation procedure such as metakaolin or type F fly ash (from coal-fired steam power plants) have low CaO contents. In this case more aggressive working conditions are required to kick-start the reactions (highly alkaline media and curing temperatures of 60–

200°C). The main reaction product formed in this case is a three-dimensional inorganic alkaline polymer, a N-A-S-H (or alkaline aluminosilicate hydrate) gel that can be regarded as a zeolite precursor ^[3]. This gel also goes by the names of geo- or inorganic polymer among others.

Today, a third alkaline activation model, a combination of the preceding two, can also be described. Here the product would be a new type of binder known as a blended or hybrid alkaline cement, formed as the result of the alkaline activation of materials with CaO, SiO₂ and Al₂O₃ contents > 20% ^[4, 5, 6]. These materials can be divided into two groups.

- **Group A** includes materials having a low Portland cement clinker content and a high proportion (over 70%) of mineral additions ^[5]. Examples are cement + slag, cement + fly ash, cement + slag + fly ash.
- **Group B** comprises blends containing no Portland cement: blast furnace slag + fly ash, phosphorous slag + blast furnace slag + fly ash and similar. The reaction products precipitating in this third group (hybrid cements) are very complex, comprising a mix of cementitious gels, including C-A-S-H (which takes sodium into its composition) and (N,C)-A-S-H (high calcium content N-A-S-H gels)gels.

As our study is concerned with alkali activated slag concrete so model 1 will be discussed here.

1.2.1. Composition of starting materials

[(Na,K)₂O-CaO-Al₂O₃-SiO₂-H₂O system – Model 1]

The material most commonly used to prepare calcium-rich alkaline cements and concretes is blast furnace slag. This vitreous steel industry waste is formed when the acid oxides (SiO₂ and Al₂O₃) from the mix of acid clay gangue present in iron ore and sulfur ash in coke combine with the basic oxides (CaO and MgO) present in the limestone or dolomite used as fluxes during high temperature (1600°C) smelting followed by abrupt cooling to temperatures of >800°C ^[7]. The majority components in slag are CaO (35–40%), SiO₂ (25–35%), MgO (5–10%) and Al₂O₃ (5–15%), while its minority constituents include S, Fe₂O₃, MnO and K₂O (with percentages of under 1%). In other words, slag contains both network-forming anions (SiO₄)⁴⁻, (AlO₄)⁵⁻ and (MgO₄)⁶⁻ and network-modifying cations, Ca²⁺ and

Al³⁺ and Mg²⁺. On average, 90–95% of slag is vitreous (a de-polymerised calcium silicate) and the rest is a solid solution of minority crystalline phases: gehlenite (2CaO.Al₂O₃.SiO₂) and akermanite (2CaO.MgO.2SiO₂), tetragonal lattice, melilite family crystals. Slag reactivity in alkaline activation processes depends largely on the vitreous phase content, for which values of over 90% are recommended. Nonetheless, Pal et al. (2003) noted that slag with low vitreous phase contents (30–65%) could also deliver good results. The degree of depolymerisation (DP), a parameter whose value normally ranges from 1.3 to 1.5, apparently bears a direct relationship to slag reactivity.

It is calculated from the following equation ^[7]:

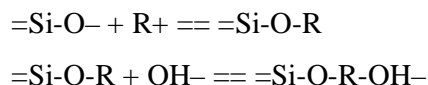
$$DP = n(\text{CaO}) - 2n(\text{MgO}) - n(\text{Al}_2\text{O}_3) - n(\text{SO}_3)/n(\text{SiO}_2) - 2n(\text{MgO}) - 0.5n(\text{Al}_2\text{O}_3)$$

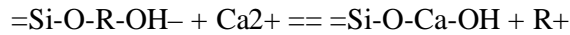
According to most of the literature, the main properties required of slag to be apt for use in activated slag cement are as listed below:

- It must be granulated or pelletized and have a vitreous phase content of >85–95%.
- It must exhibit structural disorder, for the lower the degree of polymerisation in the glass, the higher is its hydraulic activity: the degree of polymerisation depends on the (SiO₄) tetrahedra and Al and Mg coordination in the vitreous phase of the slag.
- It must be pH-basic, i.e., have a CaO + MgO/SiO₂ ratio of >1. Basic slag has a higher hydraulic potential, for the lime content in the slag controls its activation. That notwithstanding, acid slag may also be alkali-activated.
- It must be ground to a specific surface of 400–600 m²/kg. Specific surface plays an important role in the rate and intensity of the activation reaction.

1.2.2. Reaction mechanisms.

Glukhovsky and Krivenko (Glukhovsky, 1967, 1994; Krivenko, 1994)^[9,10] proposed a model that would explain the alkaline activation of silica- and lime-rich materials such as blast furnace slag by means of the series of reactions summarised below:





The alkaline cation (R^+) acts as a mere catalyser in the initial phases of hydration, via cationic exchange with the Ca^{2+} ions. These same authors believed that as the reactions advance, the alkaline cations are taken up into the structure. It has been reported that the nature of the anion in the solution also plays an instrumental role in activation, especially at early ages and in particular with regard to paste setting. One of the models that describes the reaction mechanisms (based on a model proposed by Glasser in 1990)^[11] is depicted in Figure 1.2.2.1. Slag alkaline activation forms part of a complex process that takes place by stages, in which slag destruction is followed by polycondensation of the reaction products.

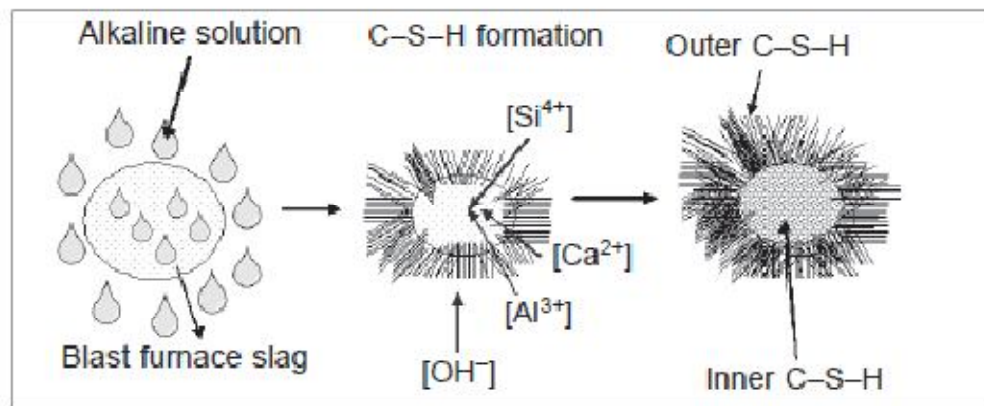


Fig 1.2.2.1.

Theoretical model for the reaction mechanism in alkali-activated slag

1.2.3. Reaction products

The majority reaction product formed in Portland cement hydration is a C-S-H-type gel to which the material primarily owes its mechanical properties. The secondary products include portlandite, ettringite and calcium mono sulfo aluminate ^[12] (Taylor,1990). The process is similar in alkaline cements. The main reaction product, a gel (whose composition and structure vary with respect to the standard C-S-H generated in OPC hydration), forms along with a series of secondary products. The type of secondary product generated depends on starting material composition, activator type and concentration, curing conditions and pH, among others.

Broadly speaking, the nature and composition of the reaction products forming during the alkaline activation of materials are among the most controversial aspects of this field of

research and the area is most in need of continued study. Nonetheless, most authors agree to the following assertions.

- The main hydration product is a calcium silicate hydrate with aluminum in its composition (C-A-S-H gel). This gel differs slightly from the gel forming in Portland cement paste, in that it has a lower C/S ratio (C/S = 0.9–1.2).

- The structure and composition of C-A-S-H gel and the presence of other secondary phases or compounds depend on the type and amount of activator used, slag structure and composition and the curing conditions in which the material hardens.

The minority phases whose formation during the alkaline activation of slag has marshaled the widest consensus are listed below.

- A phase known as hydrotalcite ($Mg_6Al_2CO_3(OH)_{16} \cdot 4HO$) has been detected in slag activated with NaOH and waterglass^[13,14](Chenget al., 1992; Wang and Scrivener, 1995). Hydrotalcite is a natural mineral whose structure consists of layers of brucite ($Mg(OH)_2$) with interstitial water molecules and CO_3^{2-} ions. These (often sub-microscopic) crystals are scattered throughout the C-A-S-H gel. Phases of this type have also been found in cement + slag blends.

- C_4AH_{13} -type phases have been detected in slag activated with NaOH. These phases form platelets 0.1–0.2 mm thick and approximately 1.5 mm in diameter^[15]. Other authors^[13] have observed carbonated phases such as $C_4A\bar{C}H_{11}$ and $C_8\bar{A}C_2H_{24}$ in slag pastes activated with NaOH and $Ca(OH)_2$.

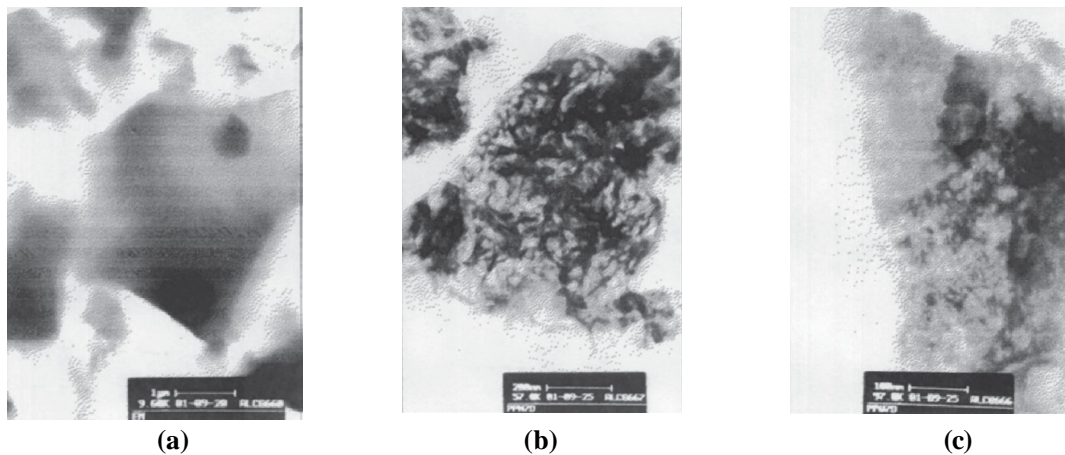


Figure 1.2.3.1

TEM micrographs of (a) anhydrous slag (Ca/Si=1.21, Ca/Al= 2.63, Si/Al= 2.13);(b) slag hydrated with NaOH for 7 days (Ca/Si=0.93, Ca/Al=1.35, Si/Al=1.45): (c) slag hydrated with waterglass for 7 days (Ca/Si= 0.64, Ca/Al = 1.49, Si/Al= 2.32)^[16]

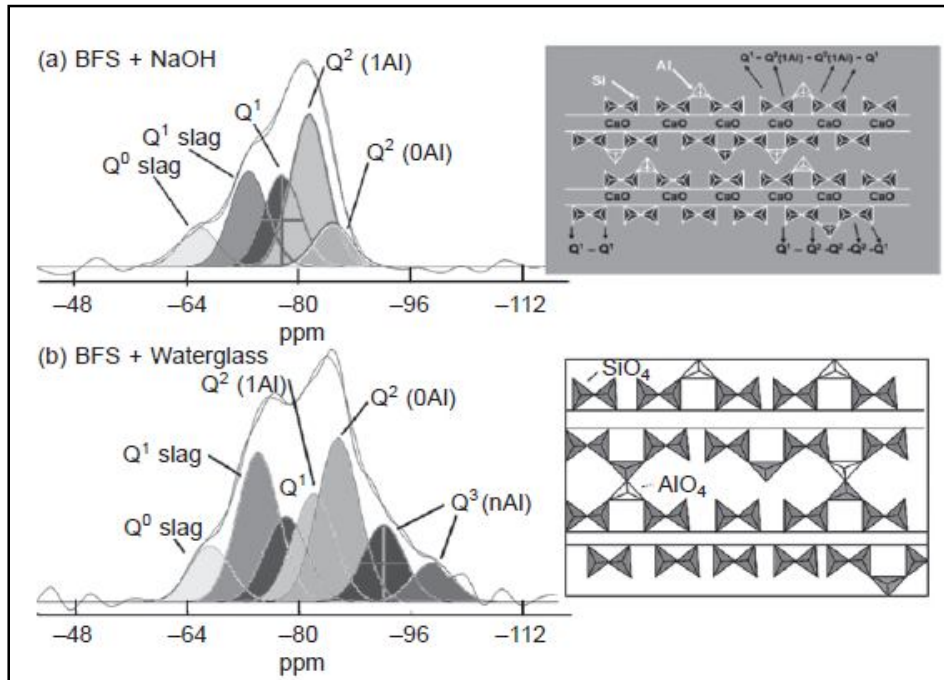


Fig 1.2.3.2

Structural model for an Al-containing C S H gel (Fernández-Jiménez, 2000)^[17]; (a) linear chains; (b) linear chains with occasional cross-linking, forming planes.

1.3. Role of constitutive material in Alkali activated composites (AAC)

1.3.1. Source material and Alkaline liquid

Source material

Alkali Activated Composites based on alumino-silicate should be rich in silicon (Si) and aluminum (Al). Several minerals and industrial by-product materials have been investigated in the past. Metakaolin or calcined kaolin, low-calcium ASTM Class-F fly ash, natural Al-Si minerals, combination of calcined mineral and non-calcined materials, combination of fly ash and metakaolin, and combination of granulated blast furnace slag and have been studied as source materials.

Alkaline activator

The most common alkaline liquid used in activation is a combination of sodium hydroxide (NaOH) or potassium hydroxide (KOH) and sodium silicate or potassium silicate.

Alkali-activation of ground granulated blast furnace slag (GGBS) has become the subject of intense research interest within the past decade. Strength development of slag cement is highly sensitive to curing conditions^[18]. Alkaline activation of blast furnace slag is affected due to the high concentration of OH⁻ ions in the mix^[19, 20, and 21]. It has been reported that all caustic alkalis and alkali compounds whose anions or anion groups can react with Ca²⁺ to produce Ca

compounds that are less soluble than $\text{Ca}(\text{OH})_2$ can act as activators of slag ^[14]. Bakharev et al. ^[15] reported that heat treatment had a significant accelerating effect on strength development of slag pastes. They further concluded that sodium silicates provided the best activation, with compressive strength of pastes and mortars exceeding that of OPC pastes of the same w/b ratio. The alkali and silicate content also plays a significant role in the development of compressive strength ^[22-23]. The curing conditions had a significant effect on the mechanical behavior of the hardened state of alkali-activated slag paste ^[18].

1.4. Application of Alkali Activated Composites

- One of the potential fields of application of geopolymeric materials is in toxic waste management because geopolymers behave similar to zeolitic materials that have been known for their ability to absorb the toxic chemical wastes.
- Balaguru et. al. (1997) reported the results of the investigation on using geopolymers, instead of organic polymers, for fastening carbon fabrics to surfaces of reinforced concrete beams. It was found that geopolymer provided excellent adhesion to both concrete surface and in the interlaminar of fabrics. In addition, the researchers observed that geopolymer was fire resistant, did not degrade under UV light, and was chemically compatible with concrete.
- In Australia, the geopolymer technology has been used to develop sewer pipeline products, railway sleepers, building products including fire and chemically resistant wall panels, masonry units, protective coatings and repairs materials, shotcrete and high performance fibre reinforced laminates (Gourley, 2003; Gourley & Johnson, 2005).
- Railway sleepers can be constructed using AAC due to high strength, higher resistant to shrinkage crack, chemical attack and carbonation.
- Protective layer over OPC concrete due higher fire resistant property
- Increase durability of porous concrete by using AAC binders.

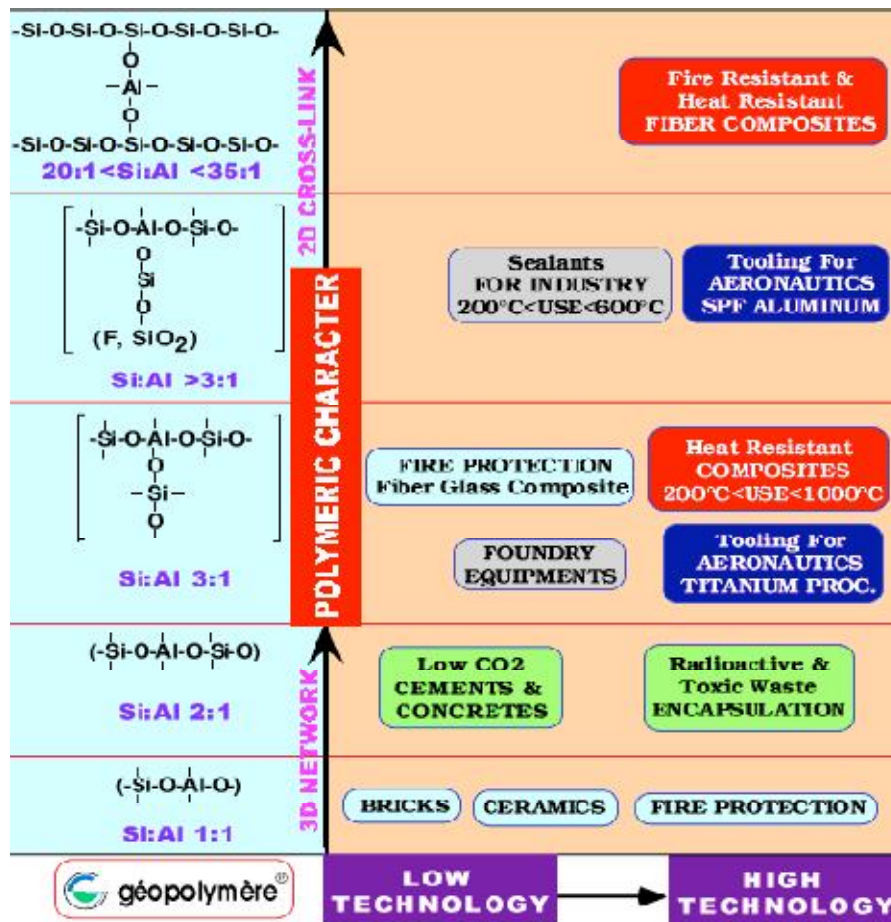


Fig.1.4.1

Application of Alkali Activated Composites

1.5. Objective of present research

The main objective of the present experimental investigation is to study the engineering properties of Alkali activated slag mortar of fresh and hardened state. Performance of admixed AAC, when exposed to high temperature has also been studied. Alkali activated mortar have been prepared by using Ground Granulated Blast furnace Slag (GGBS) as base material which was mixed with sand (Zone-II, Cement:Sand=1:1). Sodium Hydroxide (NaOH) pellets with Sodium Silicate (Na₂SiO₃-Water Glass) solution was mixed and activator solution was prepared. Workability of Alkali activated composite mix depends on amount of Na₂O content, & SiO₂ content and also on Na₂O/SiO₂ ratio, extra water content and also on dosage and type of admixtures/plasticizers added. The quantity of Na₂O content was considered as 4%, 6% and 8% by weight of GGBS. SiO₂ content of 6% by weight of GGBS was kept constant. The engineering properties have been studied in absence and presence of admixtures and an improved AAS mortar in terms of workability and strength has been achieved. A study on the

effect of high temperature exposure on strength and durability of Alkali activated GGBS mortar was also conducted systematically. The mortar test specimens have been exposed to high temperature (400°C, 600°C and 800°C) for 2-hrs and 4hrs, in a Muffle furnace. The engineering properties have been compared with the properties of specimens unexposed to high temperature. GGBS was partially replaced by Fly Ash in different proportion to get an improved performance of the composite on exposure to elevated temperature. A comparative study with conventional blast furnace slag cement based mortar, has also been made. Scanning Electron microscopy (SEM), have also been conducted to examine the microstructure of Alkali activated slag mortar and conventional cement mortar out of Portland Slag Cement. The test results are presented in tabular and graphical form and proper scientific interpretation are made. Based on test results some broad conclusions are also made and areas of future research have been indicated.

2. Review of Existing Literature

2.1.Preamble

Literatures on different aspects of Alkali Activated GGBS are available but improvement of its properties on application of admixtures is limited. The effect high temperature exposure on Alkali activated fly ash composites are available to some extent but little on Alkali activated slag composites. However, some existing literatures are presented here which will help to understand the subject as well as the importance of present research.

2.2. Literature related to Alkali Activated Composites.

2.2.1. Environmental factors.

2.2.1.1. Title: Mehta, P. K. (2001). “Reducing the Environmental Impact of Concrete.” ACI Concrete International 23(10): 61-66.

Summary:

The document states that ‘concrete technologists are faced with the challenge of leading future development in a way that protects environmental quality while projecting concrete as a construction material of choice. Public concern will be responsibly addressed regarding climate change resulting from the increased concentration of global warming gases. In this document, strategies to retain concrete as a construction material of choice for infrastructure development, and at the same time to make it an environmentally friendly material for the future have been described.

2.2.1.2. Title: Mehta, P. K. (2002). “Greening of the Concrete Industry for Sustainable Development.” ACI Concrete International 24(7): 23-28.

Summary:

In order to produce environmentally friendly concrete, Mehta suggested the use of fewer natural resources, less energy, and minimize carbon dioxide emissions. He categorized these short-term efforts as ‘industrial ecology’. The long-term goal of reducing the impact of

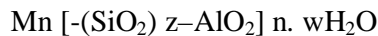
unwanted by-products of industry can be attained by lowering the rate of material consumption. Major improvements in the resource efficiency of the concrete industry are possible if, in the long run, we reduce the rate of concrete consumption by lengthening the service life of newly built structures.

2.2.2. Chemistry of Alkali Activated Composites:

2.2.2.1. Title: Davidovits, J. (1999). Chemistry of Geopolymeric Systems, Terminology. Geopolymer '99 International Conference, France.

Summary:

He suggested the potential polymerization process, which involves a substantially fast chemical reaction under alkaline condition on Si-Al minerals, that results in a three dimensional polymeric chain and bring structure consisting of Si-O-Al-O bonds, as follows :



Where: M = the alkaline element or cat-ion such as potassium, sodium or calcium; the symbol indicates the presence of a bond, n is the degree of polycondensation or polymerization; z is 1, 2, 3, or higher, up to 32.

2.2.2.2. Title: A. Palomo, P. Krivenko, I. Garcia-Lodeiro, E. Kavalerova, O. Maltseva, A. Fernández-Jiménez(2014): “A review on alkaline activation: new analytical perspectives,” Vol. 64, No 315

Summary:

The present paper analyses the most significant theoretical interpretations of the role played by alkalis in the formation of the “stony” structure of cement. It ends with a broad overview of the versatility of this type of materials for industrial applications and a discussion of the possibility of building on the existing legislation to meet the need for the future regulation of alkaline cement and concrete manufacture. They described the reaction mechanism and fundamentals of alkaline activation in calcium-rich systems: [(Na,K)₂O-CaO-Al₂O₃-SiO₂-H₂O], low-calcium systems: [(Na,K)₂O-Al₂O₃-SiO₂-H₂O] and in hybrid systems:

They used transmission electron microscopy (TEM) to confirm that the main reaction product is a leaf-like semi-crystalline calcium silicate hydrate. Micrographs of the initial slag and its alkaline activation products after hydration with NaOH and water glass are reproduced

The micro and nano structural conditions of the pastes generated in the aforementioned systems showed that a complex mix of amorphous gels (C-A-S-H+ (N, C) -A-S-H) precipitates in activated systems

The research conducted on hybrid cements appears to clearly show that the type and characteristics of the gel/s formed are significantly impacted by the working pH and the calcium concentration in the system; a high calcium content and pH level favour C-A-S-H over N-A-S-H gel formation.

2.2.2.3. Title: D.V.S.P. Rajesh, A. Narender Reddy, U. Venkata Tilak, M.Raghavendra(2013) “Performance of alkali activated slag with various alkali activators”:International Journal of Innovative Research in Science, Engineering and Technology Vol. 2, Issue 2, February 2013

Summary:

The overall aim of the study was to investigate the potential of alkali activated slag as a sole binder in producing concrete. The performance of alkali-activated slag concrete with sodium silicate, sodium hydroxide, sodium carbonate as activator are used at 4% Na₂O(by weight of slag) and 4% of hydrated lime by total weight of solid binder content if used as a retarder. The scope of the work covered four mixes: - Normal OPC mix and three alkali activated slag mixes of the same binder content and the same water binder ratio. The fresh concrete properties studied were setting time and workability and the Engineering properties studied are compressive strength was measured in 1,7,28 days, split tensile strength was measured in 7,28 days and flexure, punching shear strength was compared in 12 days only.

Sodium silicate activated slag concrete sets rapidly with the higher Na₂O dosage resulting in shorter setting time. The setting time can be prolonged by adding hydrated lime to mix, which increases the final setting time more when compared to initial setting time. Out of all these three activators NaOH is best ; Na₂CO₃ was the second; Na₂SiO₃ was the third in terms of flexural and punching shear test for the Vizag steel plant slag composition. In compressive test

the strength growth rate in early age is high when compared to traditional concrete. From Load vs. displacement curve in flexure test results, the CM mix gives the higher load carrying capacity but less ductility where as SC4 mix shows lower load carrying capacity but very high ductility. Almost all the AAS concrete shows good ductility than traditional concrete. So AAS concrete is good to use where ductile designs are needed i.e., in seismic prone areas. In punching shear test, among all the AAS.

2.2.3. Properties of Alkali Activated GGBS

2.2.3.1. Title: Jin-Kyu Song, Keun-Hyeok Yang, Geon-Woo Kim, Chan-Taek Lee, Byeong-Jo Kim, Jae-Hyeok Lee(2010) : “Properties of Sodium Alkali-Activated Ground Granulated Blast-Furnace slag(GGBS) Mortar”. Paper’s No. BUE-FISC – 107.

Summary:

They studied and compiled the data about effect for hydration of ground granulated blast furnace slag with activator concentration and to propose optimum activator combination. Strength test was conducted to develop economical efficiency, to apply in situ. In this study it was verified that 13 AAG mortar had been developed the compressive strength. 7 AAG mortar were set. Among them, sodium hydroxide and sodium silicate were shown high strength. So they were chosen to evaluate the compressive strength and the flow value of each batch. And sodium hydroxide or sodium silicate were mixed with another activators for improving performance.

2.2.3.2. Title: Suresh Thokchom, Partha Ghosh and Somnath Ghosh (2009) “Effect of water absorption, porosity and sorptivity on durability of geopolymer mortars” VOL. 4, NO. 7, SEPTEMBER 2009 ISSN 1819-6608 ARPN Journal of Engineering and Applied Sciences.

Summary:

Low calcium Class F fly ash was activated by a mixture of NaOH and Na₂SiO₃ containing 5% to 8% Na₂O with water to fly ash ratio of 0.33. The durability of geopolymer mortar specimens was evaluated on the basis of reduction in compressive strength when exposed in 10% Sulphuric acid solution for 24 weeks. Specimens containing lesser alkali were found to possess higher apparent porosity, water absorption and water sorptivity. After 24 weeks in sulphuric acid solution, specimens still had substantial residual compressive strength

ranging from 29.4% to 54.8%. Specimens with higher water absorption, porosity and water sorptivity lost more strength than those with lesser corresponding values. Results obtained in the experimental program indicate that porosity, sorptivity and water absorption of geopolymer mortar specimens influences the durability of geopolymer mortars in sulphuric acid.

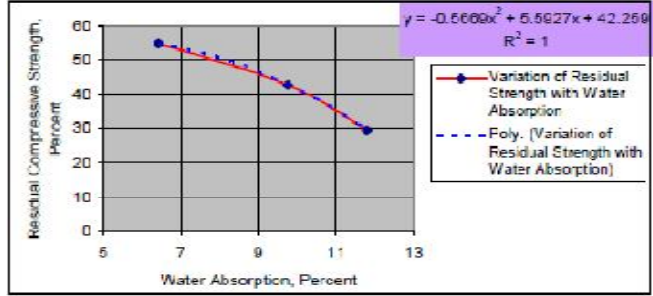


Fig.2.8.1.

Relationship between residual compressive strength and water absorption.

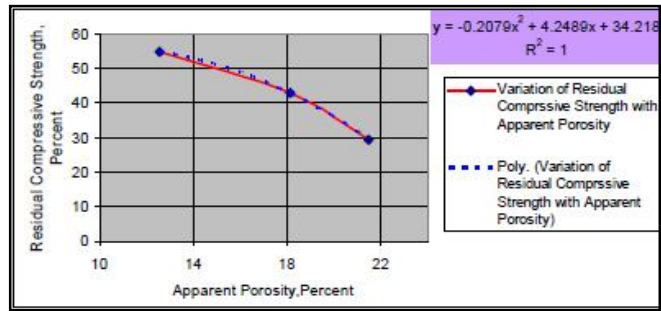


Fig.2.8.2.

Relationship between residual compressive strength and porosity.

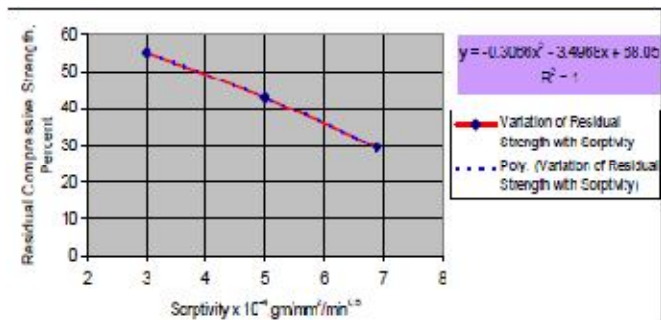


Fig2.8.3.

Relationship between residual compressive strength and sorptivity.

2.2.4. Effect of Admixtures on AAC

2.2.4.1. Title: T. Bakhareva, J.G. Sanjayana, Y.-B. Chengb(2000): “Effect of admixtures on properties of alkali-activated slag concrete”- Cement and Concrete Research Vol.30 Pg. 1367-1374

Summary:

They investigated the effect of Superplasticiser based on modified naphthalene formaldehyde polymers (S), air-entraining agent (AEA), water-reducing (WRR), shrinkage-reducing (SHR) admixtures at dosages of 6 ± 10 ml/kg, and gypsum (G) (6% of slag weight) were used. They studied the study of workability in the fresh state, shrinkage and compressive strength of AAS concrete, and the effect of admixtures and type of activator on these properties.

- An increase in workability was achieved with the lignosulphonate admixture for all types of activators tested.
- With the naphthalene formaldehyde superplasticiser, the increase in workability was only at the initial stage and then a quick set occurred, which was considered to have been accelerated by the admixture.
- Lignosulphonates may cause retarded strength development and reduced flexural strength of AAS concrete.
- Lignosulphonate-based admixture caused a slight reduction in shrinkage, while naphthalene-based superplasticiser significantly increased the shrinkage and reduced the strength of AAS concrete.
- Air-entraining and SHR admixtures, as well as G (6%) were effective in reducing shrinkage in AAS concrete; AEA also greatly improved workability Thus, it was considered that AEA is the most suitable for use in AAS concrete.

2.2.5. Effect of High Temperature on AAC

2.2.5.1. Title: H. M. Khater (2014): “Studying the effect of thermal and acid exposure on alkali activated slag geopolymer”. International Congress on Materials & Structural Stability, Volume 11, MATEC Web of Conferences.

Summary:

He studied on thermal stability as well as acid resistance of geopolymer materials prepared from ground granulated blast furnace slag (GGBFS), air-cooled slag (ACS), silica fume (SF) and cement kiln dust (CKD) using 6% of equal mix from alkaline sodium hydroxide and sodium silicate activators. The addition of ACS, SF and CKD as partially replaced GGBFS is investigated so as to improve the mechanical and microstructural properties of the geopolymer mix. Compressive strength tests and scanning electron microscopy were performed in these studies

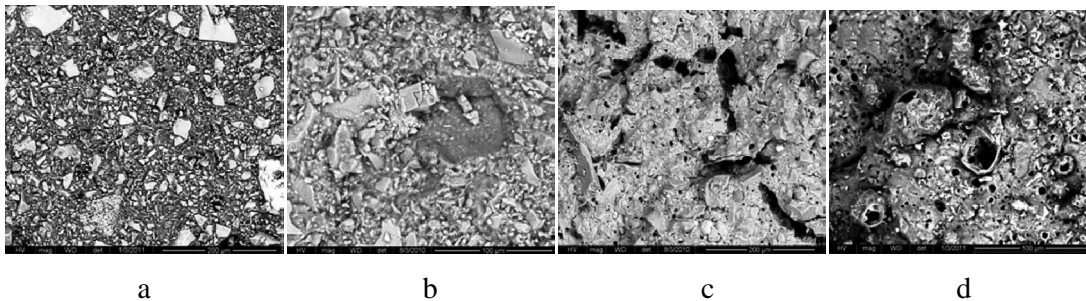


Fig 2.10.1

SEM micrograph of alkali-activated specimens using 3:3sodium hydroxide to sodium silicate having 10%SF and90%WCS: (a) unfired sample; (b) fired at 500°C; (c) fired at 800°C; (d) fired at 1000°C

- The geopolymer materials prepared with sodium-containing activators exhibit thermal stability up to 500°C more than that obtained by normal concrete mix, especially those containing SF and ACS, which could be used in the manufacture of nuclear power plant.
- Partial substitution of GGBFS by 15% ACS leads to a thermal stability up to 1000°C, reflecting the amorphous geopolymer behavior which resists heat treatment and can be used in refractory bricks. Increasing ACS content to 25% lowers the thermal stability of the geopolymer composition.
- Partial substitution of GGBFS by 10% SF with its amorphous structure exhibits thermal stability up to 500°C, giving strength values that exceed the comparable ACS mixes.

However, the strength diminishes beyond this temperature, while ACS mixes resist deterioration up to 800°C.

- Most geopolymer mixes as well as concrete specimens have low acid resistance and deteriorated after 3 months of immersion in acids. These were revealed by their loss in strength and were emphasized from the images of the visual examination, where the decreased pH leads to destabilization and dissolution of the geopolymer structure.
- The strength of 25% ACS mixes decreases and completely diminishes beyond 3 months, while SF mixes resist acid deterioration up to 2 months, providing strength values exceeding those of ACS mixes. On the other hand, mixes that have 25% CKD provide the lowest stability upon acid immersion, as reflected from their strength values, which were lower than those of concrete mixes.

2.2.6. Properties of GGBS/Fly Ash AAC Blends

2.2.6.1. Title: Arie Wardhonoa, David W. Lawb, Anthony Stranob (2015) : “ The strength of alkali-activated slag/fly ash mortar blends at ambient temperature” . The 5th International Conference of Euro Asia Civil Engineering Forum (EACEF-5) Procedia Engineering, Vol. 125, Pg 650 – 656.

Summary:

This paper reports the detail of the experimental work that has been undertaken to investigate the strength of AAS/fly ash (AASF) mortar blends. The AASF specimens were prepared using a mix of ground granulated blast-furnace slag (GGBS) and low calcium class F fly ash activated by high alkaline solution. The mix compositions of slag to fly ash were 1:0, 0.9:0.1, 0.8:0.2, 0.7:0.3, 0.6:0.4 and 0.5:0.5, respectively. The standard curing regime at ambient temperature was applied.

The results showed that the mix proportion of 0.5 slags: 0.5 fly ash produced the best strength results. The standard deviation values also reduced along with the increase of fly ash content indicating an improved stability of the specimens. It also suggested that 0.5 slag: 0.5 fly ash blend could provide a solution for the need of heat curing for fly ash-based geopolymer.

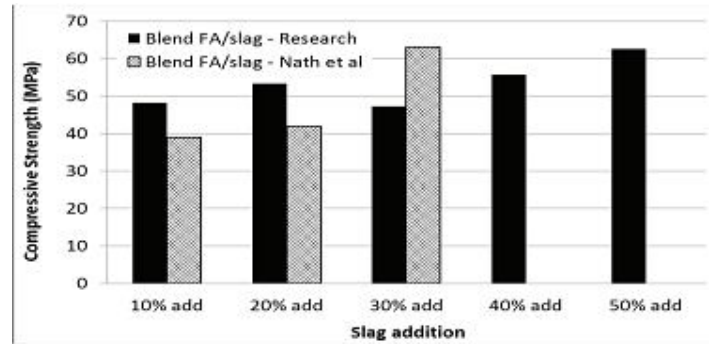


Fig.2.11.1.

Strength development of AASF mortars at 28 days age.

- AASF specimen test results suggest that the hydration reaction of slag and the polymerization reaction of fly ash could occur separately or simultaneously. The results suggest that the simultaneous reaction is the most likely with the GGBS reaction activating the fly ash, enabling it to react at room temperature.
- The addition of fly ash to the mixes resulting in lower standard deviations which means improved stability of AASF mortar specimens.

2.2.6.2. Title: N. Marjanović, M. Komljenović, Z. Baščarević, V. Nikolić, R. Petrović (2014): “Physical–mechanical and microstructural properties of alkali-activated flyash–blast furnace slag blends”. *Ceramics International* 41 (2015) 1421–1435

Summary:

This paper investigated physical–mechanical and microstructural properties of alkali-activated binders based on blends of fly ash (FA) and blast furnace slag (BFS). FA–BFS blends were alkali-activated with sodium silicate (water glass) solution. The synthesis of alkali-activated binders was conducted at 95 °C during 24 h, with different FA–BFS mass ratios (100:0; 75:25; 50:50; 25:75; 0:100), different moduli of alkali activator ($\text{SiO}_2/\text{Na}_2\text{O}$: 0.5; 1.0; 1.5) and different alkali activator concentration (% Na_2O : 4; 7; 10). The influence of alkali activation conditions on the flexural and compressive strengths, setting time, drying shrinkage and the microstructure of synthesized binders was investigated.

It was found that the compressive strength mostly depended on the composition of the FA–BFS blends and the water/binder ratio. The setting time highly depended on the activator concentration, while the drying shrinkage was mostly affected by the curing temperature. The

blend comprising FA 25%–BFS 75%, activated with the activator of modulus 1.0 and the activator concentration of 10% Na₂O yielded optimal mortar regarding investigated physical–mechanical characteristics. Microstructural examination showed that the chemical composition of the binding phase varied as a function of the blend composition. Predominance of FA alkali reaction products in the binding phase positively influenced the flexural strength, while the predominance of BFS alkali reaction products positively affected the compressive strength.

2.3. Summary on Literature review.

The following aspects were appreciated

- Researches done with an aim to increase the use of Alkali activated composites in construction industry will help to reduce CO₂ emission.
- The reaction mechanism and fundamentals of alkaline activation in calcium-rich systems, low-calcium systems and in blended systems are proposed.
- Water plays an important role during dissolution, poly-condensation & hardening stages of geopolymerisation. Reduction of water content improved the compressive strength.
- AAGGBS concrete shows good tensile property than traditional concrete.
- Results obtained in the experimental program indicate that porosity, sorptivity and water absorption of geopolymer mortar specimens influences the durability.
- An increase in workability was achieved with the lignosulphonate admixture for all types of activators tested.
- Air-entraining admixtures were effective in reducing shrinkage in AAGGBS concrete.
- Partial substitution of GGBFS by 10% Fly Ash structure exhibits thermal stability up to 500°C, giving strength values that exceed the comparable AAGGBS mixes.
- The mixed proportion of higher fly ash than GGBS produces better strength result than mixed proportion of lower fly ash.
- However, the effect of admixture has not yet been well defined for Alkali activated slag mortar. There have not been still enough literatures with respect to high performance of Alkali activated slag mortar.
- No definite paper to compare the properties of alkali activated slag and conventional slag has not yet been done.

3. Experimental Investigation

3.1 Preamble

An experimental programme has been undertaken to study the engineering properties of Blast furnace slag based Alkali Activated composites. Studies have been conducted on specimens exposed to high temperature also

The scope of the experimental investigation includes:

- Preparation of Alkali Activated Ground Granulated Blast Furnace Slag (AAGGBS) mortars in presence and absence of admixtures including its flow behaviour
- To study the mechanical properties of AAGGBS mortar test specimens.
- To study the mechanical properties of AAGGBS mortar test specimen exposed to high temperatures (different temperature level and duration of heating)
- Comparison of results with Alkali activated slag +fly ash mortar test specimens and conventional slag cement mortar test specimen exposed to high temperature

All the casting and testing have been conducted in the Concrete Laboratory of Civil Engineering Department, Jadavpur University and SEM has been conducted in the Materials Testing Lab of Metallurgy Engineering Department of Jadavpur University.

3.2. Manufacturing process of Alkali Activated Composites

Alkali Activated Composites were prepared by mixing GGBFS with alkali activator solution to produce 'alkali activated paste'. The mixture is usually homogenous slurry which is gray in colour. The silica sand was added in the paste to produce mortar. Both Alkali activated paste and mortar mix were found cohesive in nature in fresh state. Mixes are prepared for different alkali content (4, 6 & 8 %) under different water/GGBFS ratio (0.38, 0.35 & 0.33 by weight) i.e. 9 sets. Mixes are also prepared using admixtures i.e. a total of 18 sets. 3 sets of mixes have also been prepared using GGBS with fly ash.



Fig 3.2.1

Ground Granulated Blast furnace Slag



Fig 3.2.2

Sodium Silicate solution, Sodium Hydroxide Platelets and their solution (L to R)

Alkali Activated GGBS mortar mix (9 sets):

- GGBFS : 1000 gm
- Alkali content (Percentage Na_2O) : Varied as 4,6 & 8 % by weight of GGBFS
- Silica content (Percentage SiO_2) : 6 % by weight of GGBFS
- Sand to GGBFS ratio : 1:1
- Water to GGBFS ratio : 0.38, 0.35 and 0.33 by weight.

Alkali Activated GGBS mortar mix with Admixture (9 sets):

- GGBFS : 1000 gm
- Alkali content (Percentage of Na_2O) : 4,6 & 8 Percentage by weight of GGBS

- Silica content (Percentage of SiO₂) : 6 Percentage by weight of GGBFS
- Sand to GGBFS ratio : 1:1.
- Water to GGBFS ratio : 0.38, 0.35 and 0.33 by weight
- Admixtures : VMA (Sika-Viscocrete 2004-NS=1.2%) & Retarder (Fosroc –R=1.5%) by weight of GGBFS

Alkali Activated GGBS & Fly Ash mortar mix with Admixture (3 sets):

- GGBFS & Fly Ash : Varied as 800 & 200 gm; 700 & 300 gm; 600&400gm
- Alkali content (PercentageNa₂O) : 8 Percentage by weight of binder
- Silica content (PercentageSiO₂) : 6 Percentage by weight of binder
- Sand to GGBFS ratio : 1 by weight.
- Water to GGBFS ratio : 0.33 by weight
- Admixtures : VMA (Sika-Viscocrete 2004-NS=1.2%) & Retarder (Fosroc –R=1.5%) by weight of binder

To make a comparative study a set with conventional cement mortar was prepared:

Conventional Slag Cement mortar mix (1 set):

- Portland Slag Cement : 1000 gm
- Sand to Slag Cement ratio : 1 by weight
- Water to Cement ratio : 0.33 by weight
- Admixtures : VMA (Sika-Viscocrete 2004-NS=0.5%) by weight cement

Following manufacturing process was adopted for preparing alkali activated mortar specimens.

- Mix sodium silicate solution, sodium hydroxide pellets and water according to pre decided mix proportion. Alkaline activator was made, at least one day prior to its use.
- Hobart mixer with rotating blades was used for preparing alkali activated mortar (Refer to Fig.3.2.3).Mix GGBFS and Alkaline activator in the Hobart mixer for about four to five minutes to make homogeneous paste.

- For preparing mortar specimens, sand in saturated surface dry condition is slowly added to alkali activated paste in the Hobart mixer and continue mixing for another 4-5 minutes after adding the sand.
- Alkali activated mortar was then poured in 50mm x 50mm x 50mm cubes.
- Vibrate the fresh alkali activated mortar mix in the moulds on a vibration table for 2-3 minutes to remove entrapped air in the mix.
- After a period of 24 hrs, the specimens were removed from mould and placed in water for curing for 28 days.
- After curing for 28 days the specimens were left to air until tested.
- 50 mm mortar cubes of conventional slag cement were also prepared using standard procedure (refer to Fig. 3.2.4.).



Fig 3.2.3
Hobart mixer



Fig 3.2.4
Preparation of PSC Mortar

3.3. Testing and characterization of alkali activated composites:

The main purpose of testing of alkali activated composites is to investigate the engineering properties of GGBFS based alkali activated composites and to characterize microstructure of alkali activated specimens, synthesized at different test conditions. The laboratory tests will be conducted as per relevant Indian standard codes and in some special tests, ASTM standard may be followed. After curing the samples they were air dried for 24 hrs and then placed inside the maple furnace. The microstructure characterization of alkali activated mortar was carried out using XRD, SEM and EDX. The details of various test procedures used in the present research are described in the following sections.

3.3.1. Workability and Setting time measurements

The workability of alkali activated mix may be determined using mini slump cone as shown in the Fig. 3.3.1 (a) & (b). The dimensions of the mini slump cone^[33] mould are: top diameter 70mm, bottom diameter 100mm and height 60mm. The mould was fixed firmly on a flow table and filled with fresh mortar. The mortar was tamped down with spatula to ensure proper compaction. When the mould was full the top surface was leveled and it was immediately lifted vertically allowing mortar to flow on flow table. The flow diameter is measured in two perpendicular directions and average of the two was considered as flow diameter as a measure of workability. Depending on flow diameter, workability of alkali activated mix may be classified as stiff, moderate and high.



Fig 3.3.1(a)
Mini Slump Cone with flow table



Fig 3.3.1(b)
Flow measurement

A Vicat needle, as shown in Fig., is used to measure the setting time of alkali activated binders. The modified Vicat needle essentially consisted of a needle of 1 mm diameter cross-section and 100 g, conforming to ASTM C191-04. A cylindrical container of 75 mm diameter was filled with 50 mm of the freshly prepared alkali activated pastes. Values of penetration distances were obtained every ten minutes. The initial setting time is obtained when the penetration of needle is 25mm.

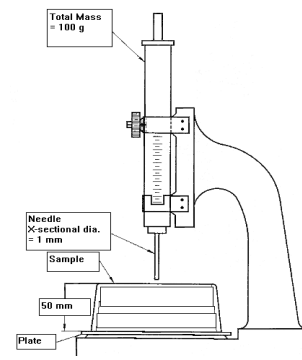


Fig. 3.3.1(c).
Vicat Apparatus

On the other hand, the final setting time is the time required to reach a penetration distance of zero mm. At the final setting, alkali activated binders are almost in hardened state. Molding without causing material damage is not possible thereafter.

3.3.2 Bulk density and apparent porosity

The bulk density and apparent porosity for alkali activated specimen is determined according to Archimedes principle with water as immersion medium. The procedure to be adopted is as follows: dry all specimens in a ventilated oven at 65°C for 48 hours. Record the weight of dried specimens as W_d . Immerse the specimens in water at room temperature (28°C) for minimum 48 hours. Weight the specimens while suspended by a thin wire and completely submerged in water and record W_i . Remove specimens from water and allow water to drain off by placing them on a wire mesh, removing visible surface water with a damp cloth; weigh and record saturated weight as W_s . The bulk density and apparent porosity was then calculated as follow:



Fig.3.3.2.
Arrangement to measure Apparent Porosity

$$\begin{aligned} \text{Dry density (Kg/m}^3\text{)} &= [W_d / (W_s - W_i)] \times 1000 \\ \text{Apparent Porosity (\%)} &= [(W_s - W_d) / (W_s - W_i)] \times 100 \end{aligned}$$

3.3.2. Compressive strength

The direct compressive strength of hardened alkali activated specimens will be obtained at the age of 28 days, using 2000kN capacity digital compressive testing machine. At 28 days age, three identical samples were tested in accordance with ASTM C-109 -02^[34] and the mean values of compressive strength are reported in relevant tables and graphs. Also for the specimens exposed to temperature, compressive strength will be measured. A typical testing of mortar specimen is shown in Fig.3.3.3



Fig.3.3.3
Digital compression testing machine

3.3.4. Split tensile strength

Indirect tensile strength of alkali activated mortar was determined in accordance with the procedure detailed in American Society for Testing and Materials, ASTM C-496, using 100mm diameter by 200mm long cylinders. The split tensile strength of specimen may be calculated in the following way

$$T = \frac{2 \times P}{\pi \times L \times D}$$

Where

T= splitting tensile strength in KPa.

P= Maximum applied load indicated by the testing machine (KN).

L= Length in meter and D= Diameter in meter.



Fig-3.3.4
Split tensile strength testing

3.3.5. Water Absorption

The volume of pore space in specimen matrix, as distinct from the ease with which a fluid can penetrate it, is measured as absorption^[30]. Water absorption is usually measured by drying a specimen to a constant mass, immersing it in water, and measuring the increase in mass as a percentage of dry mass. In the present research, water absorption of specimens will be determined as per ASTM C-642. The 28 days aged specimens are dried for 48 hours at 65°C & then immersed in water for 24 hours. The test specimens soaked in water are removed from the immersion container, wiped clean and weighed immediately in saturated-surface-dry (SSD) condition to find increase in mass.



Fig. 3.3.5.
Setup for water sorptivity test

3.3.6. Water Sorptivity

Permeability tests measure the response of concrete to pressure, which is rarely the driving force of fluid entering concrete, there is a need of another type of test. Such a test measures the rate of absorption^[29] of water by capillary suction of unsaturated concrete placed in contact with water; no head of water exists. The sorptivity test according to Neville A.M. determines the rate of capillary rise absorption by mortar/ concrete cube. The specimens rest on small supports in a manner such that only the lowest 2 to 5mm of the cube is submerged. The increase in the mass of the prism with time is recorded. The specimens should be dried prior to the experiment. It has been shown that there exists a relation of form.

$$I = S\sqrt{t}$$

Where,

I=increase in mass per unit area (gm/mm^2) since beginning of the test per unit of cross-sectional area in contact with water; as increase in mass is due to ingress of water. It is expressed in mm.

t=time, measured in minutes, at which the mass is determined.

S=Sorptivity in $\text{mm}/\text{min}^{0.5}$

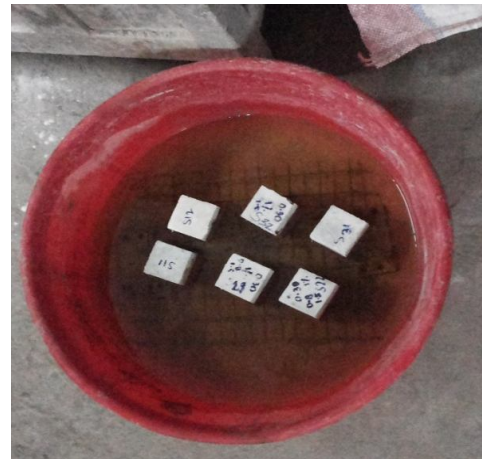


Fig-3.3.6:
Setup for water sorptivity test

3.3.7. Ultrasonic Pulse Velocity (UPV)



Fig 3.3.7

Ultrasonic Pulse Velocity (UPV) Test

Ultrasonic Pulse Velocity^[32] method consists of measuring the time of travel of an ultrasonic pulse, passing through the mortar/ concrete. The mortar cube specimens having dimension $50\text{mm} \times 50\text{mm} \times 50\text{mm}$ and cylinder specimen of dia. 75mm and 150 mm long was used in this study. Typical test setup for ultrasonic testing is shown in Fig. Direct transmission of pulse is employed for the study. Although the pulse velocity is affected by a number of factors, the most important parameter is the porosity of mix. The ultrasonic pulse velocity measurement was conducted as per IS 13311 (Part-1): 1992 standard using a commercially available PUNDIT system. The testing system consisted of a pulsar/receiver unit with a built-in data acquisition system and a pair of narrow band, 150-kHz transducers. UPV measurements could be performed to monitor the internal structure of mortar nondestructively. The test procedure is as follows: Ultrasonic pulse is produced by the transducer which is held in contact with the surface of the specimen. The transit time (T) of the pulse is measured for path length (L). The Pulse Velocity (V) is given by: $V = L / T$

3.3.8 Heating of samples in muffle furnace.

After curing the samples they are air dried for 24 hrs and the placed inside the muffle furnace. The samples are exposed for 2hr-each for 400°C, 600°C and 800°C.



Fig 3.4.8
Muffle furnace

3.3.9 Mercury Intrusion Porosimetry (MIP) Analysis

MIP samples are made by cutting a cylinder of $\frac{1}{4}$ in. dia. To $\frac{1}{2}$ in. height, having a bulk sample volume of 1.00cc which is tested on a Micromeritics Auto pore II MIP from 0-60,000 pis, with a Hg surface tension 480.00erg/cm²,contact angle (I)140.00⁰. MIP is used to examine a statistical comparison of the tested samples in terms of mean and median pore size, pore distribution, total porosity, bulk density and apparent density is possible through mercury intrusion porosimetry test. Mercury Intrusion Porosimetry (MIP) provide PSD distribution curve for intrude-able pores in the form of pore volume versus apparent pore entry radius. In this method mercury is intruded under pressure in an evacuated sample and volume of intruded mercury is monitored against pressure. Mercury being a non-wetting liquid for most of the solids (especially for cement based materials and ceramics), the intrusion process follows Washburn's equation that relates pressure to equivalent pore entry radius. Thus radius versus intruded volume



Fig.3.4.9
MIP testing machine

3.3.10. X-Ray Diffraction (XRD) Analysis

The X-ray diffraction (XRD) technique offers a convenient way to determine the mineralogical analysis of crystalline solids. If a crystalline mineral is exposed to X-rays of a particular wavelength, the layers of atoms diffract the rays and produce a pattern of peaks, which is characteristic of the mineral. The horizontal scale (diffraction angle) of a typical XRD pattern gives the crystal lattice spacing, and the vertical scale (peak height) gives the intensity of the diffracted ray. When the powder specimen being X-rayed contains more than one mineral the intensity of characteristic peaks from the individual minerals are proportional to their amount. An X-Ray diffraction analysis of powdered specimens will be made using a Rigaku Geigerflex D-max II automated diffractometer (Rigaku, Japan) with Cu-K α radiation with the following conditions: 40 kV, 22.5 mA. The XRD patterns were obtained by scanning at 1° (2 θ) per min and in steps of 0.5° (2 θ). The slow scanning rate will be used to improve resolution of peaks. Fig. 3.10.9 shows a photograph of X-ray diffractometer used in the present research.

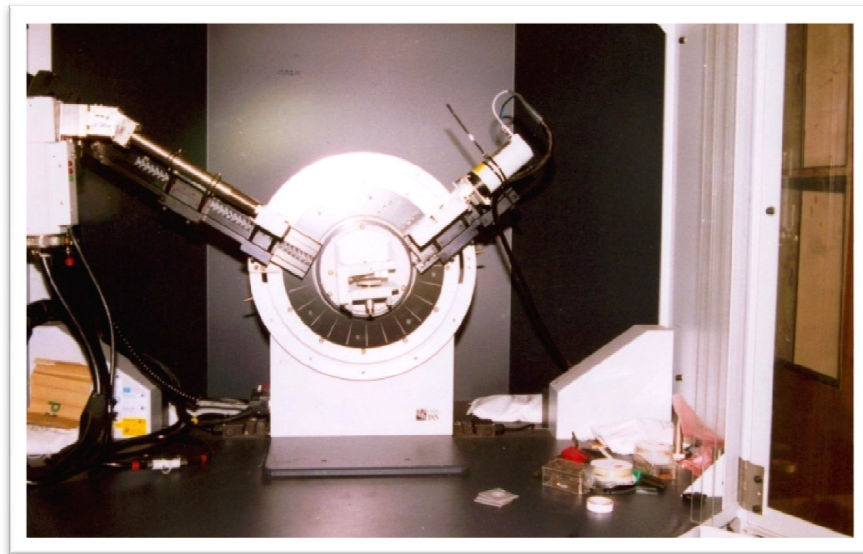


Fig.3.10.9:
A photographs of X-ray Diffractometer

3.3.11. Scanning Electron Microscopy (SEM)

Scanning Electron Micrograph study is used to make qualitative assessment of microstructure. SEM specimens is dried, then gold coated and stored in the desiccators prior to examination using a Joel JSM 5200 as shown in Fig.3.10.11(a)



Fig. 3.10.11(a)

JEOL JSM 5200 Scanning electron microscope

Theory

Interaction of the scanning electron microscope electron beam with the specimen surface causes a range of effects where electrons and X-rays are emitted of that generated from the specimen under the beam. The beam is composed of primary electrons, which collide with electrons in orbit around the nuclei of elements present in the specimen. Primary electrons have energy values (up to 40 KeV), and can cause displacement of these secondary electrons, that may subsequently leave the specimen surface and be detected. Secondary electrons do not have high-energy values, compared to backscattered electrons; conventionally bellow 50eV, whereas backscattered electrons have energies ranging from 50eV to that of the primary electrons. Backscattered electrons can therefore be a mixture of high energy secondary electrons knocked out of position by higher energy primary electrons, or primary electrons that have re-emerged from the specimen surface after colliding with and being re-directed by, in-situ electrons that have not been displaced.

If an electron has been knocked out of orbit around an atoms nucleus, and is subsequently replaced by another electron from a lower energy electron shell of the atom, an X-ray can be generated that is characteristic of the element from which it came. The interaction of the beam with the specimen is known as the interaction volume, and is the volume below the surface of the material, in which, primary electrons interact with electrons orbiting around atoms of the material, in which primary material. Secondary electrons are detected from a near surface region of this volume, while backscattered electron can be detected from this and much deeper parts of the specimen. Many electrons escaping from this region are absorbed into the material or not detected at all, as is the case with a small number of x-rays. The range of detected electrons and X-rays is similar in size to the interaction volume as the material less easily absorbs X-rays.

Sample preparation

SEM examination was made on a cylindrical sample of 10mm diameter and 10mm height approximately that are prepared from the broken samples of geopolymers specimen and immediately gold coated to avoid further chemical reactions. The specimens are stored in the desiccators. So, it is expected that the carbonation effect is insignificant. Also, as all the specimens are prepared in similar condition such effect must be identical in all specimens. The micrographs are obtained at different magnification for the justification of improvement in microstructure of paste/mortar samples. This test was done in the Department of Metallurgy, Jadavpur University, and Kolkata, India.



Fig. 3.10.11(b)

Sample preparation in auto fine coater

3.3.12. Summary of Experimental programme

Table 3.3.12.

Summary of Experimental programme

Sl. No	Specimen type	Specimen per experiment	Name of the Experiment	Objective of the test
1.	Alkali activated slag mortar	3	Flow Table	To appreciate workability level of Alkali activated mortar
2	Cube (50mmx50mmx50mm)	4	Compressive strength Test	To get 28 days compressive strength
3	Cylinder (75mm dia x150mm long)	3	Split Tensile Strength	To get indirect tensile strength
4	Cube (50mmx50mmx50mm)	4	Sorptivity Test	To appreciate tendency of unidirectional flow through the test specimen
5	Cube (50mmx50mmx50mm)	4	Water absorption	To appreciate the porosity level
6	Cube (50mmx50mmx50mm)	4	Weight loss	To assess the weight loss due to leaching etc.
7	Cube (50mmx50mmx50mm)	4	Apparent porosity	To appreciate the porosity level
8	Cube (50mmx50mmx50mm)	4	Sp. Gravity	To appreciate the Sp.Gravity
9	Cylinder (50mmx50mmx50mm)	3	UPV	To appreciate the density, void, incipient flaws, strength and metallic behaviour
10	Fraction of Cube (10mmx10mmx5mm)	1	SEM	To study the microstructure of the mortar specimens

3.4. Alkali Activated Composite mortars (AAGGBS & AASF) and Conventional Slag Cement (CSC) mortars mixes

Details of mixes of different types of mortars are tabulated below:

Table 3.4.1.

Details of mixes for AAGGBS test specimens:

Sl.No.	Sample Type	Test name	%Na ₂ O	%SiO ₂	GGBFS/Sand	Water /binder
1	Mortar	Workability	4%	6%	1	0.38
			6%			
			8%			
			4%	6%	1	0.35
			6%			
			8%			
			4%	6%	1	0.33
			6%			
			8%			
2	Mortar	Water sorptivity	4%	6%	1	0.38
			6%			
			8%			
			4%	6%	1	0.35
			6%			
			8%			
			4%	6%	1	0.33
			6%			
			8%			

Table 3.4.1. Contd.

Sl.No.	Sample Type	Test name	%Na ₂ O	%SiO ₂	GGBFS/Sand	Water /binder
3	Mortar	Water Absorption	4%	6%	1	0.38
			6%			
			8%			
			4%	6%	1	0.35
			6%			
			8%			
			4%	6%	1	0.33
			6%			
			8%			
4	Mortar	Apparent Porosity	4%	6%	1	0.38
			6%			
			8%			
			4%	6%	1	0.35
			6%			
			8%			
			4%	6%	1	0.33
			6%			
			8%			
5	Mortar	Compressive Strength	4%	6%	1	0.38
			6%			
			8%			
			4%	6%	1	0.35
			6%			
			8%			
			4%	6%	1	0.33
			6%			
			8%			
6	Mortar	Split Tensile Strength	4%	6%	1	0.38
			6%			
			8%			
			4%	6%	1	0.35
			6%			
			8%			
			4%	6%	1	0.33
			6%			
			8%			

Table 3.4.1 Contd.

Sl.No.	Sample Type	Test name	%Na ₂ O	%SiO ₂	GGBFS/Sand	Water /binder
7	Mortar	UPV	4%	6%	1	0.38
			6%			
			8%			
			4%	6%	1	0.35
			6%			
			8%			
			4%	6%	1	0.33
			6%			
			8%			
8	Mortar	SEM	8%	6%	1	0.33

Table 3.4.2.

Details of mixes for AAGGBSA (Alkali activated slag + admixtures) test specimens

Sl.No.	Sample Type	Test name	Admixture	%Na ₂ O	%SiO ₂	GGBFS/Sand	Water /binder
1	Mortar	Workability	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	4%	6%	1	0.38
				6%			
				8%			
				4%	6%	1	0.35
				6%			
				8%			
				4%	6%	1	0.33
				6%			
				8%			
2	Mortar	Water sorptivity	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	4%	6%	1	0.38
				6%			
				8%			
				4%	6%	1	0.35
				6%			
				8%			
				4%	6%	1	0.33
				6%			
				8%			

Table 3.4.2 Contd.

Sl.No.	Sample Type	Test name	Admixture	%Na ₂ O	%SiO ₂	GGBFS/Sand	Water /binder
3	Mortar	Water Absorption	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	4%	6%	1	0.38
				6%			
				8%			
				4%	6%	1	0.35
				6%			
				8%			
				4%	6%	1	0.33
				6%			
8%							
4	Mortar	Apparent Porosity	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	4%	6%	1	0.38
				6%			
				8%			
				4%	6%	1	0.35
				6%			
				8%			
				4%	6%	1	0.33
				6%			
8%							
5	Mortar	Compressive Strength	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	4%	6%	1	0.38
				6%			
				8%			
				4%	6%	1	0.35
				6%			
				8%			
				4%	6%	1	0.33
				6%			
8%							
6	Mortar	Split Tensile Strength	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	4%	6%	1	0.38
				6%			
				8%			
				4%	6%	1	0.35
				6%			
				8%			
				4%	6%	1	0.33
				6%			
8%							

Table 3.4.2 Contd.

Sl.No.	Sample Type	Test name	Admixture	%Na ₂ O	%SiO ₂	GGBFS/Sand	Water /binder				
7	Mortar	UPV	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	4%	6%	1	0.38				
				6%							
				8%							
				8	Mortar	SEM	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	4%	6%	1	0.35
								6%			
								8%	6%	1	0.33
								8%			
				8	Mortar	SEM	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	8%	6%	1	0.33

Table 3.4.3.
Details of mixes for AAGBSA test specimens exposed to Temperature

Sl.No.	Sample Type	Test name	%Na ₂ O	%SiO ₂	GGBFS /Sand	Water /binder	Admixture	Hours of Exposure to High Temp.	Temp Range
1	Mortar	Loss in Weight	8	6	1	0.33	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	2hr	400
									600
									800
								4hr	400
									600
									800
2	Mortar	Apparent Porosity	8	6	1	0.33	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	2hr	400
									600
									800
								4hr	400
									600
									800

Table 3.4.3 Contd.

Sl.No.	Sample Type	Test name	%Na ₂ O	%SiO ₂	GGBFS /Sand	Water /binder	Admixture	Hours of Exposure to High Temp.	Temp Range
3	Mortar	Water absorption	8	6	1	0.33	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	2hr	400
									600
									800
								4hr	400
									600
									800
4	Mortar	Water Sorptivity	8	6	1	0.33	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	2hr	400
									600
									800
								4hr	400
									600
									800
5	Mortar	Ultrasonic Pulse Velocity	8	6	1	0.33	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	2hr	400
									600
									800
								4hr	400
									600
									800
6	Mortar	Compressive strength	8	6	1	0.33	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	2hr	400
									600
									800
								4hr	400
									600
									800
7	Mortar	Split Tensile Strength	8	6	1	0.33	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	2hr	400
									600
									800
								4hr	400
									600
									800
8	Mortar	SEM	8	6	1	0.33	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Conplast-R =1.5%	4hr	800

Table 3.4.4
Details of mixes for AAGGBSFA (Alkali activated slag+Fly ash+ Admixture) test specimens

Sl. No	Sample Type	Test name	Admixture	%Na ₂ O	%SiO ₂	Binder /Sand	Water /binder	GGBS (gm)	Fly Ash (gm)
1	Mortar	Workability	Viscocrete=1.2% & Fosroc R=1.8%	8%	6%	1	0.33	800	200
								700	300
								600	400
2	Mortar	Water sorptivity	Viscocrete=1.2% & Fosroc R=1.8%	8%	6%	1	0.33	800	200
								700	300
								600	400
3	Mortar	Water Absorption	Viscocrete=1.2% & Fosroc R=1.8%	8%	6%	1	0.33	800	200
								700	300
								600	400
4	Mortar	Apparent Porosity	Viscocrete=1.2% & Fosroc R=1.8%	8%	6%	1	0.33	800	200
								700	300
								600	400
5	Mortar	Compressive Strength	Viscocrete=1.2% & Fosroc R=1.8%	8%	6%	1	0.33	800	200
								700	300
								600	400
6	Mortar	Split Tensile Strength	Viscocrete=1.2% & Fosroc R=1.8%	8%	6%	1	0.33	800	200
								700	300
								600	400
7	Mortar	Ultrasonic Pulse Velocity	Viscocrete=1.2% & Fosroc R=1.8%	8%	6%	1	0.33	700	300

Table 3.4.5.
Details of mixes for AASFA (Alkali activated slag+Fly ash+ Admixture) test specimens for temperature study

Sl. No.	Sample Type	Test names	Admixture	% Na ₂ O	% SiO ₂	Binder/ Sand	Water/ binder	GGBS (gm)	Fly Ash (gm)	Hrs of Exposure	Temperature (°C)
1	Mortar	Water Absorption, Apparent Porosity, Water Sorptivity, Compressive Strength, Split Tensile Strength & UPV	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Complast-R =1.5%	8	6	1	0.33	800	200	2	400
											600
											800
										4	400
											600
											800
2	Mortar	Water Absorption, Apparent Porosity, Water Sorptivity, Compressive Strength, Split Tensile Strength & UPV	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Complast-R =1.5%	8	6	1	0.33	700	300	2	400
											600
											800
										4	400
											600
											800
3	Mortar	Water Absorption, Apparent Porosity, Water Sorptivity, Compressive Strength, Split Tensile Strength & UPV	Sika: Viscocrete 2004 NS=1.2% & Fosroc: Complast-R =1.5%	8	6	1	0.33	600	400	2	400
											600
											800
										4	400
											600
											800

Table 3.4.6.
Details of mixes for CSC (Portland slag cement + admixture) test specimens

Sl.No.	Sample Type	Test name	Admixture	GGBFS/Sand	Water /binder
1	Mortar	Workability	Sika: Viscocrete 2004 NS=0.5%	1	0.38
				1	0.35
				1	0.33
2	Mortar	Water sorptivity	Sika: Viscocrete 2004 NS=0.5%	1	0.33
3	Mortar	Water Absorption	Sika: Viscocrete 2004 NS=0.5%	1	0.33
4	Mortar	Apparent Porosity	Sika: Viscocrete 2004 NS=0.5%	1	0.33
5	Mortar	Compressive Strength	Sika: Viscocrete 2004 NS=0.5%	1	0.33
6	Mortar	Split Tensile Strength	Sika: Viscocrete 2004 NS=0.5%	1	0.33
7	Mortar	UPV	Sika: Viscocrete 2004 NS=0.5%	1	0.33
8	Mortar	SEM	Sika: Viscocrete 2004 NS=0.5%	1	0.33

Table 3.4.7.
Details of mixes for CSC (pozolona slag cement + fly ash+ admixture) test specimens for temperature study

Sl..No	Sample Type	Test name	Admixture	GGBFS /Sand	Water /binder	Hrs of Exposure	Temperature (°C)
1	Mortar	Water Absorption, Apparent Porosity, Water Sorptivity, Compressive Strength, Split Tensile Strength & UPV	Viscocrete= 0.5%	1	0.33	2	400
							600
							800
						4	400
							600
							800
2	Mortar	Water Absorption, Apparent Porosity, Water Sorptivity, Compressive Strength, Split Tensile Strength & UPV	Viscocrete= 0.5%	1	0.33	2	400
							600
							800
						4	400
							600
							800
3	Mortar	Water Absorption, Apparent Porosity, Water Sorptivity, Compressive Strength, Split Tensile Strength & UPV	Viscocrete= 0.5%	1	0.33	2	400
							600
							800
						4	400
							600
							800
4	Mortar	SEM	Viscocrete= 0.5%	1	0.33	2	400
						4	400

4. Results And Discussion

4.1. Preamble

Ground Granulated Blast-furnace slag (GGBS) have been taken as base material for the preparation of Alkali activated GGBS mortar. Sodium hydroxide pellets (NaOH) and Sodium Silicate (SiO₂) solution were mixed to be used as alkali activator and standard river sand as fine aggregate. Workability, strength and durability varies both with the changes in composition & proportion of materials. Na₂O content of 4, 6, & 8 % by weight of GGBS has been taken with a constant silica content of 6% by weight of GGBS. Our first aim was to find out an optimum strength with a desired workability and second aim was to find a right proportion to have high performance mortar mix with good fire resisting properties and third aim was to compare the result with ordinary Portland Slag Cement mortar. For this, the experimental works have been carried out in 5 phases which developed according to the results of the previous phase.

In Phase-I , the experiments are being carried out by varying Na₂O as 4,6 & 8 % by weight of GGBS keeping constant silica content of 6% by weight of GGBS.To have different workable mix we varied Water/GGBS ratio as 0.38,0.35 & 0.33 for each variation of Na₂O% and performed all the test (refer Table.3.4.1)

In Phase-II, the experiments are again repeated with the test samples prepared by using admixture. Superplasticizer, Sika: Viscocrete 2004NS @ 1.2% by wt. of GGBS and a Retarder, Fosroc: Conplast R @ 1.5% by wt. of GGBFS have been used as admixture. The same sets of tests are again performed (refer Table: 3.4.2.).

In Phase-III, the mix, which has given the maximum compressive strength in Phase-II, was considered. The same mix was prepared again and the same tests are performed after exposing them to varying temperature of 400, 600 and 800 °C for a period of 2hrs and 4hrs for each temperature level (refer Table: 3.4.3). The results are then compared with the unexposed mortar specimens.

In Phase IV, the Alkali activated mortars have been prepared by using GGBS and Fly mixed in proportion 80:20, 70:30 and 60:40 in presence of same type and dosage of admixture (refer Table:3.4.4 & 3.4.6.). The results were then compared with AAGGBS and CSC mortar specimens.

In Phase V, the mortar specimens were exposed to same Temperature levels and duration of heating and the same set of experiments were performed (refer Table.3.4.5 & Table 3.4.7) and the results were compared.

4.2. Physio-Mechanical properties of Alkali Activated Specimens

4.2.1. Workability

The workability of alkali activated GGBS mortars is determined according to ASTM C-124 using mini slump cone. The diameter of the mortar flow is determined in two perpendicular directions immediately after mixing the alkali activated GGBFS mortars in Hobart mixer. The same test procedure is repeated for another time and the average of the two test results is taken.

4.2.1.1. Effect of Na₂O content in presence and absence of admixtures for different Water/GGBS ratio.

It was observed that workability decreases with the increase in alkali concentration (measured in terms of percentage of Na₂O) from 4 to 8% (by weight of GGBS).

Workability increases with the increases in Water/GGBFS ratio and the rate of increase was observed more at lower concentration of Na₂O.

Workability also increases with the addition of Admixtures but its effect on workability is less at lower water/GGBS ratio and at higher Na₂O content. The effect of admixture is best at our water/GGBS ratio of 0.38 and Na₂O content of 4% where flow increases by 21.21%.

Table 4.2.1.1.
Flow values for 4%, 6% & 8% Na₂O in presence and absence of admixtures

Water / GGBS	Flow Values (mm) for different Na ₂ O content					
	4% Na ₂ O (No Admixture.)	4% Na ₂ O (Admixture)	6%Na ₂ O (No Admixture)	6%Na ₂ O (Admixture)	8%Na ₂ O (No Admixture)	8%Na ₂ O (Admixture.)
0.33	135	142	130	138	125	130
0.35	155	175	145	165	140	160
0.38	165	200	155	180	150	170

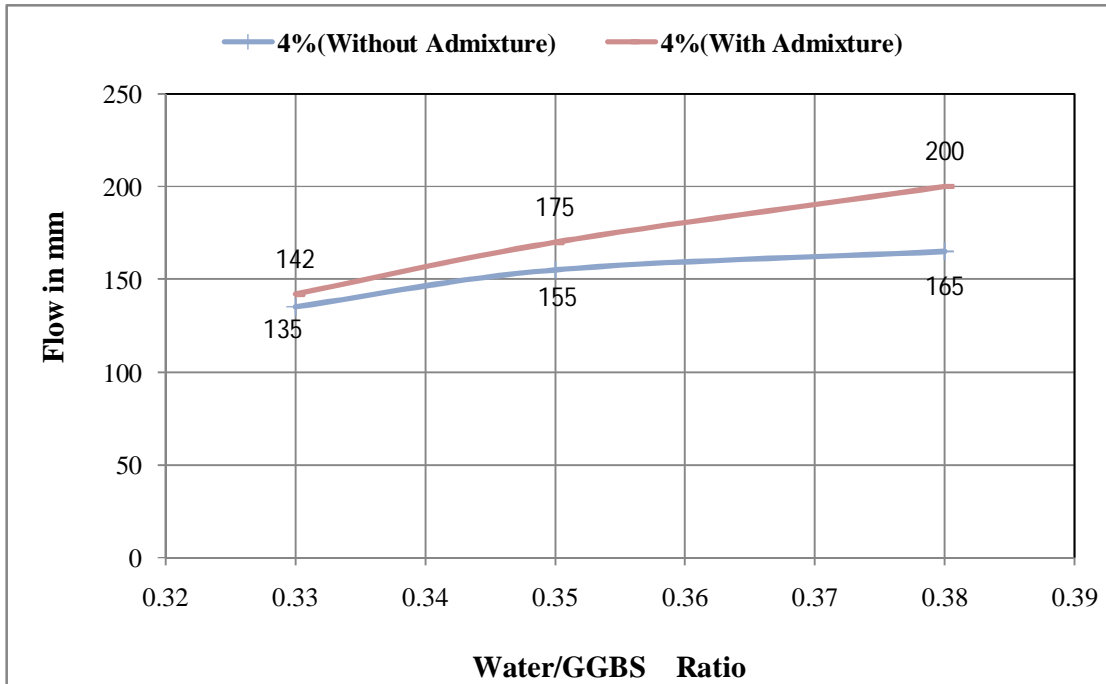


Fig.4.2.1.1(a)
Flow values for 4% Na₂O in presence and absence of Admixtures.

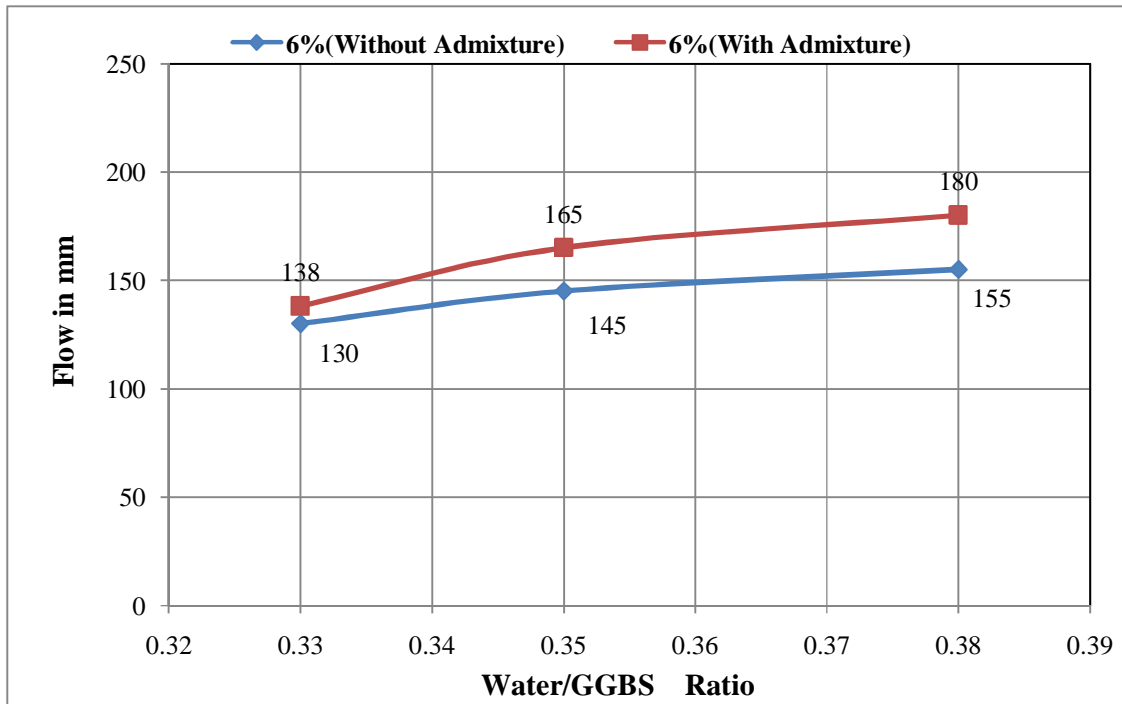


Fig.4.2.1.1(b)
Flow values for 6% Na₂O in presence and absence of Admixtures

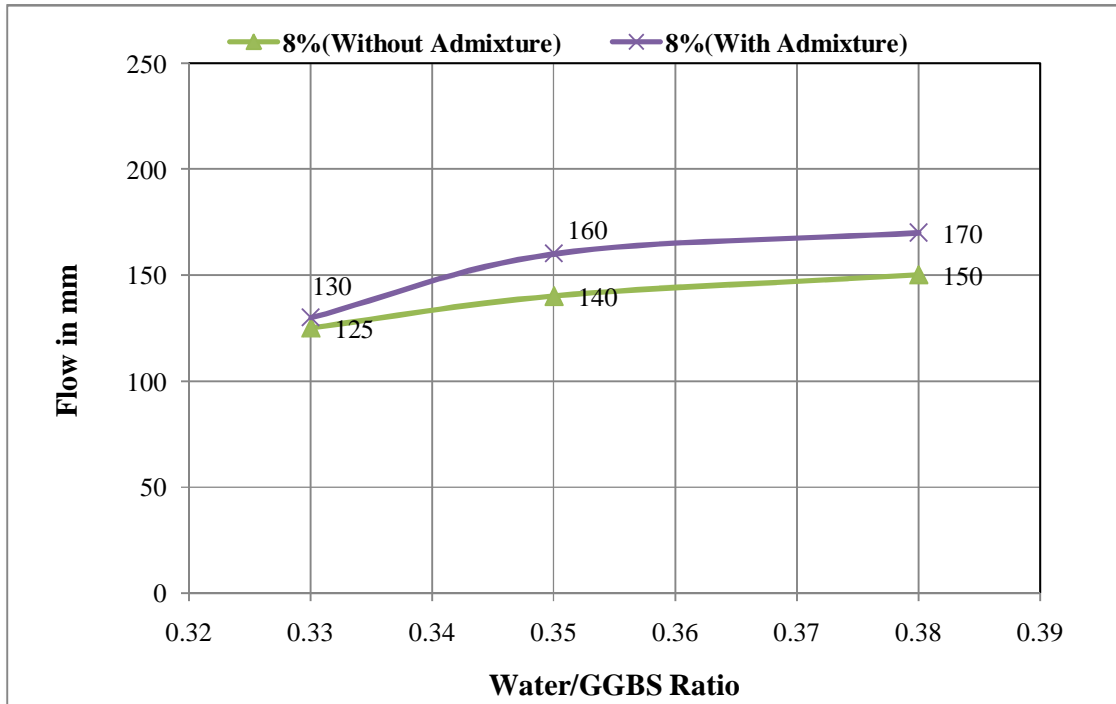


Fig.4.2.1.1(c). Flow values for 8% Na₂O in presence and absence of Admixtures

With increase in alkali content, the rate of chemical reaction in GGBS mortars increases resulting in more gel formation and as a result the resistance to flow increases which causes decrease in workability.

Addition of admixtures also increases the flow but it is more effective at higher Water/GGBS ratio and lesser alkali concentration. Viscocrete is carboxylic base admixtures and its effectiveness reduces at higher alkali concentration. Due to high reactivity of GGBS with alkali the hydration is very rapid and the availability of free water gets reduced and there is no sufficient moisture for the admixture to act upon.

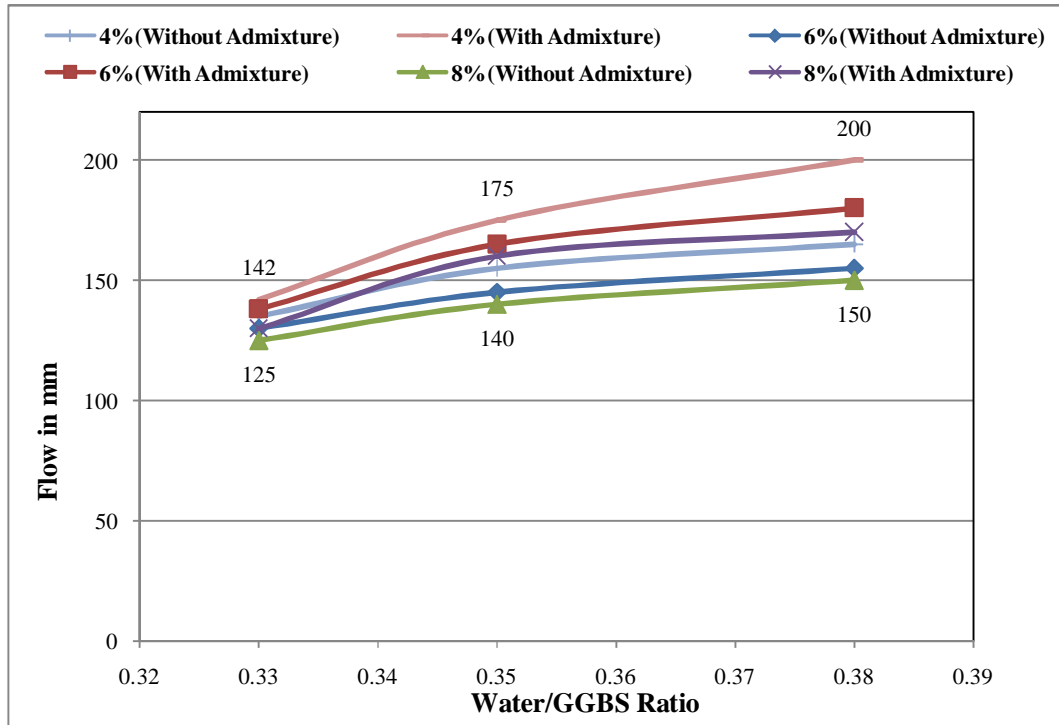


Fig.4.2.1.1(d).

Comparison of Flow values for 4%,6% & 8% Na₂O in presence and absence of Admixtures

4.2.2. Water Absorption:

The water absorption of alkali activated GGBFS mortars is determined according to ASTM standard. The average of three values are considered and presented in Table 4.2.2.1.

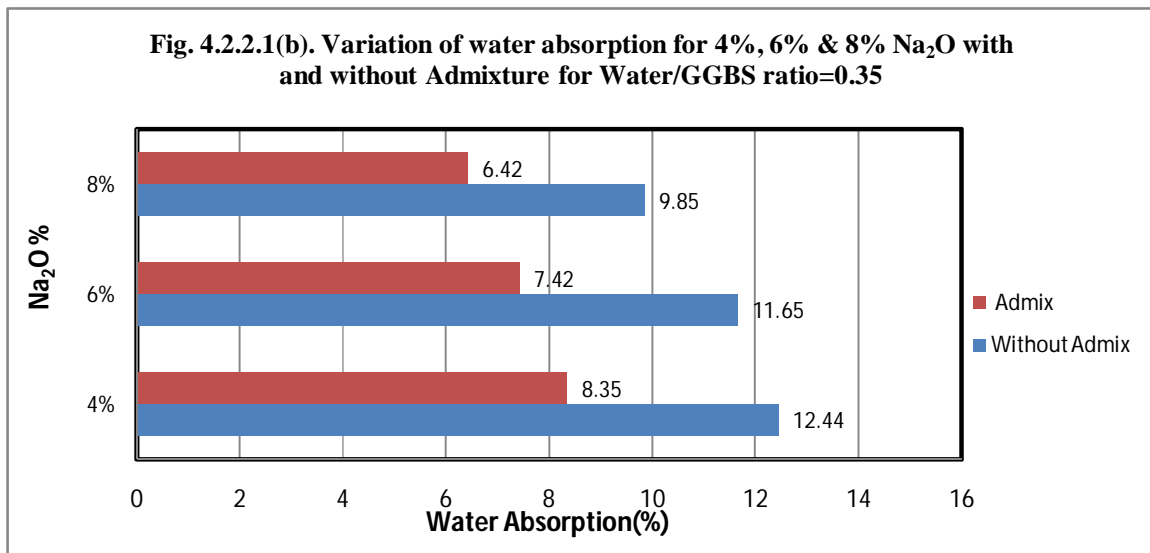
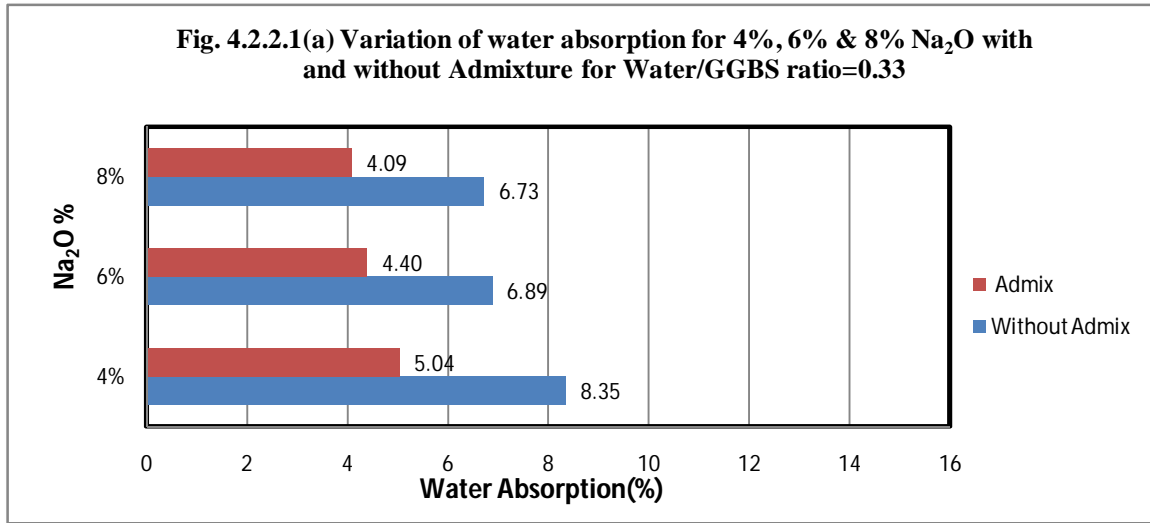
4.2.2.1. Effect of Na₂O content in presence and absence of admixtures for different water/GGBS ratio.

It has been observed that water absorption decreases with the increase of alkali concentration, but at 0.33 Water/GGBS ratio, an increase of water absorption was found at 8% Na₂O content.

In general there is decrease in water absorption on addition of admixtures but reduction is more effective at higher water/GGBS ratio and at lower alkali content. It has been observed that a 51.54 % reduction of water absorption is possible at 4% Na₂O content for 0.38 Water/GGBS ratio.

Table: 4.2.2.1
Water Absorption vs. Percentage Na₂O content for varying
Water/GGBS ratio in presence and absence of admixture

Na ₂ O	Water Absorption at different Water/GGBS ratio ratio					
	0.33(No Admix)	0.33(Admix)	0.35(No Admix)	0.35(Admix)	0.38(No Admix)	0.38(Admix)
4%	8.35	5.04	12.44	8.35	14.88	7.21
6%	6.89	4.40	11.65	7.42	11.15	6.37
8%	6.73	4.09	9.85	6.42	9.20	5.97



*Admix. =Admixture

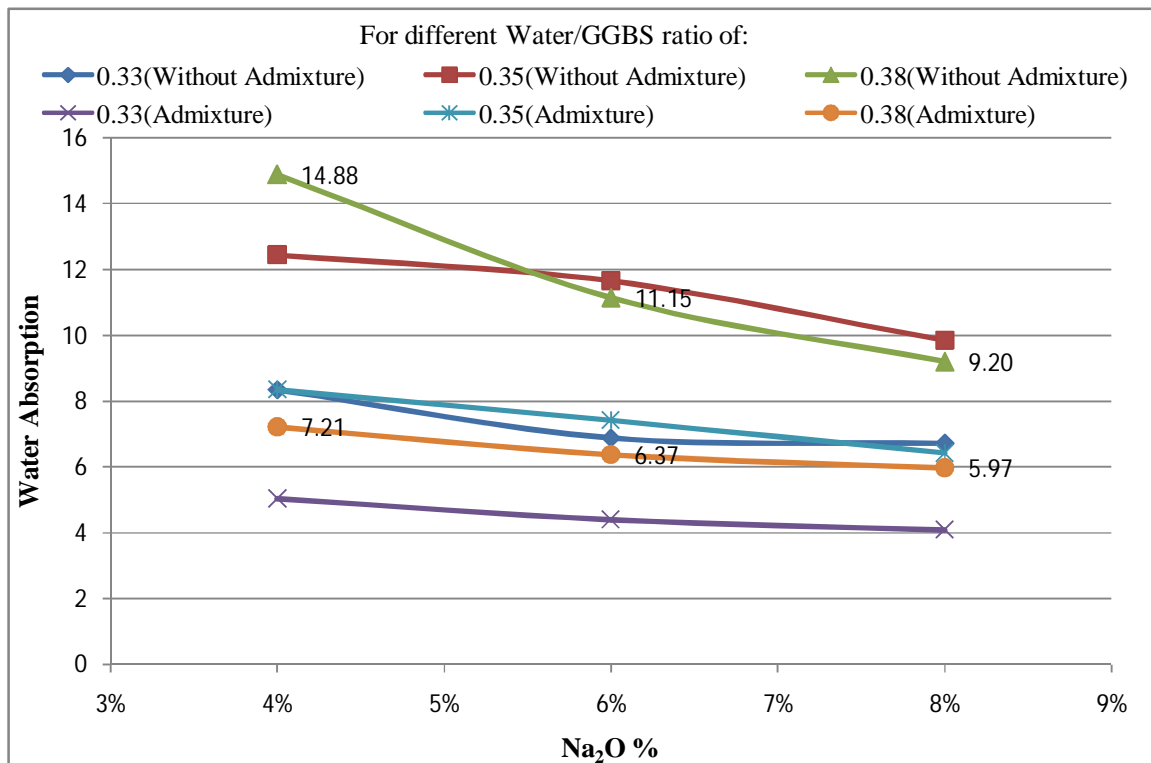
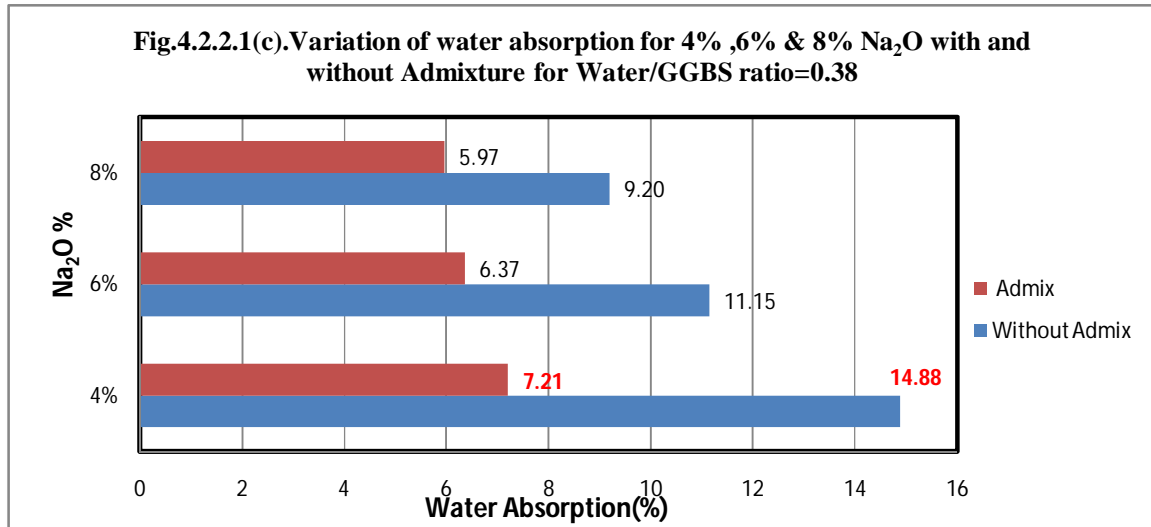


Figure 4.2.2.1(d).
Variation of Water Absorption for different percentage of Na₂O content and Water/GGBS ratio in presence and absence of admixture

At higher concentration of alkali the rate of chemical reaction was very fast with evolution of heat, Micro cracks are formed which resulted in reduction of water absorption. Again when Water/GGBS ratio is reduced to greater extent, sufficient water is not available for proper

compaction and more voids are produced resulted in reduction of water absorption. But in general with the increase in OH⁻ ions more Ca²⁺ ions present in GGBS get converted to calcium silicate hydrate (C-S-H) gel. With the increased amount conversion of Ca(OH)₂ to C-S-H gel resulted in decrease of water absorption.

4.2.3. Apparent Porosity.

The apparent porosity of alkali activated GGBFS mortars is determined according to ASTM standard. The average of three values are considered and presented in Table 4.2.3.1

4.2.3.1. Effect of Na₂O content in presence and absence of admixtures for different water/GGBS ratio.

It has been observed that apparent porosity decreases with the increase of alkali concentration but at 0.33 Water/GGBS ratio an increase of apparent porosity was found at 8% Na₂O content..

In general there is decrease in apparent porosity on addition of admixtures but reduction is more effective at higher Water/GGBS ratio and lower alkali content. It has been observed that a reduction of 48.39% of apparent porosity is possible at 4% Na₂O content for 0.38 Water/GGBS ratio.

**Table: 4.2.3.1
Apparent porosity vs. Percentage Na₂O content for varying Water/GGBS ratio in presence and absence of admixture.**

Na ₂ O	Apparent Porosity at different Water/GGBS ratio					
	0.33 (No Admixture)	0.33 (Admixture)	0.35 (No Admixture)	0.35 (Admixture)	0.38 (No Admixture)	0.38 (Admixture)
4%	10.4	6.49	15.77	11.01	17.68	9.13
6%	8.82	5.68	14.59	8.83	13.90	8.58
8%	8.32	5.38	12.53	8.00	11.46	7.55

Fig.4.2.3.1(a).
Variation of apparent porosity for 4%, 6% & 8% Na₂O in presence and absence of Admixture for Water/GGBS ratio=0.33

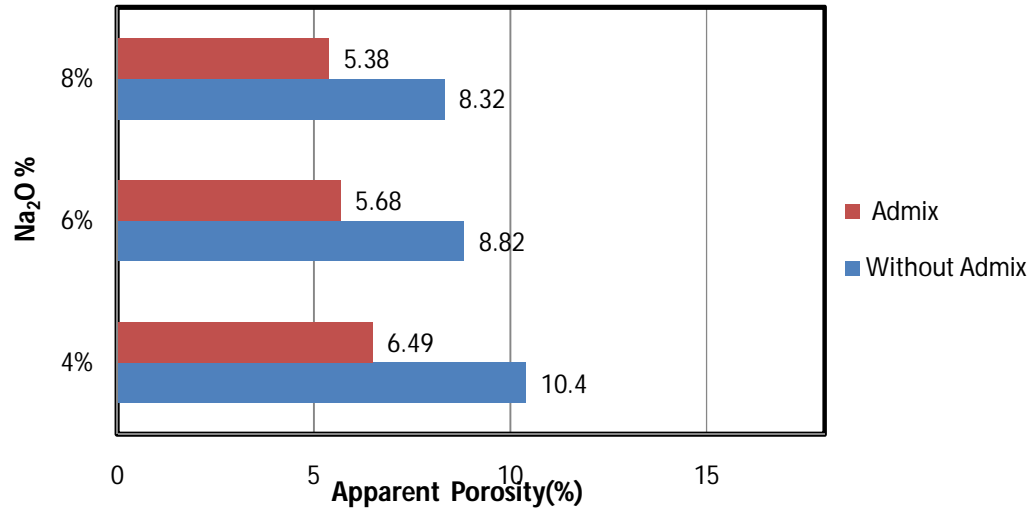
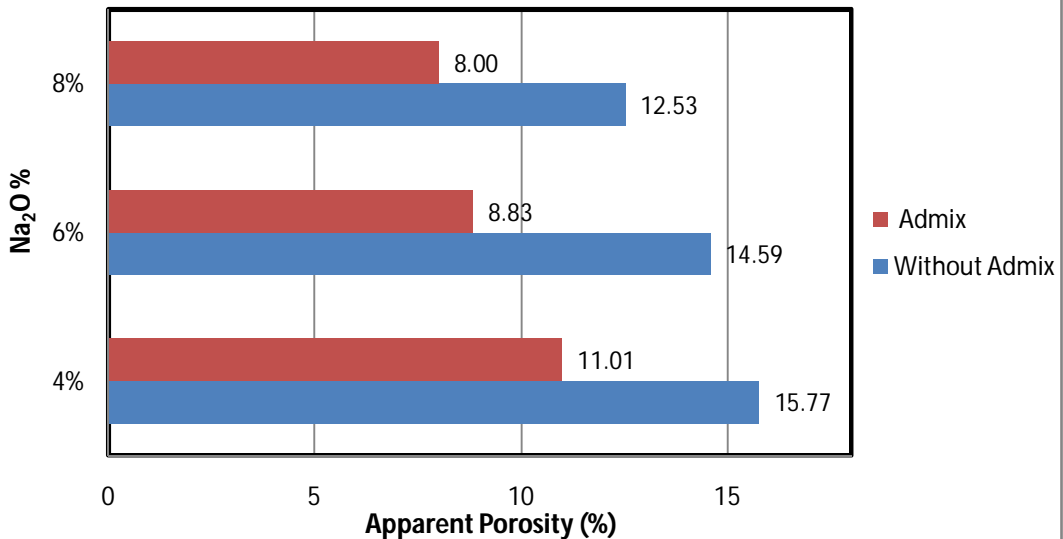
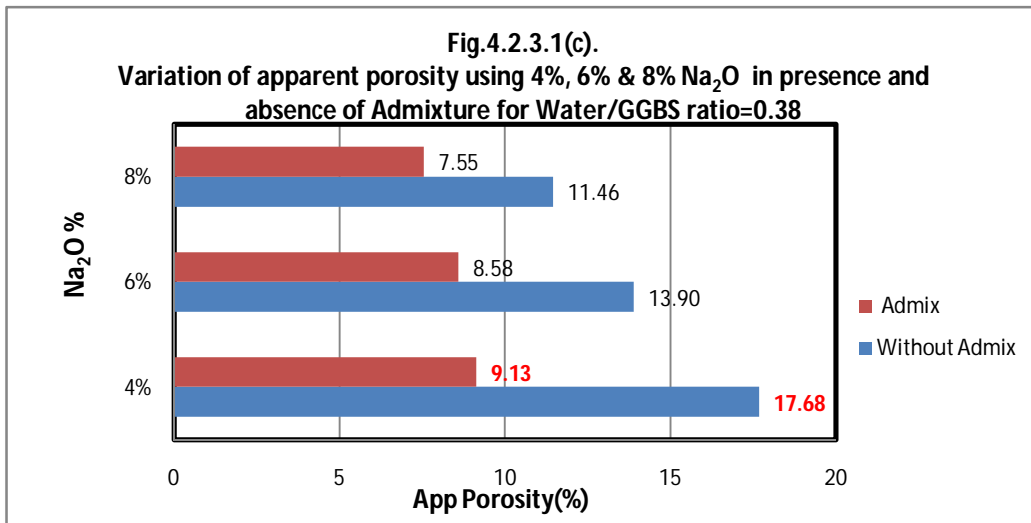


Fig.4.2.3.1(b).
Variation of apparent porosity for 4%, 6% & 8% Na₂O in presence and absence of Admixture for Water/GGBS=0.35





*Admix. =Admixture

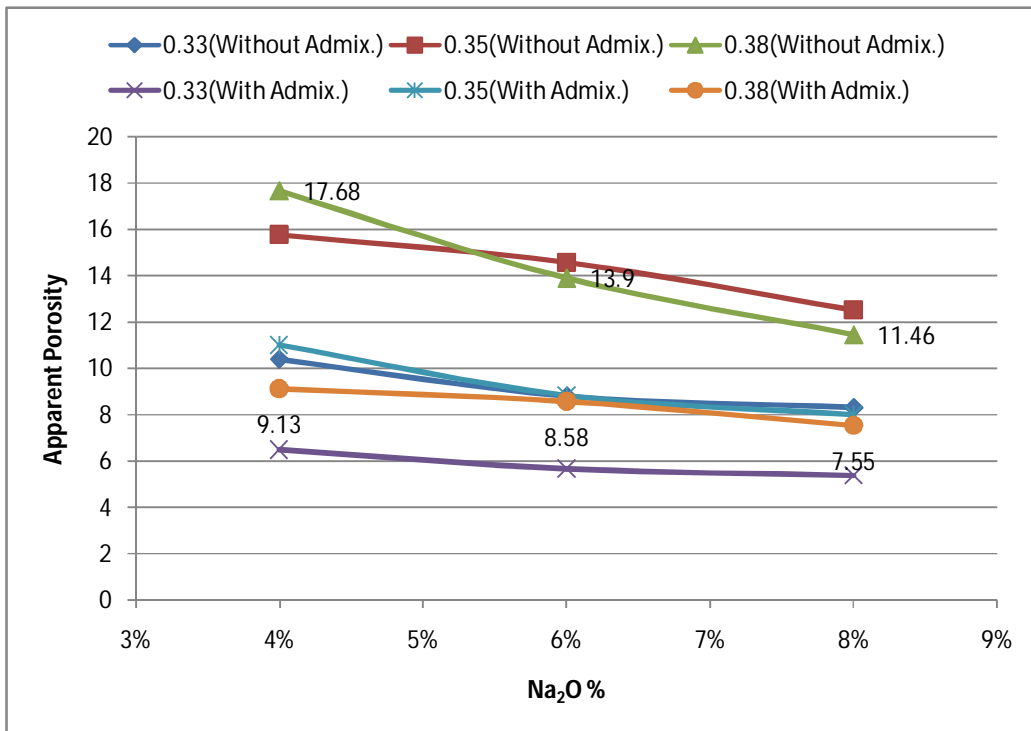


Figure 4.2.3.1(d)
Variation of App. Porosity with respect to Na₂O % for 0.33,0.35,0.38 Water/GGBS ratio ratio with and without Admixture.

At higher concentration of alkali the rate of reaction is very fast with evolution of heat as a result micro cracks are more hence there is marked reduction of apparent porosity. Again when Water/GGBS ratio ratio is reduced sufficient water is not available for proper compaction and more voids are produced resulting in reduction of apparent porosity.

4.2.4. Sorptivity.

The characteristic of pore structure can be assessed by measuring the rate of unidirectional capillary absorption of water by unsaturated alkali activated composite mortars when placed in contact with water when no head of water exists, is known as sorptivity. Sorptivity measurement has primary importance in durability assessment of alkali activated composite mortars.

4.2.4.1. Effect of Na_2O content in presence and absence of admixtures for different Water/GGBS ratio.

For Water/GGBS ratio= 0.33:

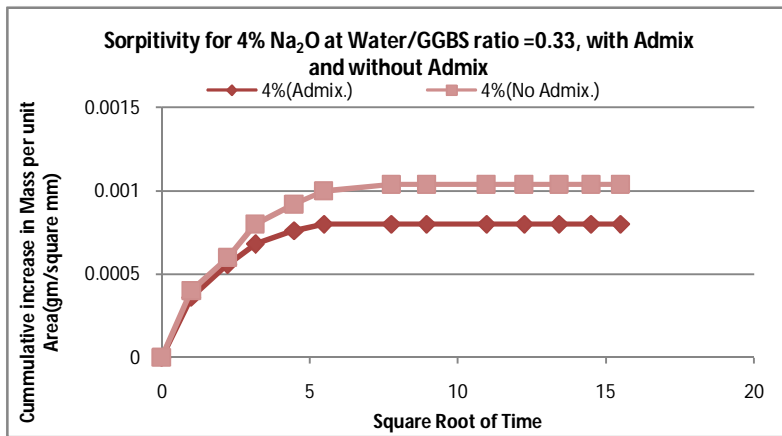


Fig 4.2.4.1(a)

Sorptivity for 4% Na_2O at Water/GGBS ratio=0.33 in presence and absence of admixture

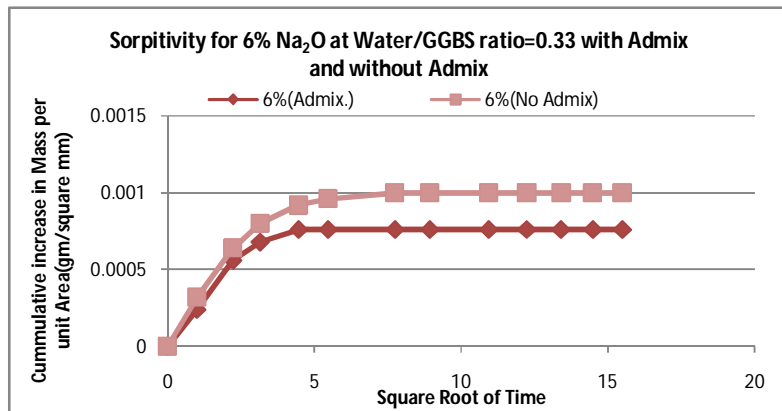


Fig 4.2.4.1(b)

Sorptivity for 6% Na_2O at Water/GGBS ratio=0.33 in presence and absence of admixture

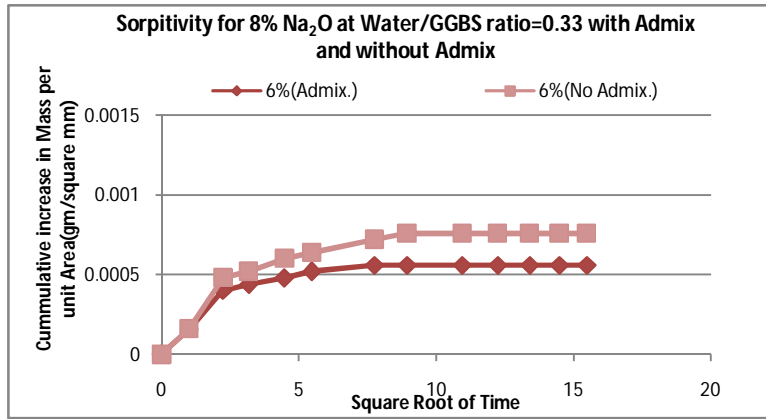


Fig 4.2.4.1(c)
Sorptivity for 8% Na₂O at Water/GGBS ratio=0.33 in presence and absence of admixture

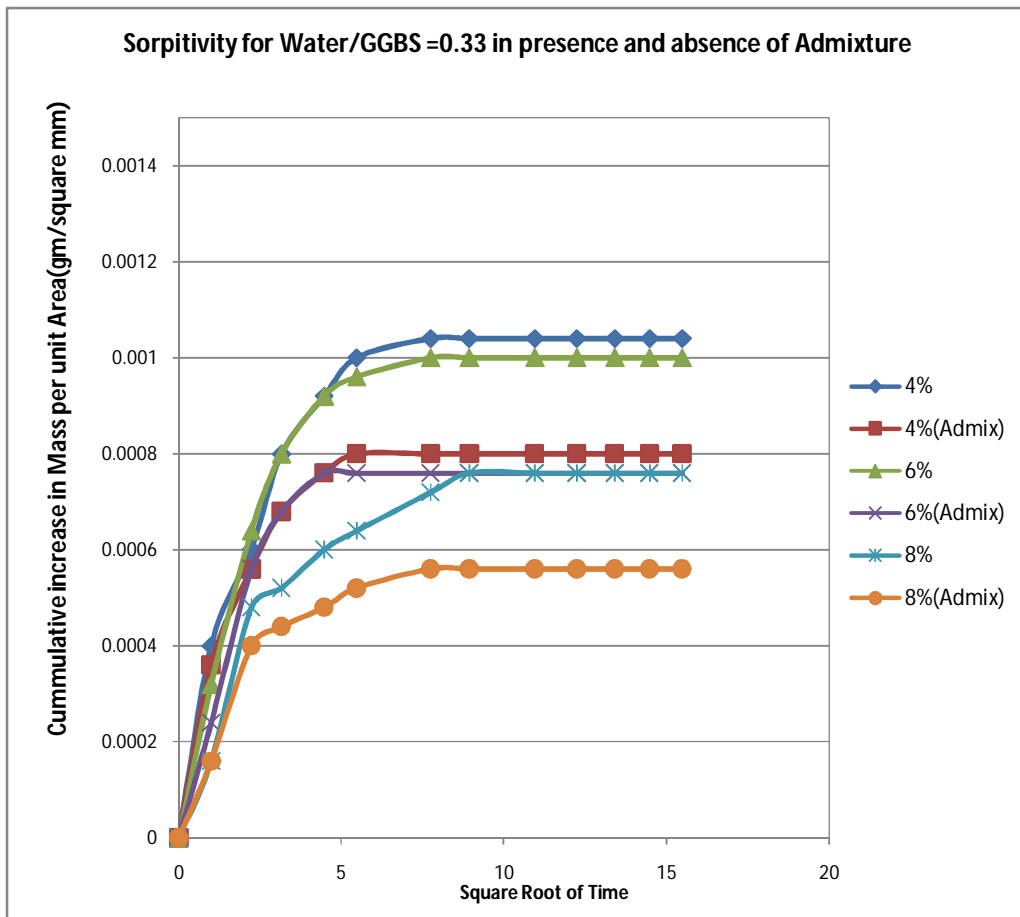


Fig 4.2.4.1(d)
Sorptivity for Water/GGBS ratio=0.33 in presence and absence of Admixture

For Water/GGBS ratio= 0.35:

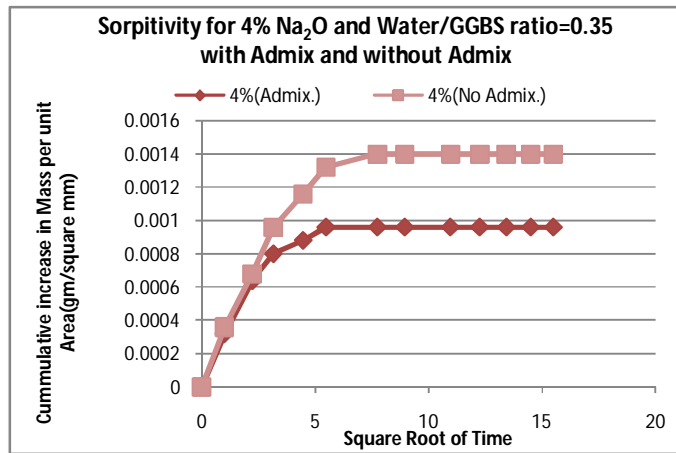


Fig 4.2.4.1(e)
Sorptivity for 4% Na₂O at Water/GGBS ratio=0.35 in presence and absence of admixture

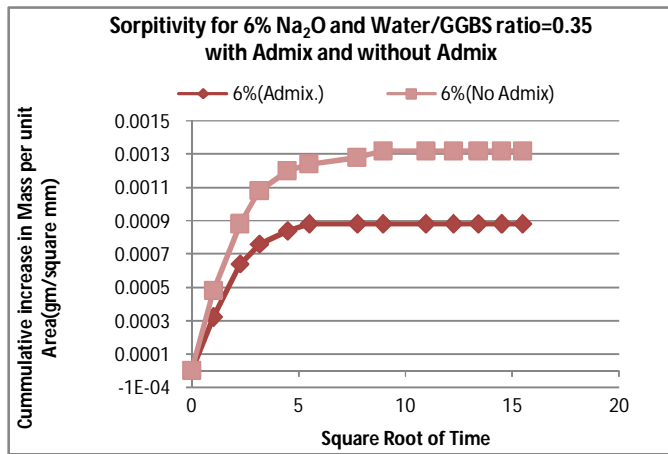


Fig 4.2.4.1(f)
Sorptivity for 6% Na₂O at Water/GGBS ratio=0.35 in presence and absence of admixture

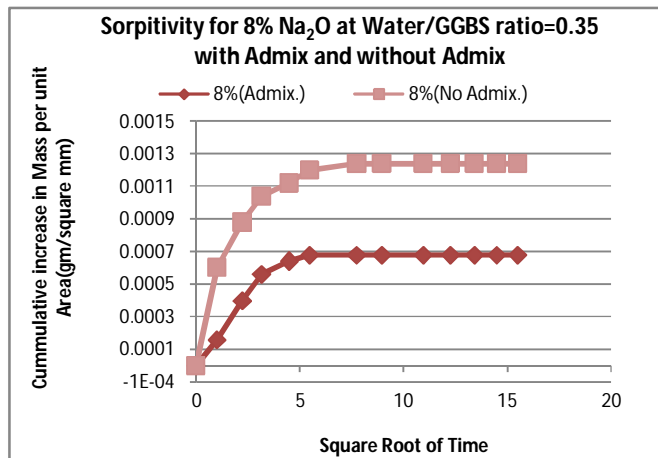


Fig 4.2.4.1(g)
Sorptivity for 8% Na₂O at Water/GGBS ratio=0.35 in presence and absence of admixture

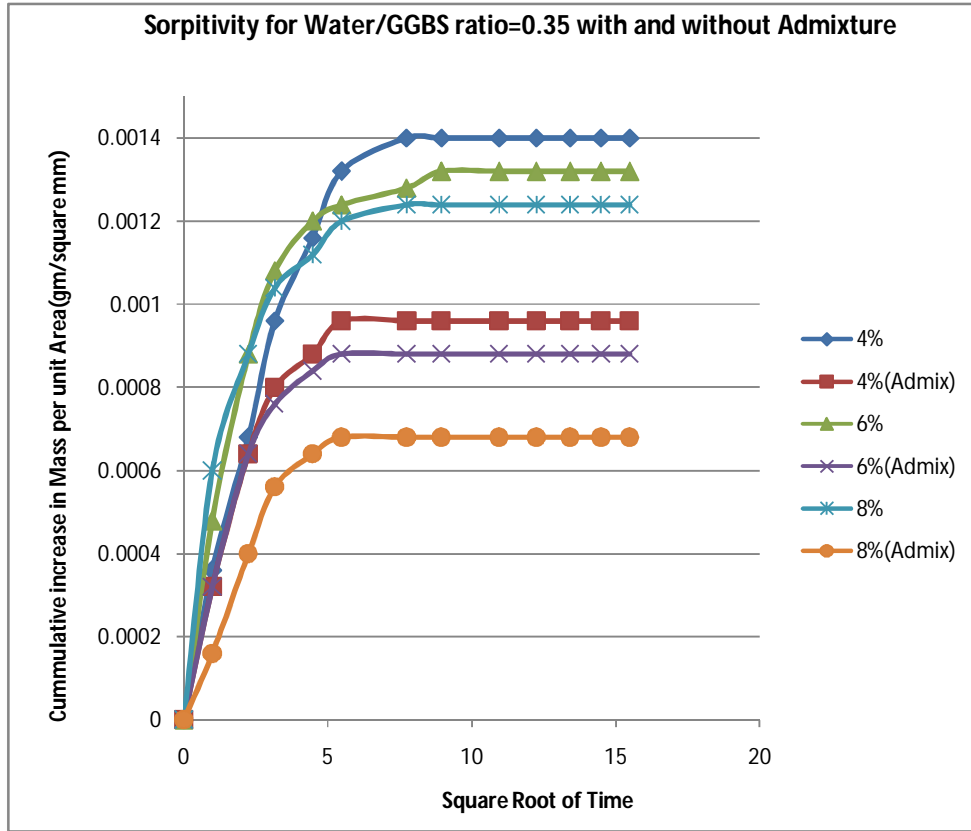


Fig 4.2.4.1(h).
Sorptivity for Water/GGBS ratio =0.35 in presence and absence of admixture

For Water/GGBS ratio ratio= 0.38:

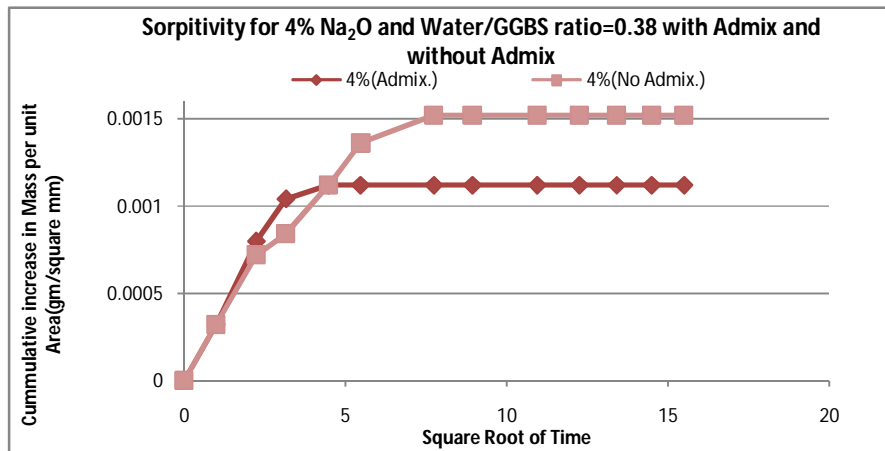


Fig 4.2.4.1(i)
Sorptivity for 4% Na₂O at Water/GGBS ratio=0.38 in presence and absence of admixture

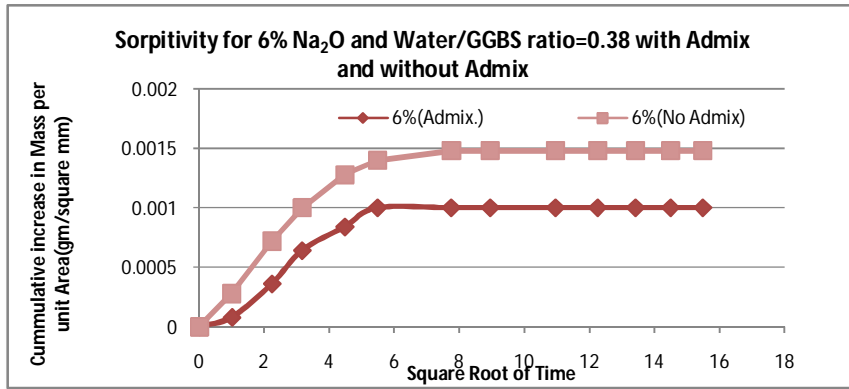


Fig 4.2.4.1(j)

Sorptivity for 6% Na₂O at Water/GGBS ratio=0.38 in presence and absence of admixture

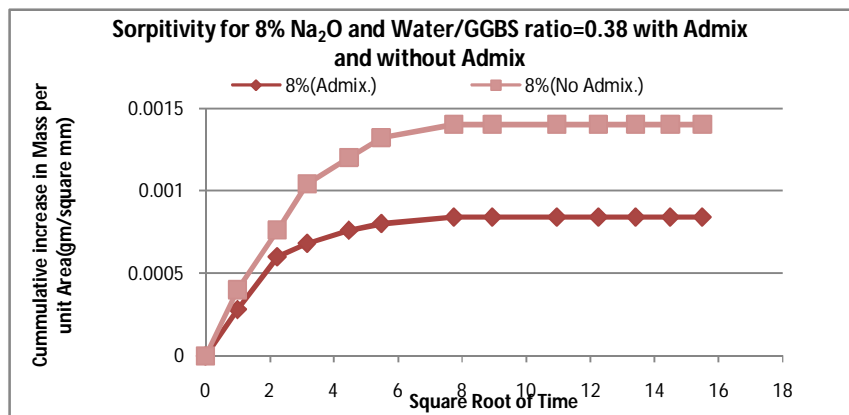


Fig 4.2.4.1(k)

Sorptivity for 8% Na₂O at Water/GGBS ratio=0.38 in presence and absence of admixture

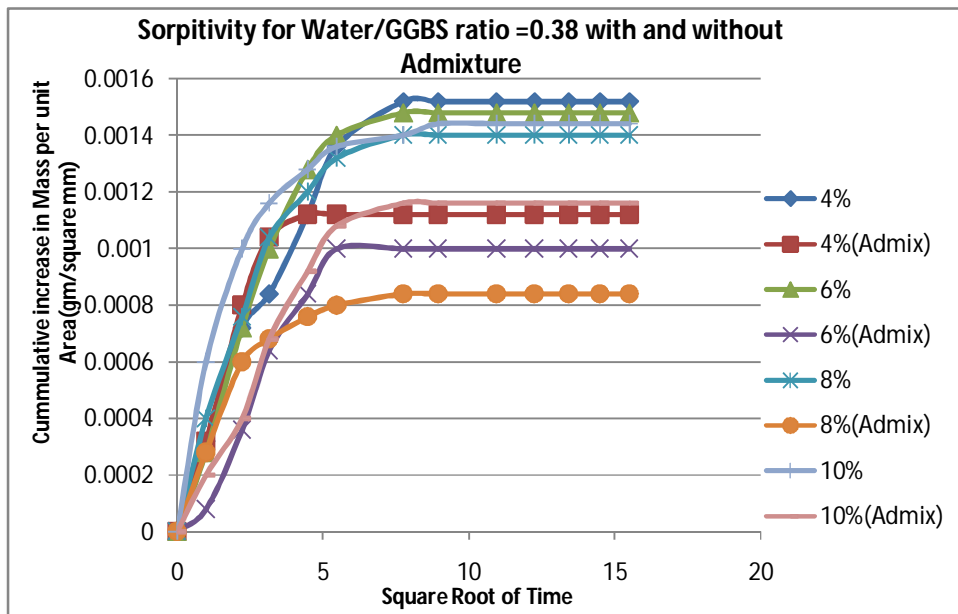


Fig 4.2.4.1(l)

Sorptivity for W/GGBS ratio =0.38 in presence and absence of admixture

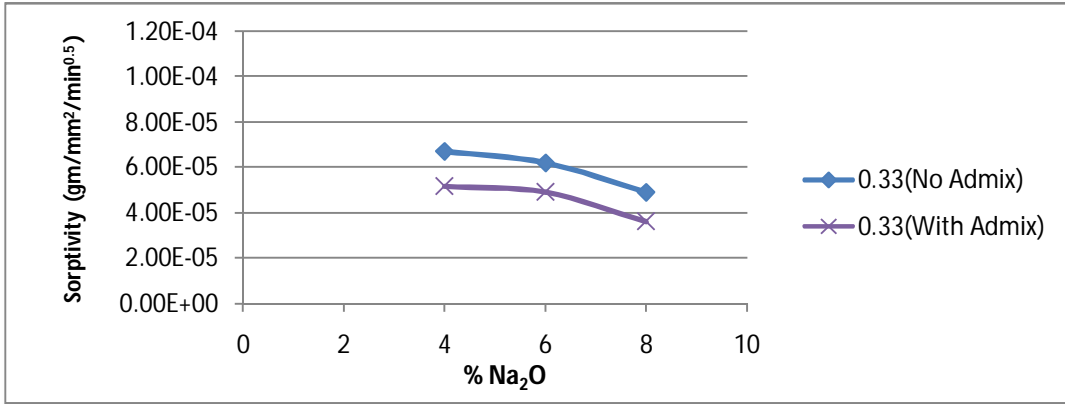


Fig.4.2.4.1(m).
Sorptivity at Water/GGBS ratio of 0.33 in presence and absence of admixtures for varying Na₂O content.

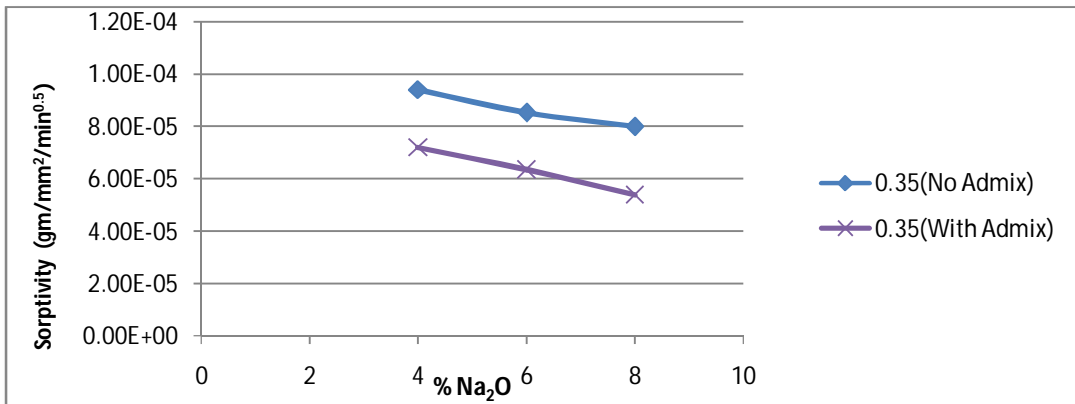


Fig. 4.2. 4.1(n).
Sorptivity at Water/GGBS ratio of 0.35 in presence and absence of admixtures for varying Na₂O content.

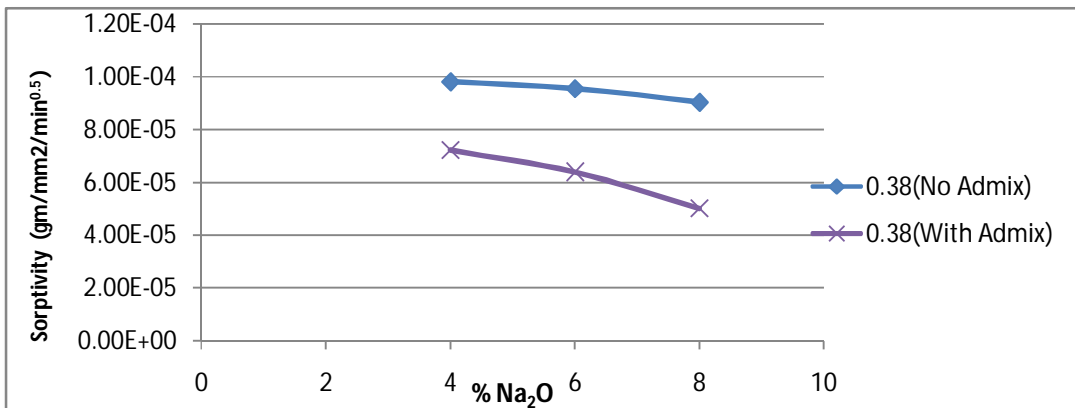


Fig. 4.2.4.1(o).
Sorptivity at Water/GGBS ratio of 0.38 in presence and absence of admixtures for varying Na₂O content.

The cumulative increase in mass of alkali activated composite mortars due to unidirectional capillary water absorption with square root of time is shown in the figures above. Initial rate of water absorption through capillarity is almost linear. After four hours the unsaturated alkali activated composite mortars gets almost saturated and no further water absorption occurs. It is observed that cumulative water absorption decreases with increasing percentage of alkali (NaOH). From the graphical representation it is observed that water sorptivity is more at lower alkali content (4% Na₂O). Water sorptivity decreases with increase in alkali content.

The sorptivity decreases with increase in alkali content because as alkali content increases the rate of reaction of GGBFS with activator solution also increases resulting in more gel/binder formation which makes the alkali activated GGBFS mortars more compact with less pores. There is notable decrease in Sorptivity using admixtures a maximum reduction of 40% is achieved at 0.35 Water/GGBS ratio at 8% Na₂O.

Table 4.2.4.1
Sorptivity vs. Percentage Na₂O content for different
Water/GGBS ratio in presence and absence of admixture.

Mix ID	%Na ₂ O	%SiO ₂	W/ GGBS ratio	Dry weight of specimen	Mass after 4 hrs of capillary absorption	Increase in mass of specimen	Increase in mass/cross sectional ares	Sorptivity S=i(t) ^{0.5}
S _p -1 No Admixture	4	6	0.33	260	262.6	2.6	0.01040	6.71E-05
			0.35	266.4	269.9	3.5	0.00140	9.4 E-05
			0.38	267.4	271.2	3.8	0.00152	9.81E-05
Admixture			0.33	265	267	2	0.00080	5.16E-05
			0.35	268	270.4	2.4	0.00096	6.2E-05
			0.38	272	274.8	2.8	0.00112	7.23E-05
S _p -2 No Admixture	6	6	0.33	266.2	268.6	2.4	0.00096	6.2E-05
			0.35	271.3	274.6	3.3	0.00132	8.52E-05
			0.38	266.4	270.1	3.7	0.00148	9.55E-05
Admixture			0.33	272	273.9	1.9	0.00076	4.91E-05
			0.35	274	276.2	2.2	0.00088	7.19E-05
			0.38	273	275.5	2.5	0.0010	6.4 E-05
S _p -3 No Admixture	8	6	0.33	279.5	281.4	1.9	0.00076	4.91E-05
			0.35	264.2	267.3	3.1	0.00124	8.0 E-05
			0.38	278.2	281.7	3.5	0.00140	9.04E-05
S _p -3 Admixture			0.33	277.4	278.8	1.4	0.00056	3.61E-05
			0.35	273.8	275.5	1.7	0.00068	4.39E-05
			0.38	272	274.1	2.1	0.00084	5.42E-05

4.2.5. Compressive Strength

4.2.5.1. Effect of Na₂O content in presence and absence of admixtures for different water/GGBS ratio.

Table 4.2.5.1
Compressive Strength vs. Percentage Na₂O content for different
Water/GGBS ratio in presence and absence of admixture

Water/GGBS	28 days Compressive Strength for different Na ₂ O.(in Mpa)					
	4%		6%		8%	
	No Admixture	Admixture	No Admixture	Admixture	No Admixture.	Admixture.
0.33	40.3	50.2	46.5	56.5	52.2	62.5
0.35	38.4	48.8	42.9	53.3	48.2	58.6
0.38	35.1	46.2	39.5	49.6	45.6	55.6

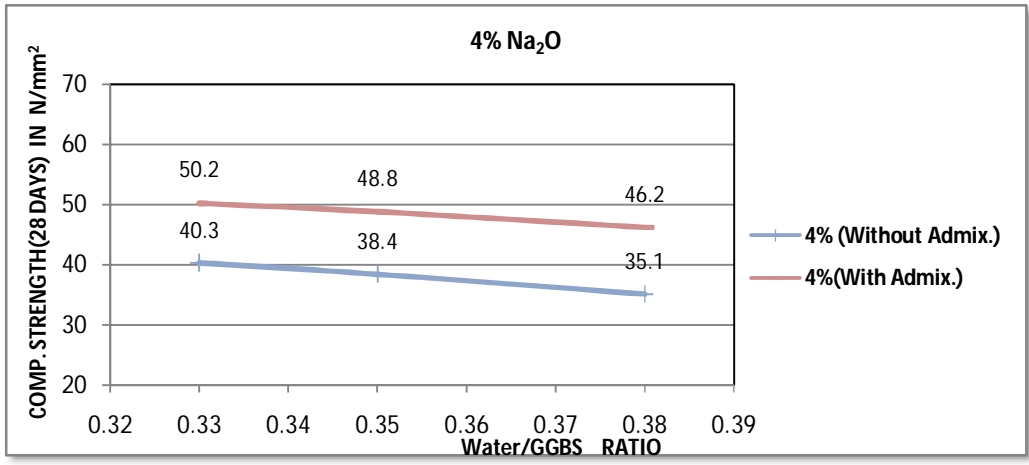


Fig. 4.2.5.1(a)
Compressive strength at 4% Na₂O in presence and absence of admixture

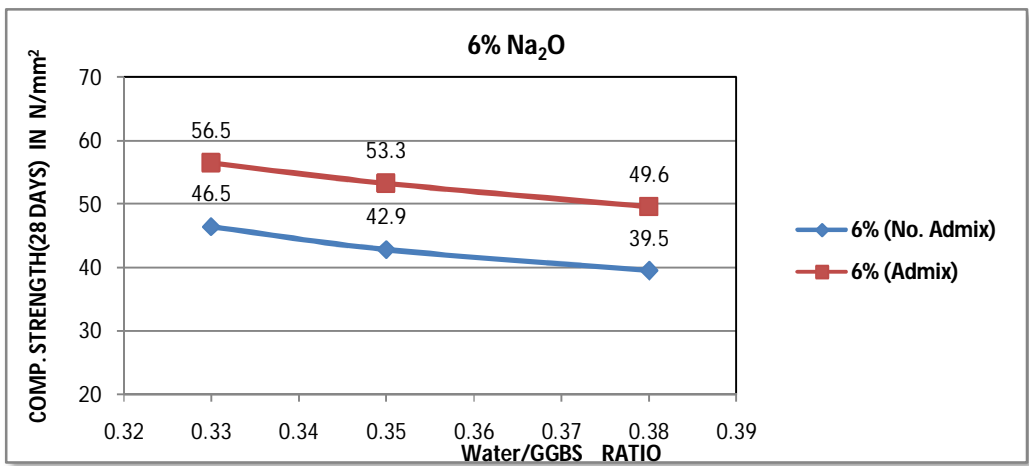


Fig. 4.2.5.1(b)
Compressive strength at 6% Na₂O in presence and absence of admixture

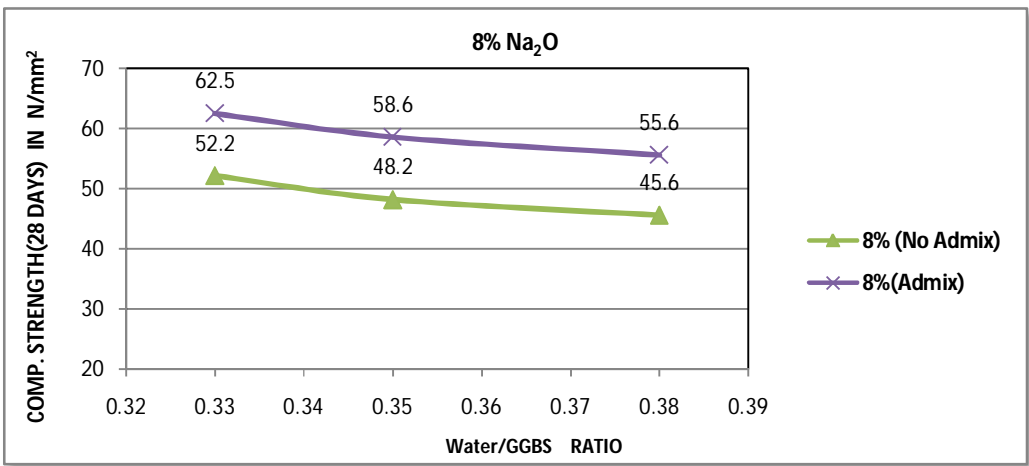


Fig. 4.2.5.1(c).
Compressive strength at 8% Na₂O in presence and absence of admixture

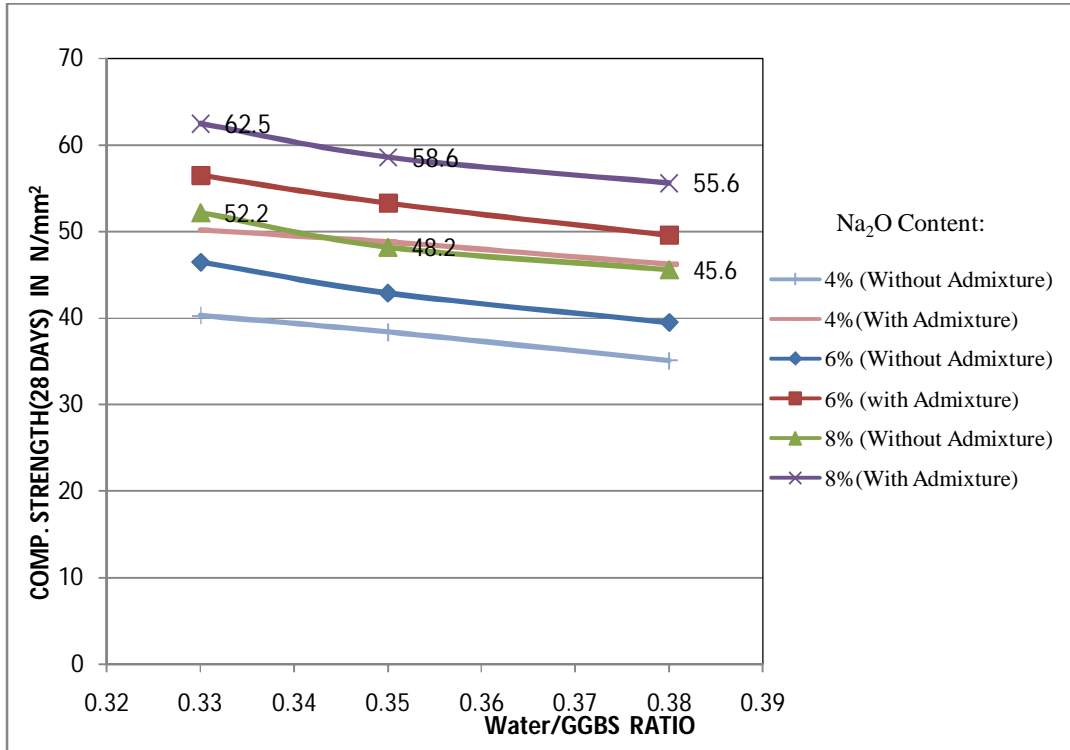


Fig. 4.2.5.1(d).
Compressive strength vs. Water/GGBS ratio for different Na₂O content in presence and absence of Admixtures.

The average values of compressive strength are presented graphically under varying Water/GGBS ratio, without and with admixtures under different Na₂O content. It has been observed from the graphical representation that compressive strength increases with increase in alkali content from 4% to 8% Na₂O under reduction of water/GGBS ratio from 0.38 to 0.33. With increase in OH⁻ concentration of the alkali activator solution, the compressive strength increases because the anion group [OH⁻] react with the Ca²⁺ ions present in GGBFS to produce Ca-compounds which serve as the main binder material or cementations material. With the increase of OH⁻ ions due to the increase of Na₂O concentration, more amounts of Ca-compounds which are nothing but calcium silicate hydrates are generated as reaction products. The increased quantity of cementations material causes increase in strength. There is a average increase of 29.7% compressive strength with the increase of Na₂O concentration of 4 to 8%. The addition of admixtures increases the compressive strength but its percentage increase is maximum (31.62%) for lower alkali concentration and higher Water/GGBS ratio. These may due to the fact that higher alkali concentration reduces the action of poly carboxylic admixture.

4.2.6. Split Tensile Strength.

4.2.6.1. Effect of Na₂O content in presence and absence of admixtures for different water/GGBS ratio.

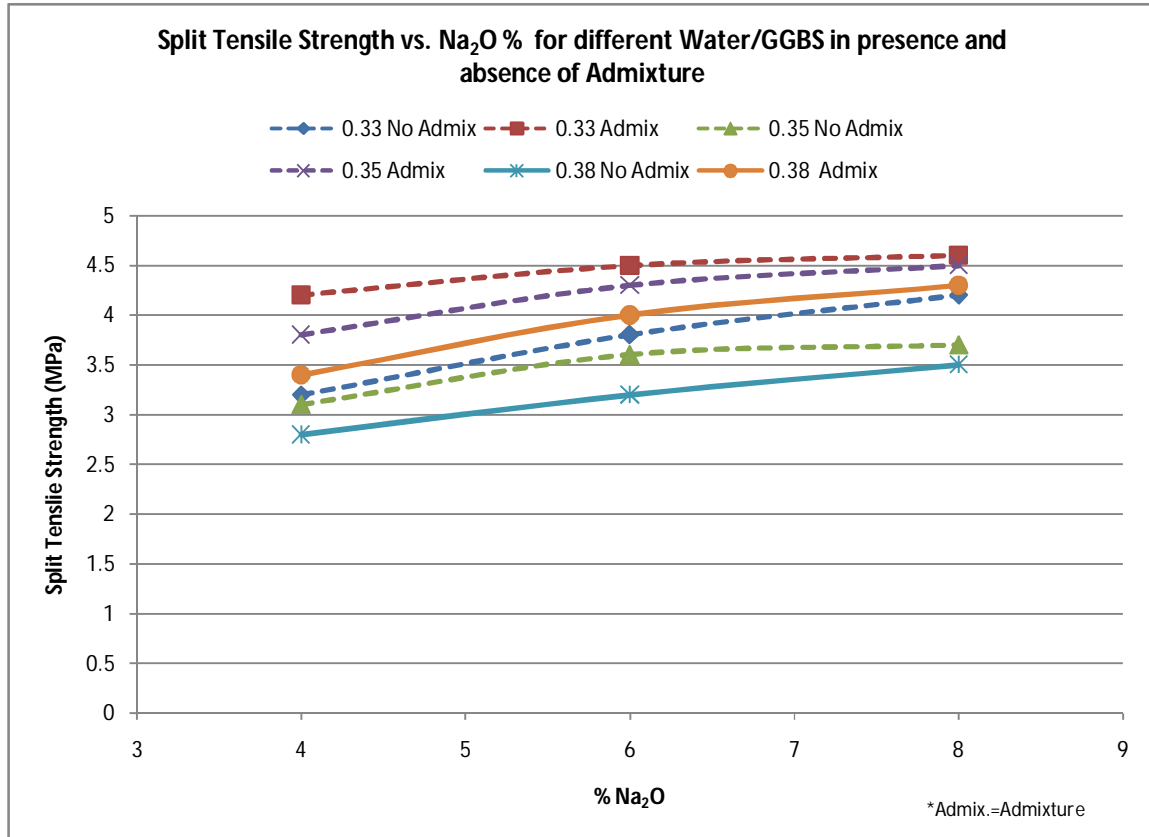


Fig.4.2.6.1.
Split Tensile Strength vs. Na₂O in presence and absence of Admixture

Table 4.2.6.1
Split Tensile Strength in presence and absence of admixtures

Water/GGBS ratio	0.33		0.35		0.38	
	Split Tensile Strength.(N/mm ²)					
Na ₂ O %	No Admix.	Admix.	No Admix.	Admix.	No Admix.	Admix.
4	3.2	4.2	3.1	3.8	2.8	3.4
6	3.8	4.5	3.6	4.3	3.2	4.0
8	4.2	4.6	3.7	4.5	3.5	4.3

An improvement of Split Tensile Strength has been observed on application of Admixture. For 6% Na₂O content at 0.35 Water/GGBS ratio there is maximum increase of Split tensile strength of

44.4% on application admixture (1.2% Viscocrete and 1.5% returder. Due to application of admixture there may be reduction of shrinkage cracks which resulted in higher split tensile strength.

4.2.7. Ultrasonic Pulse Velocity Test.

Ultrasonic Pulse velocity is done on 28 day cured cylindrical mortar specimens of 75 mm diameter and 150 mm length. Transducers are held along length as shown in Fig. with adequate acoustical coupling between the mortar surface and the face of each transducer. Path length (150 mm) is measured and divided by the recorded time (μ sec), which eventually gives the pulse velocity.

4.2.7.1.Effect of Na₂O content in presence and absence of admixtures for different water/GGBS ratio.

Table 4.2.7.1.
UPV values for AAS mortars:

Water/GGBS ratio	4% Na ₂ O		6%Na ₂ O		8% Na ₂ O	
	UPV in km/s					
	No Admix.	Admix.	No Admix.	Admix.	No Admix.	Admix.
0.33	3.15	3.92	3.62	4.37	3.94	4.83
0.35	2.85	3.67	3.34	4.22	3.42	4.52
0.38	2.65	3.67	2.98	3.95	2.95	3.96

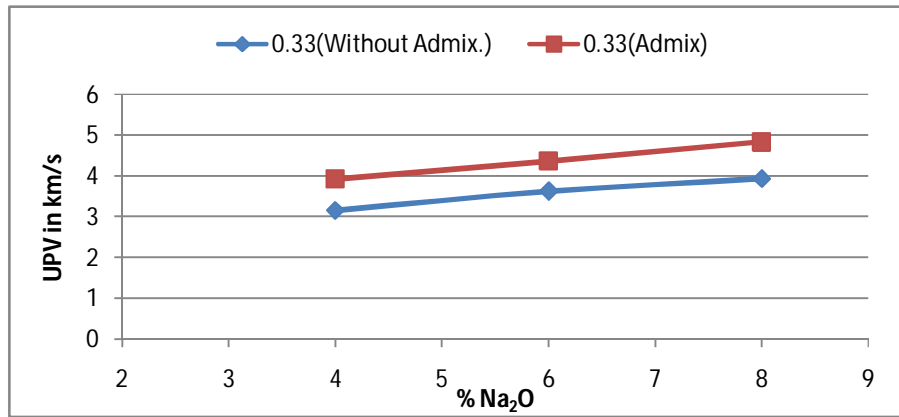


Fig.4.2.7.1(a).

UPV vs. % Na₂O in presence and absence of admixture for 0.33 Water/GGBS ratio

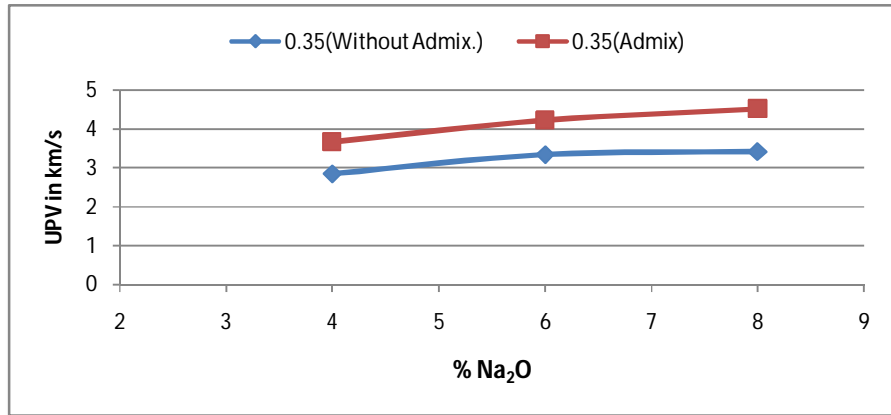


Fig.4.2.7.1(b).

UPV vs. % Na₂O in presence and absence of admixture for 0.35 Water/GGBS ratio

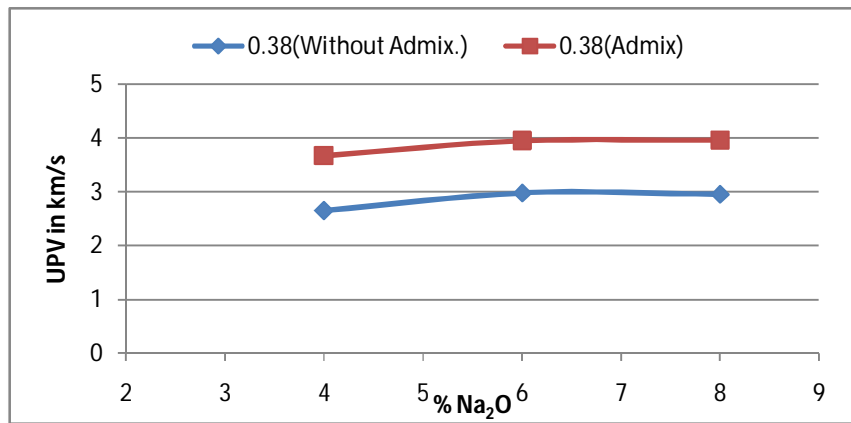


Fig.4.2.7.1(c).

UPV vs. % Na₂O in presence and absence of admixture for 0.38 Water/GGBS ratio

It has been observed from the graphical representation that UPV value increases with increase in alkali content from 4% to 8% Na₂O. Its value also increases with the reduction of Water/GGBS ratio from 0.38 to 0.33.

An average increase of 16% UPV value with the increase of Na₂O content of 4 to 8% has been found. The addition of admixtures increases the UPV value, but its percentage increase is maximum (38.49%) for lower alkali concentration and higher Water/GGBS ratio, which is in confirmation with compressive strength values.

4.2.8. Comparison of properties Alkali Activated Slag mortar with Alkali Activated Slag-Fly Ash mortar & Normal Slag Cement(PSC) mortar.

Inorder to improve the property of alkali activated mortar GGBS is partially replaced by Fly-Ash in proportion 80:20, 70:30 and 60:40 with water/binder ratio of 0.33 and same amount of Admixture was used as done in preparation of AAGGBS mortar. Mortar samples are also prepared using conventional slag cement (PSC) using same proportion of sand (1:1) .Viscocrete @0.5% was used as a admixture. The same sets of experiments are done and the results are represented graphically.

4.2.8.1.Apparent Porosity, Water Absorption & Water Sorptivity.

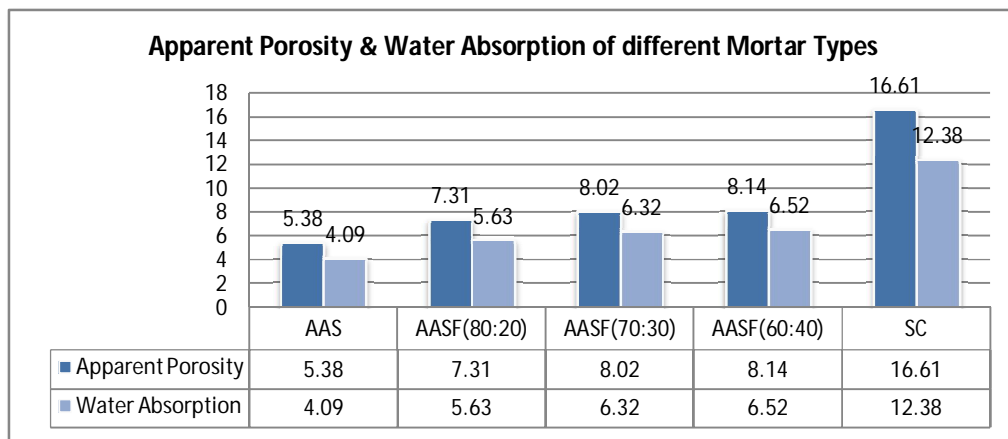


Fig.4.2.8.1(a).

Apparent Porosity & Water Absorption of different Mortar Types

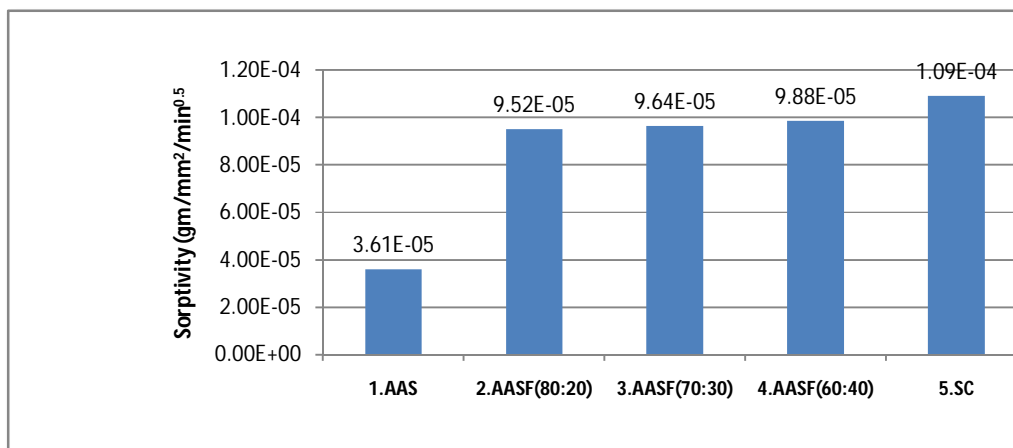


Fig.4.2.8.1.(b).

Sorptivity of different mortar types

*AASF(80:20)=AASF(Slag:Flyash=80:20)

Blended Alkali activated mortars are prepared with different proportion GGBS and Fly Ash (80:20, 70:30 and 60:40) with same proportion of alkali activators and Admixtures. Na_2O @ 8%; Na_2SiO_3 @ 6%; W/Binder ratio @ 0.33; Admixture @ 1.2% Sika: Viscocrete 2004 NS and 1.5% Fosroc: Conplast R and Binder: Sand @ 1:1 are taken to prepare the mixes.

A substantial reduction in Apparent Porosity and Water Absorption was seen for Alkali activated mortars over normal Slag Cement Mortar. However there was increase of both Water Absorption and Porosity for blended alkali activated mortars with the increase of fly ash proportion.

The reduction may be due to the fact C-S-H gel production is more in AAS than AASF as AAS has more Calcium rich compounds than AASF mortars. The reduction of Apparent Porosity and Water Absorption may be achieved by controlling extent and temperature of curing of AASF as strong geopolymeric compounds are formed at higher curing temperature. The above properties are measured after curing was done at 65°C for two days and there after normal water curing is done for a total period of 28 days.

4.2.8.2.Compressive Strength and Split Tensile Strength.

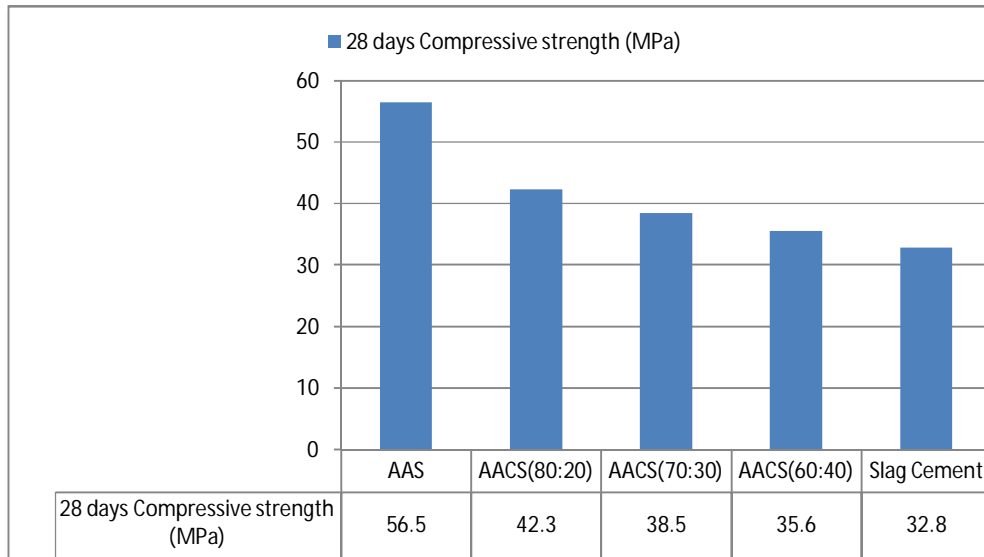


Fig. 4.2.8.2.1(a).
28-days Compressive Strength for different Mortar types

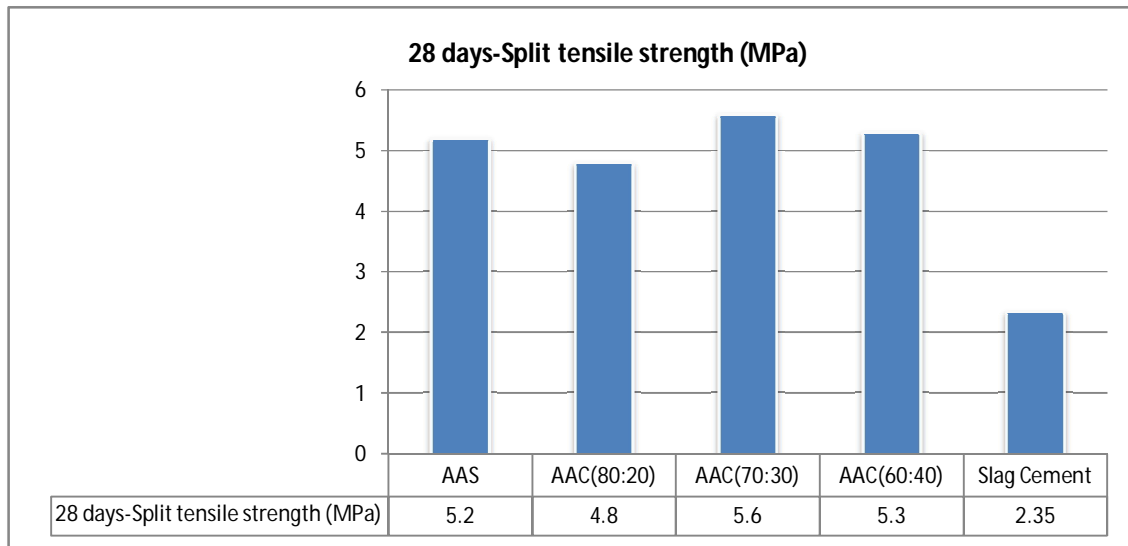


Fig. 4.2.8.2.1(a).
28-days Split Tensile Strength for different Mortar types

The compressive strength of alkali activated mortar was observed to be lesser than AAS mortar but greater than normal slags cement mortar. The C-S-H formed in AAS mortar is more and stronger than the blended variety. However Split tensile strength was observed to be greater this may be due to formation three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds resulting in higher tensile strength.

4.3. Properties of Alkali Activated Mortars specimens Exposed to High Temperature

4.3.1. Loss of Weight.

4.3.1.1. Percentage loss of weight vs. Temperature Levels.

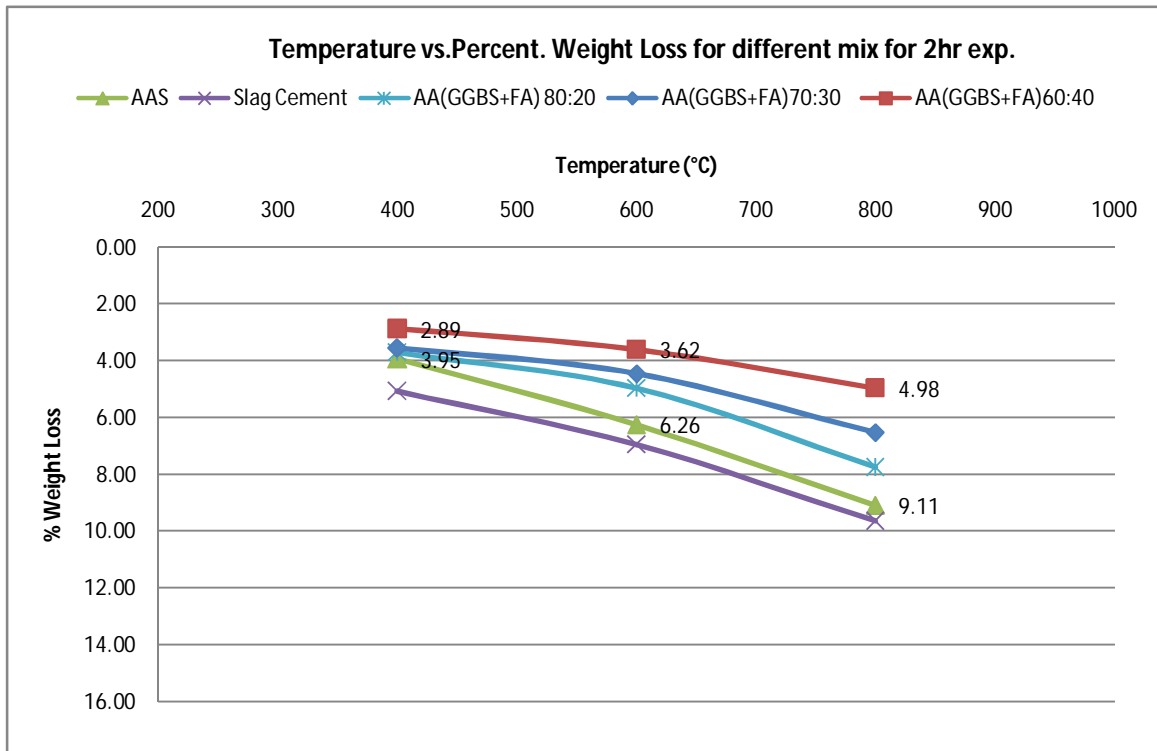


Fig.4.3.1.1.(a) .

Percentage loss of weight vs. Temperature levels (2hrs duration of heating) for different mortar types.

Table 4.3.1.1.(a).

Percentage loss of weight vs. Temperature levels (2hrs duration of heating) for different mortar types

For 2 hr Exposure	% WEIGHT LOSS				
	AAS	Slag Cement	Alkali Activated Composites (GGBS:FA)		
			AA(GGBS+FA) 80:20	AA(GGBS+FA) 70:30	AA(GGBS+FA) 60:40
Temperature (°C)					
400	3.95	5.07	3.72	3.57	2.89
600	6.26	6.96	4.98	4.46	3.62
800	9.11	9.66	7.76	6.55	4.98

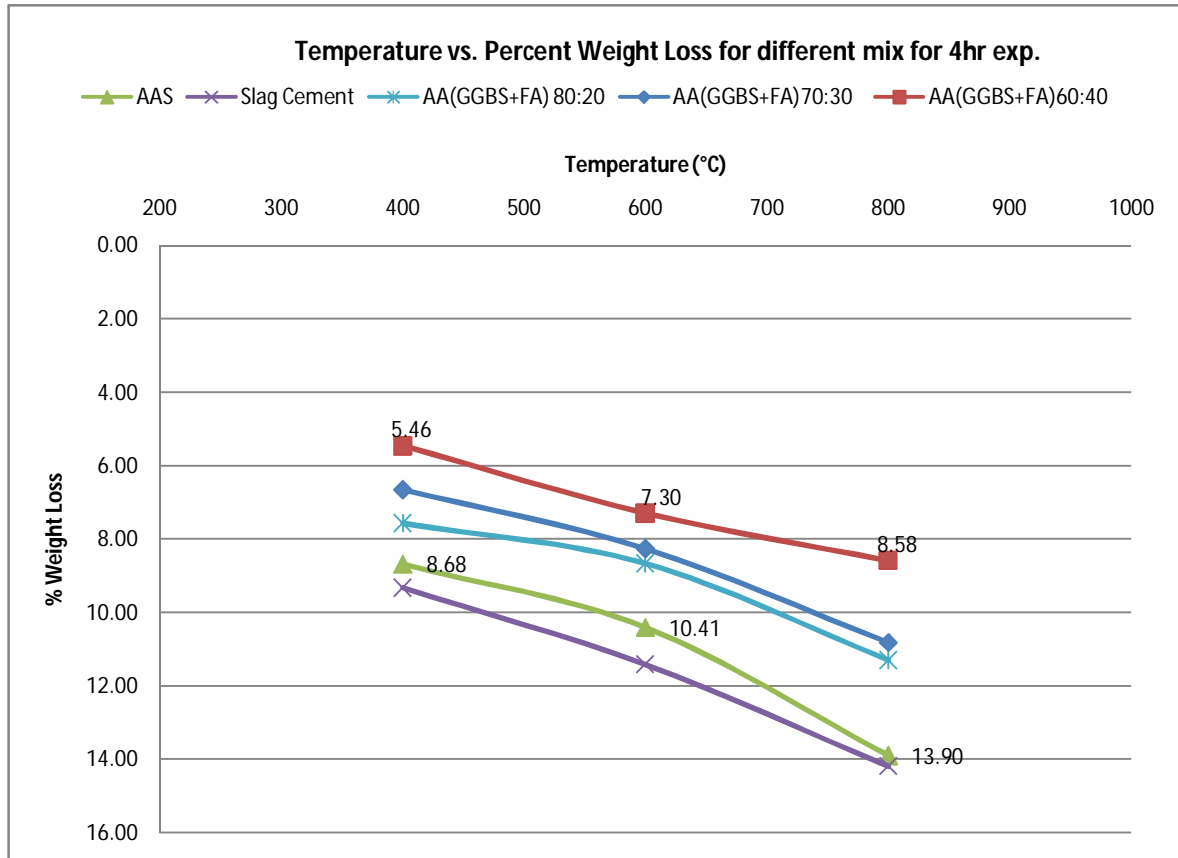


Fig.4.3.1.1(a)

Percentage loss of weight vs. Temperature levels (4hrs duration of heating) for different mortar types

Table 4.3.1.1(b).

Percentage loss of weight vs. Temperature levels (4hrs duration of heating) for different mortar types

For 4 hr Exposure	% WEIGHT LOSS				
	AAS	Slag Cement	Alkali Activated Composites (GGBS:FA)		
			AA(GGBS:FA) 80:20	AA(GGBS:FA) 70:30	AA(GGBS:FA) 60:40
Temperature (°C)					
400	8.68	9.33	7.57	6.66	5.46
600	10.41	11.43	8.67	8.27	7.30
800	13.90	14.19	11.30	10.81	8.58

4.3.1.2. Percentage loss of weight vs. Duration of heating

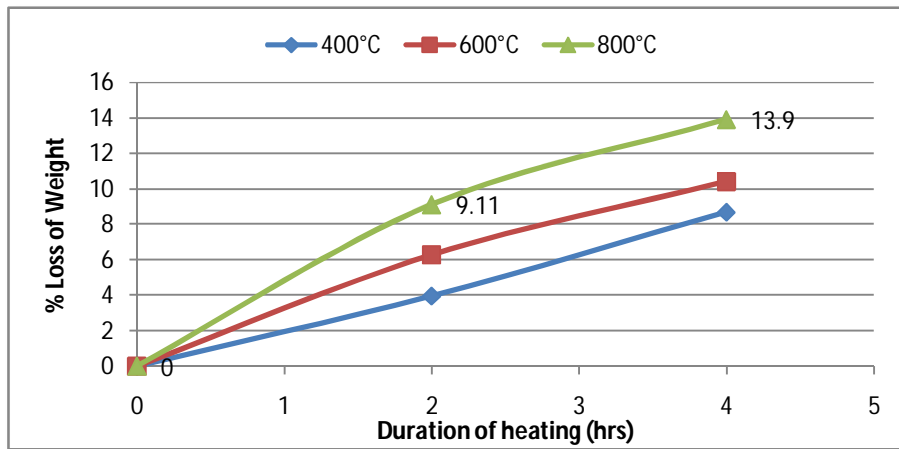


Fig.4.3.1.2 (a). Percentage loss of weight vs. Duration of heating for AAGGBS mortar

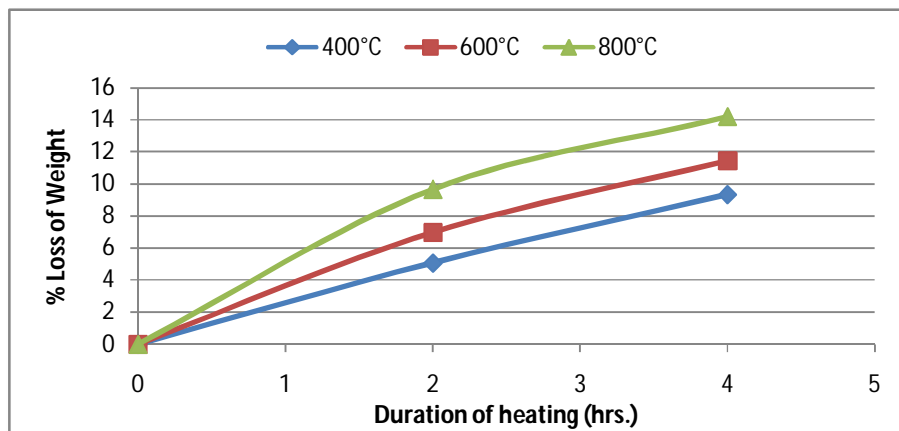


Fig.4.3.1.2 (a). Percentage loss of weight vs. Duration of heating for AASF(Slag:Fly ash=80:20) mortar

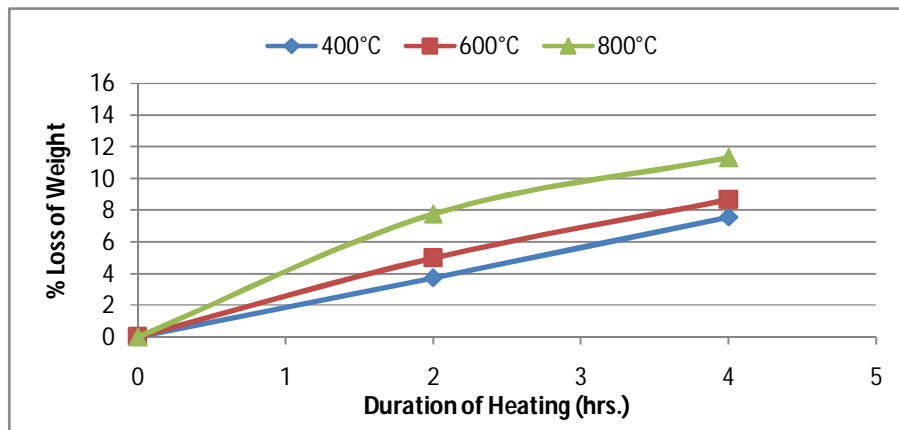


Fig.4.3.1.2 (a).Percentage loss of weight vs. Duration of heating for AASF(Slag:Fly ash=70:30) mortar

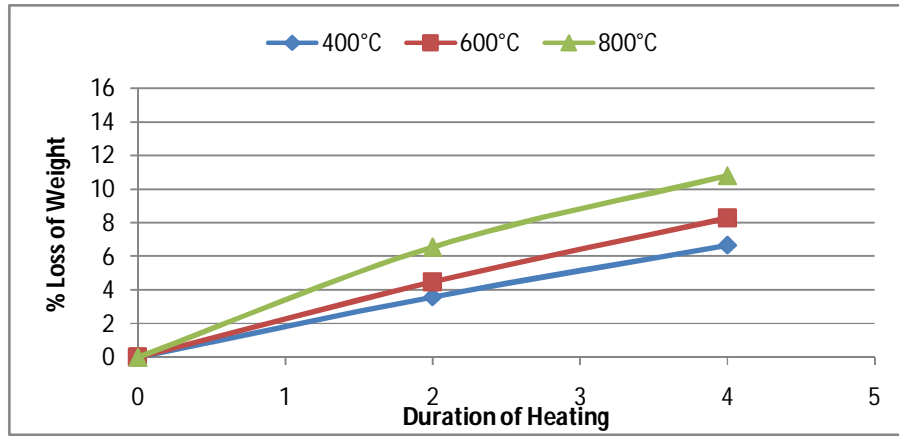


Fig.4.3.1.2 (a).Percentage loss of weight vs. Duration of heating for AASF(Slag:Fly ash=60:40) mortar

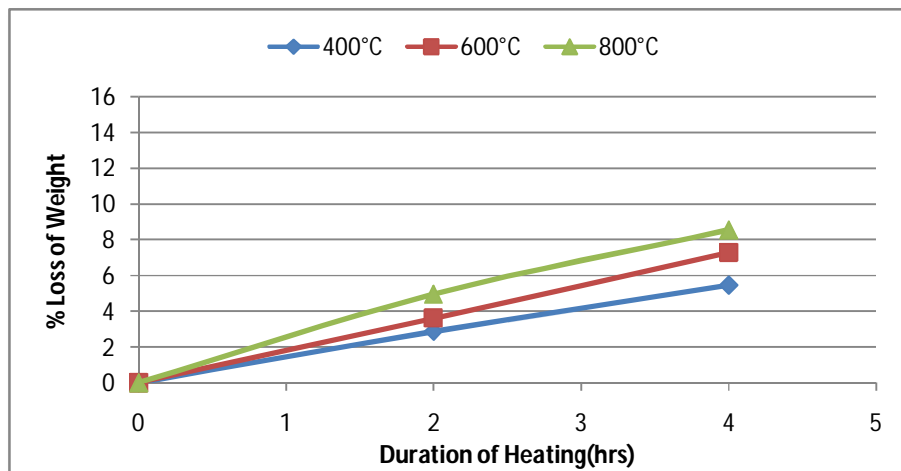


Fig.4.3.1.2 (a).Percentage loss of weight vs. Duration of heating for CSC mortar

Percentage weight loss has been plotted against different temperature levels for 2hrs and 4hrs heating duration . The weight loss for AAS is lesser than conventional slag cement mortar specimens but the rate of weight loss increases at higher temperature levels and duration of heating than the conventional slag cement mortar specimens. So in order to improve the performance of AAS mortars against high temperature we replace GGBS with Fly Ash, and found better result as shown in the figures above. The percentage reduction of weight loss for 600°C to 800°C of 80:20 AAC mix is 35.57 % and 17.53% for 2hr and 4hr exposure respectively as compared with AAS mix it is found to be 45.53 % and 33.52% for 2hr and 4hr exposure respectively.

AAS mortar specimens are CaO rich as compared to AAC(Slag:Fly ash=80:20) mortars, the reaction products are assumed to be more or less similar to Conventional Slag cement Mortar and so similar results are obtained under high temperature exposure. When different proportion of Fly Ash (F-type) have been introduced by replacing GGBS inorganic polymeric materials are formed due to alkali

activation and upon heating sodium aluminosilicate phases are formed with high melting points. Therefore they retain structural integrity at elevated temperature.

4.3.2. Water Absorption:

4.3.2.1. Water Absorption vs. Temperature Levels

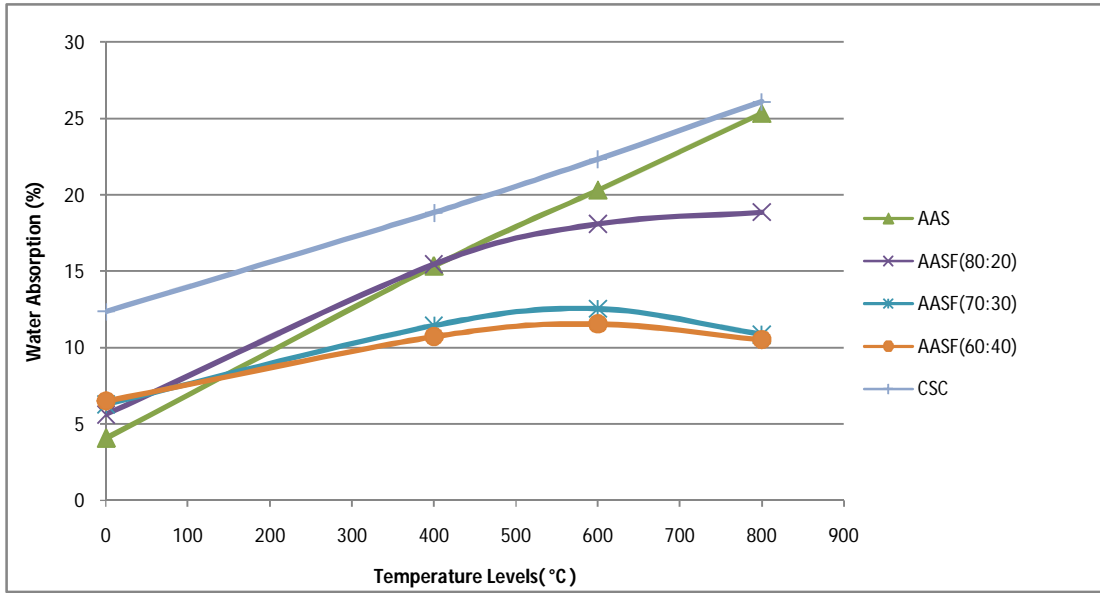


Fig.4.3.2.1 (a).

Water Absorption vs. Temperature Levels (2 hrs Duration of heating) for different types of mortars.

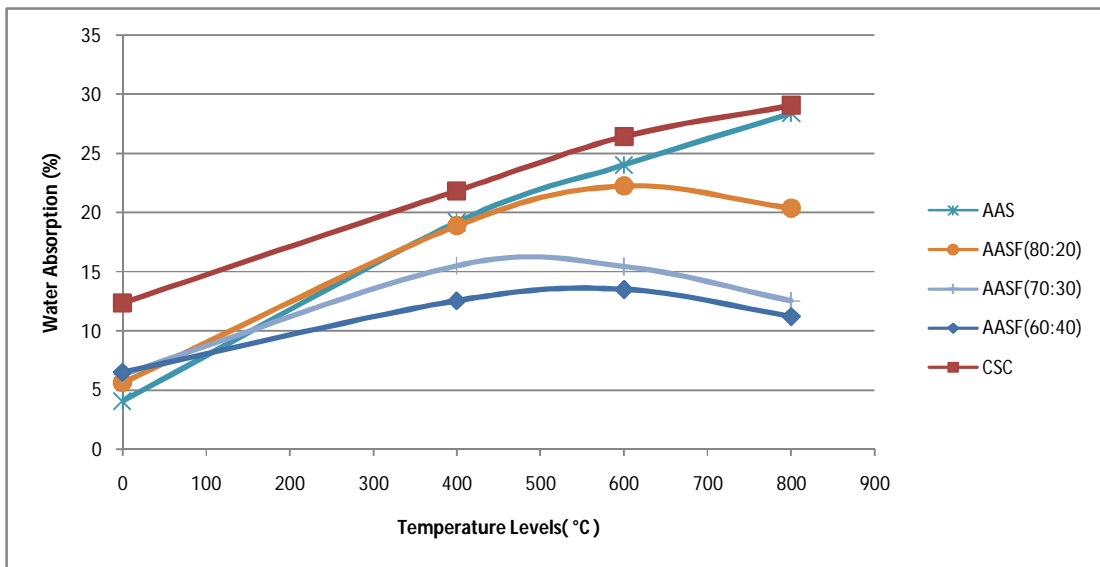


Fig.4.3.2.1(b).

Water Absorption vs. Temperature Levels (4 hrs Duration of heating) for different types of mortars.

Table 4.3.2.1(a)
Water Absorption vs. Temperature Levels (2 hrs Duration of heating) for different types of mortars.

2 hr Exposure	Mortar Types	Water Absorption on exposure to Elevated Temperature Levels (°C)			
		0	400	600	800
	AAS	4.09	15.35	20.32	25.33
	AASF(80:20)	5.63	15.46	18.11	18.88
	AASF(70:30)	6.32	11.45	12.55	10.88
	AASF(60:40)	6.52	10.72	11.55	10.52
	CSC	12.38	18.85	22.35	26.1

Table 4.3.2.1(b)
Water Absorption vs. Temperature Levels (4 hrs Duration of heating) for different types of mortars

4 hr Exposure	Mortar Types	Water Absorption on exposure to Elevated Temperature Levels (°C)			
		0	400	600	800
	AAS	4.09	19.20	24.05	28.41
	AASF(80:20)	5.63	18.91	22.27	20.40
	AASF(70:30)	6.32	15.50	15.44	12.55
	AASF(60:40)	6.52	12.56	13.53	11.23
	CSC	12.38	21.87	26.44	29.08

4.3.2.2. Water Absorption vs. Duration of Heating.

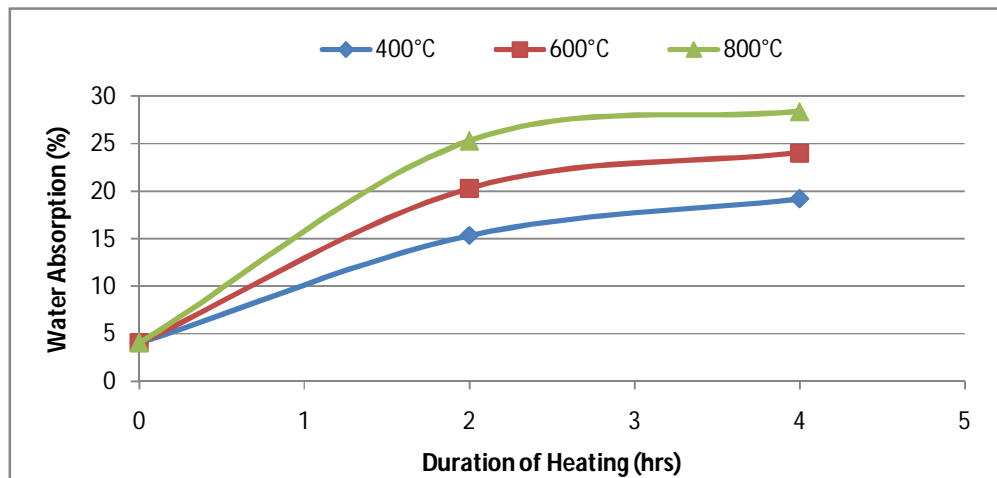


Fig. 4.3.2.2 (a).
Water Absorption vs. Duration of heating for AAGGBS mortar

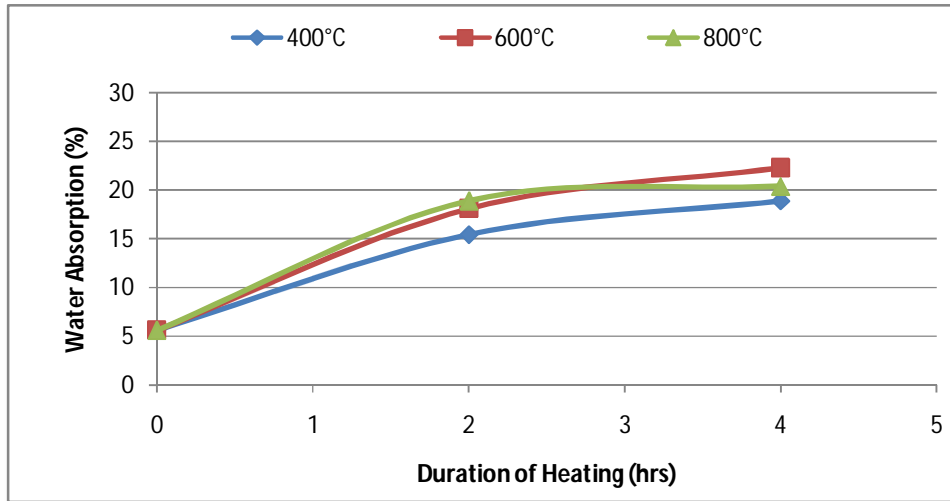


Fig. 4.3.2.2 (b).

Water Absorption vs. Duration of heating for AASF(Slag: Fly Ash=80:20) mortar

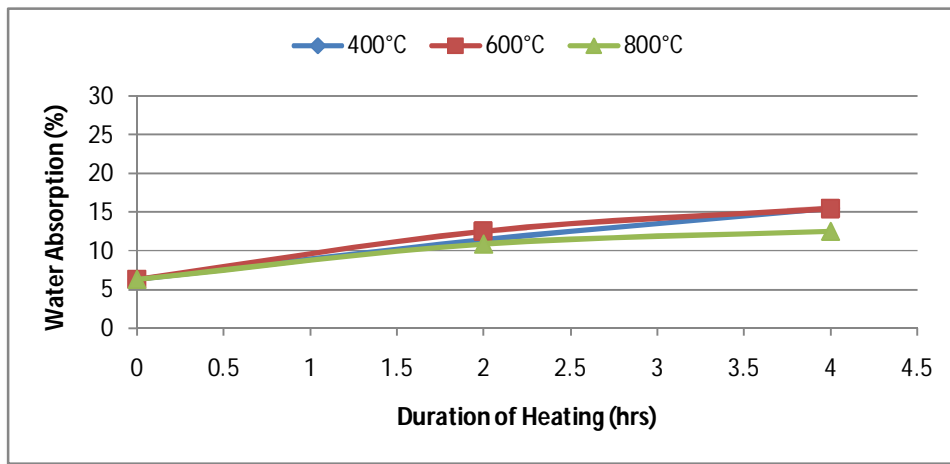


Fig. 4.3.2.2(c)

Water Absorption vs. Duration of heating for AASF(Slag:Fly ash=70:30) mortar

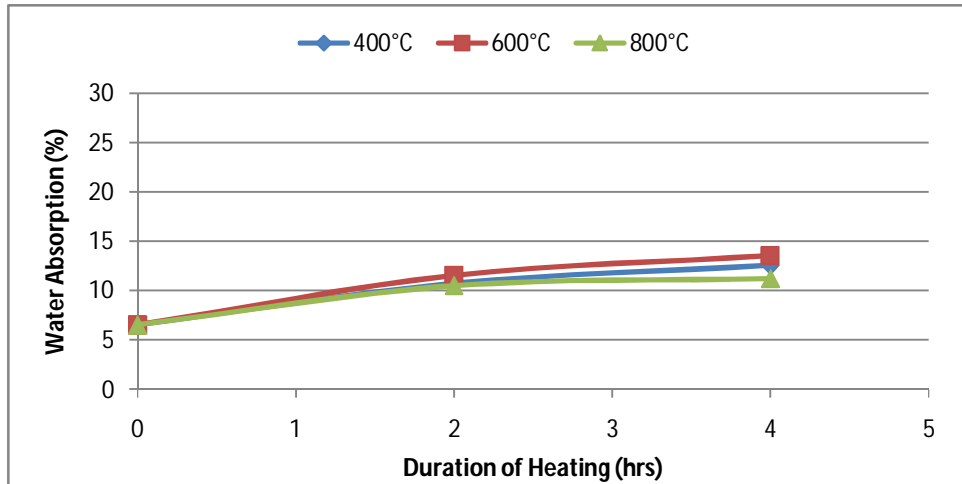


Fig. 4.3.2.2 (d)
Water Absorption vs. Duration of heating for AASF(Slag:Fly ash =60:40) mortar

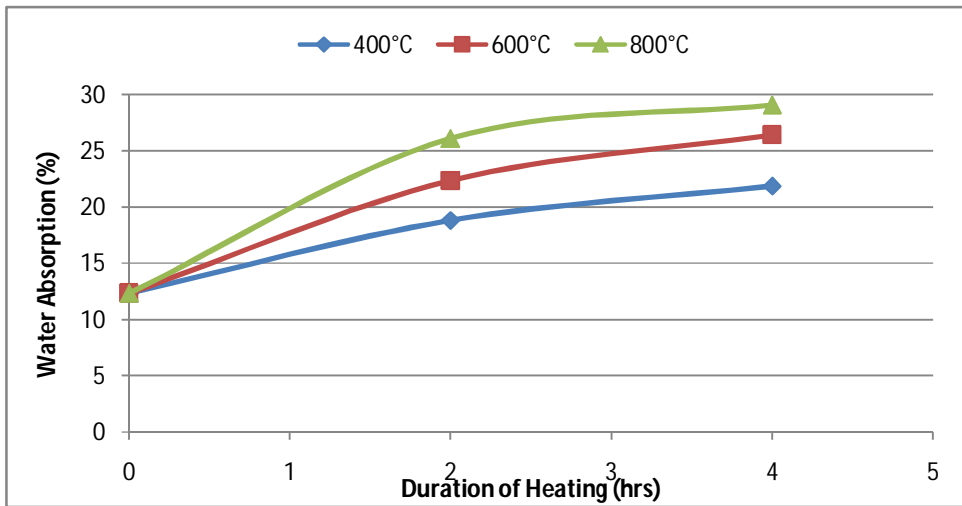


Fig. 4.3.2.2 (e)
Water Absorption vs. Duration of heating for CSC mortar

From the above tables and figures has been observed that there was rapid increase of apparent porosity initially for all types of mortar. The rate of increase is more for AAS and SC mortar. The rate is almost linear upto 600°C for AAS and CSC. The rate slightly decreases from 600°C to 800°C. However for AASF mortar specimens a decrease of apparent porosity has been observed at higher temperature when the proportion of Fly Ash was increased. Higher rate of decrease has been found when the duration of heating was increased at higher temperature.

Dehydration/dehydroxylation of the main hydration products calcium silicate hydrate (C-S-H) occurs below 300 °C and portlandite at temperatures between 400 °C and 500 °C [50]. This leads to shrinkage

of the mortar specimens, and its consequent cracking and spalling with the progressive decomposition of these compounds which causes increase of apparent porosity.

The microstructure of alkali activated slag gets changed between 600 and 800 °C, when the dehydration of C-A-S-H phase was completed and new phases such as akermanite[51] started to crystallize which causes a slight decrease in rate of increase of apparent porosity

For AASF verity when silica rich fly ash was increased highly crosslinked sodium aluminosilicate are formed which are more stable at high temperature and causes a decrease of apparent porosity at higher temperature.

4.3.3. Apparent Porosity

4.3.3.1.Apparent Porosity vs. Temperature Levels

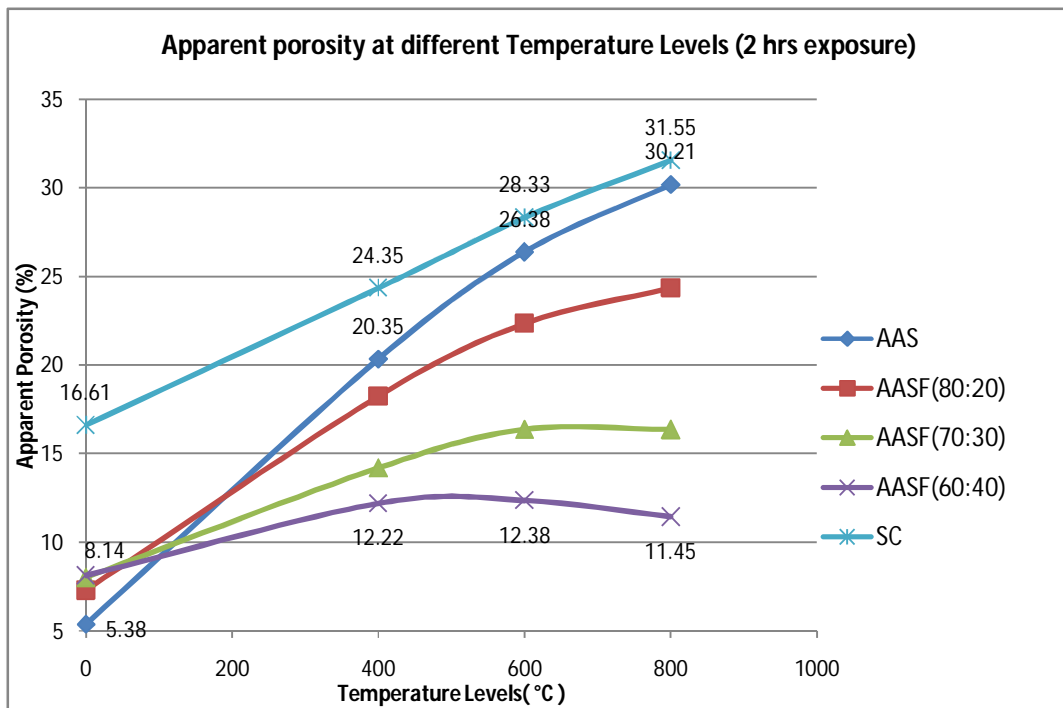


Fig.4.3.3.1(a).
Apparent porosity vs. Temperature Levels (2 hrs duration of heating) for different types of mortars.

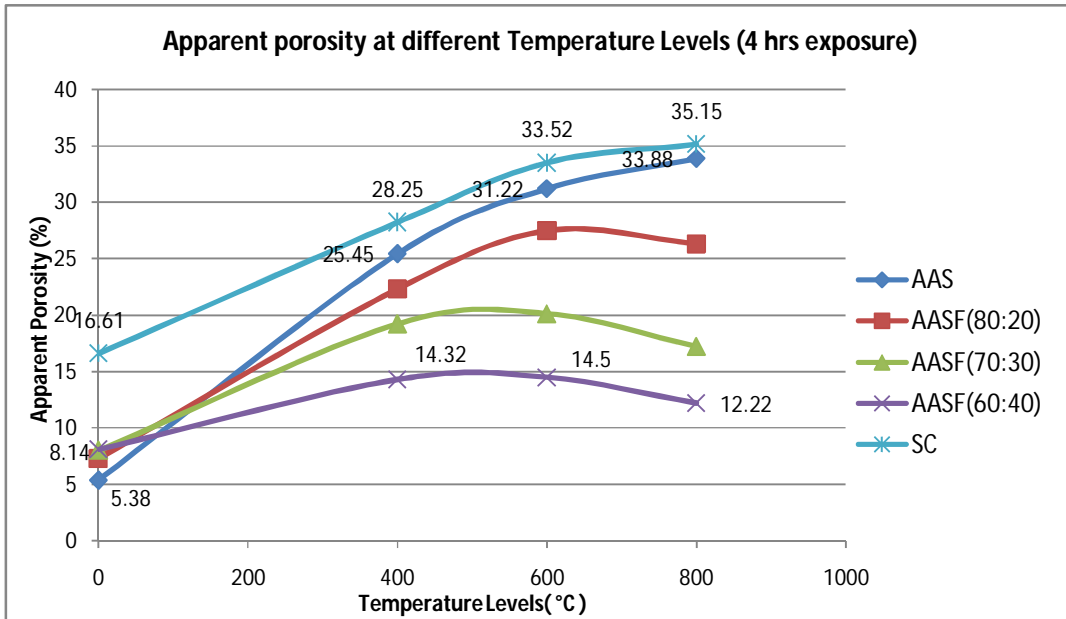


Fig.4.3.3.1(b).

Apparent porosity vs. Temperature Levels (4 hrs duration of heating) for different types of mortars.

Table 4.3.3.1(a):

Apparent porosity vs. Temperature Levels (2 hrs duration of heating) for different types of mortars

2 hr Exposure	Mortar Types	Apparent Porosity at Elevated Temperature Levels (%)			
		Unexposed	400°C	600°C	800°C
	AAS	5.38	20.35	26.38	30.21
	AASF(80:20)	7.31	18.25	22.35	24.37
	AASF(70:30)	8.02	14.22	16.38	16.37
	AASF(60:40)	8.14	12.22	12.38	11.45
	SC	16.61	24.35	28.33	31.55

Table4.3.3.1(b) :

Apparent porosity vs. Temperature Levels (4 hrs duration of heating) for different types of mortars

4 hr Exposure	Mortar Types	Apparent Porosity at Elevated Temperature Levels (%)			
		Unexposed	400°C	600°C	800°C
	AAS	5.38	25.45	31.22	33.88
	AASF(80:20)	7.31	22.32	27.48	26.33
	AASF(70:30)	8.02	19.25	20.15	17.25
	AASF(60:40)	8.14	14.32	14.5	12.22
	SC	16.61	28.25	33.52	35.15

4.3.3.2.Apparent Porosity vs. Duration of Heating

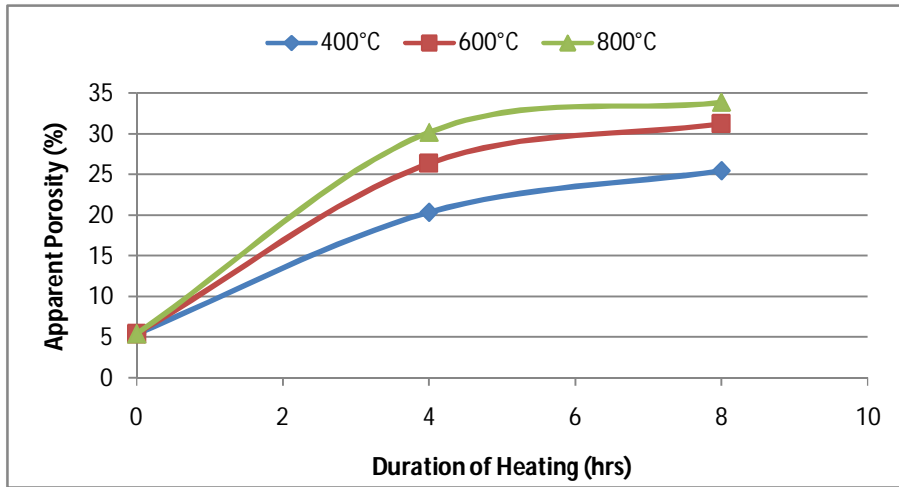


Fig. 4.3.3.2(a). Apparent Porosity vs. Duration of heating for AAGBS mortar

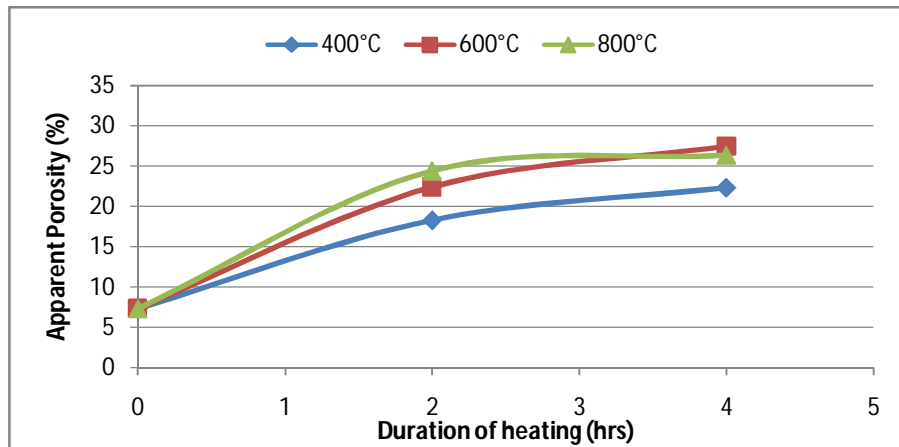


Fig. 4.3.3.2(b).Apparent Porosity vs. Duration of heating for AASF (Slag:FlyAsh=80:20) mortar

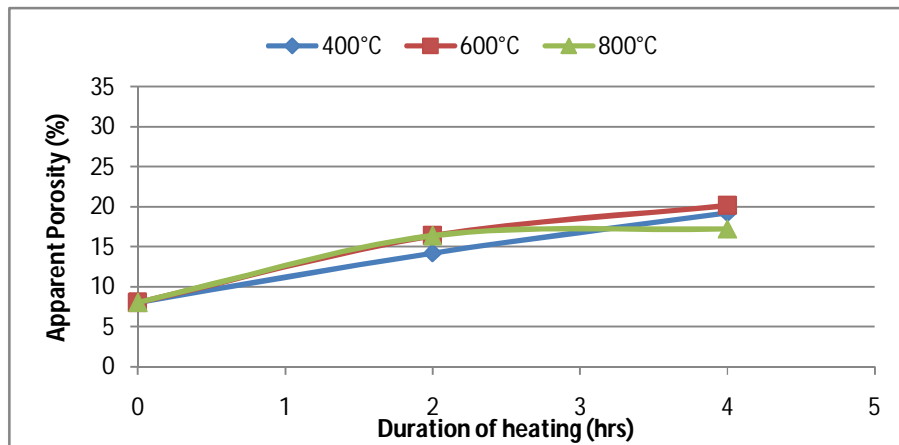


Fig. 4.3.3.2(c). Apparent Porosity vs. Duration of heating for AASF (Slag:FlyAsh=70:30) mortar

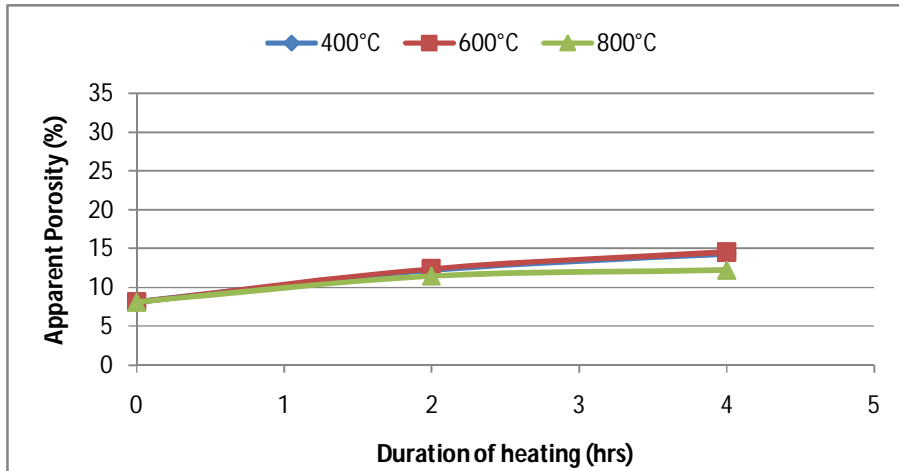


Fig. 4.3.3.2(d). Apparent Porosity vs. Duration of heating for AASF (Slag:FlyAsh=60:40) mortar

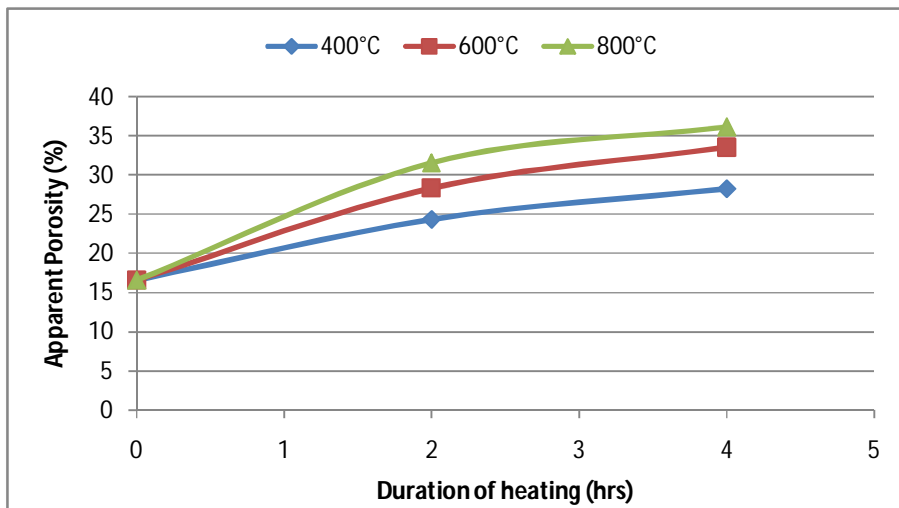


Fig. 4.3.3.2(e). Apparent Porosity vs. Duration of heating for CSC mortar

4.3.4. Sorptivity

4.3.4.1. Sorptivity vs. Temperature Levels

Table.4.3.4.1. Sorptivity vs. Temperature Levels (2 hrs duration of heating) for different types of mortars

2 HR. Exp.	Exp. Temp.(°C)	Sorptivity(gm/mm ² /s ^{0.5})				
		1.AAS	2.AASF(80:20)	3.AASF(70:30)	4.AASF(60:40)	5.SC
	0	3.61E-05	9.52E-05	9.64E-05	9.88E-05	1.09E-04
	400	6.84E-04	8.78E-04	8.05E-04	8.55E-04	8.55E-04
	600	8.31E-04	9.95E-04	9.98E-04	9.25E-04	9.85E-04
	800	9.75E-04	9.85E-04	9.88E-04	8.92E-04	1.01E-03

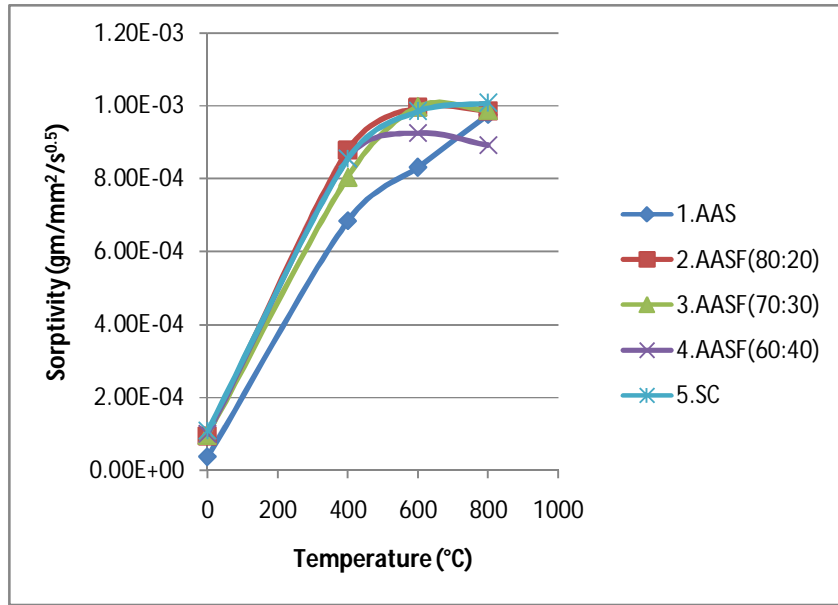


Fig.4.3.4.1(a). Sorptivity vs. Temperature Levels (2 hrs duration of heating) for different types of mortars

Table.4.3.4.1. Sorptivity vs. Temperature Levels (4 hrs duration of heating) for different types of mortars

Exp. Temp.(°C)	Sorptivity(gm/mm ² /s ^{0.5})				
	1.AAS	2.AASF(80:20)	3.AASF(70:30)	4.AASF(60:40)	5.SC
0	3.61E-05	9.52E-05	9.64E-05	9.88E-05	1.09E-04
400	1.03E-03	1.51E-03	1.75E-03	1.83E-03	1.53E-03
600	1.26E-03	1.85E-03	1.86E-03	1.81E-03	1.87E-03
800	1.84E-03	1.91E-03	1.84E-03	1.51E-03	1.94E-03

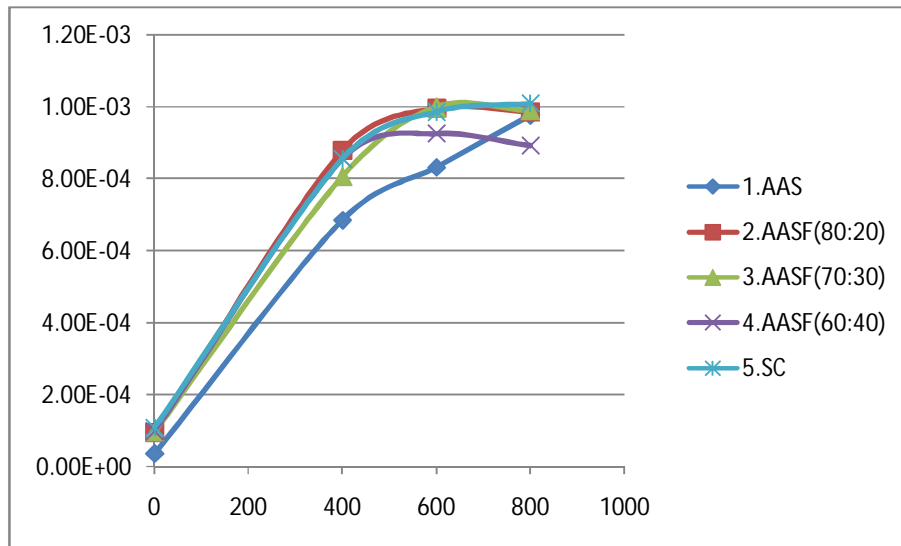


Fig.4.3.4.1(b). Sorptivity vs. Temperature Levels (4 hrs duration of heating) for different types of mortars

4.3.4.2. Sorptivity vs. Duration of Heating

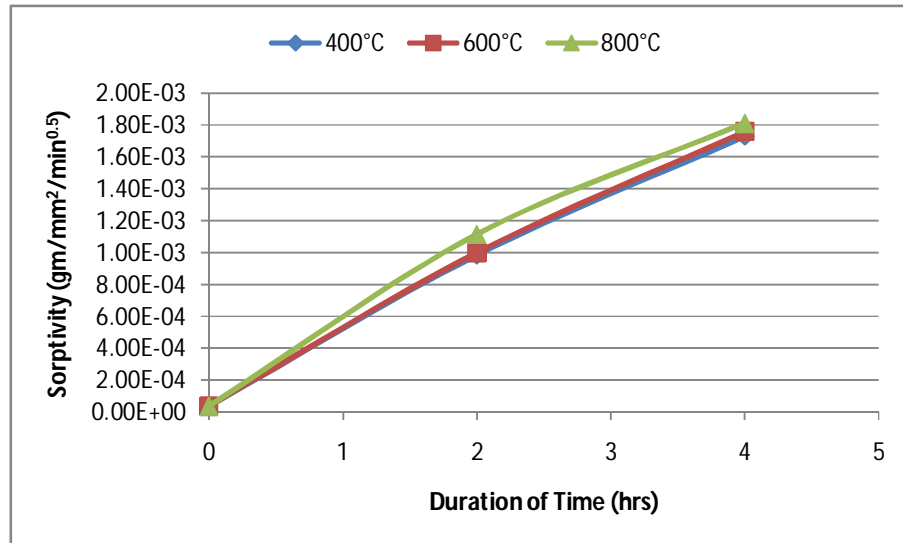


Fig.4.3.4.2(a)
Sorptivity vs. Duration of heating for AAGGBS mortar

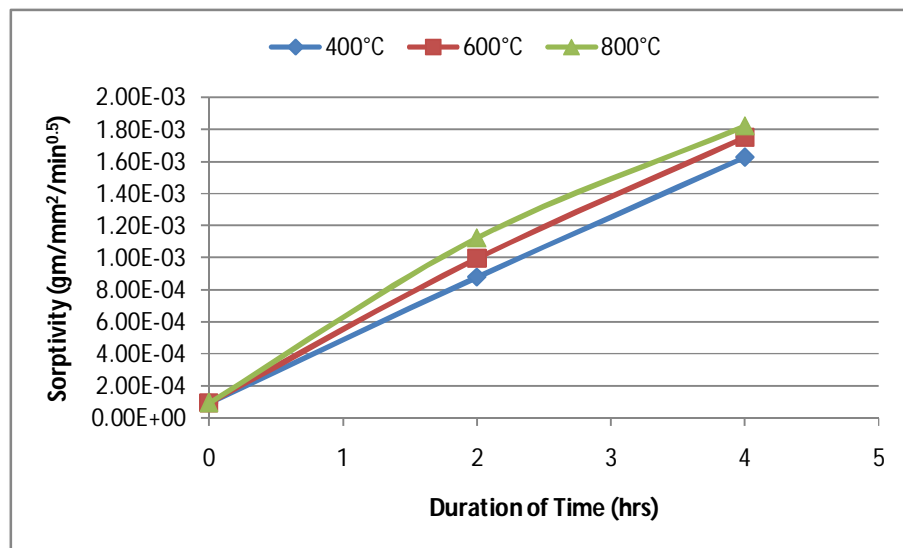


Fig.4.3.4.2(b)
Sorptivity vs. Duration of heating for AASF(Slag:Fly ash=80:20) mortar

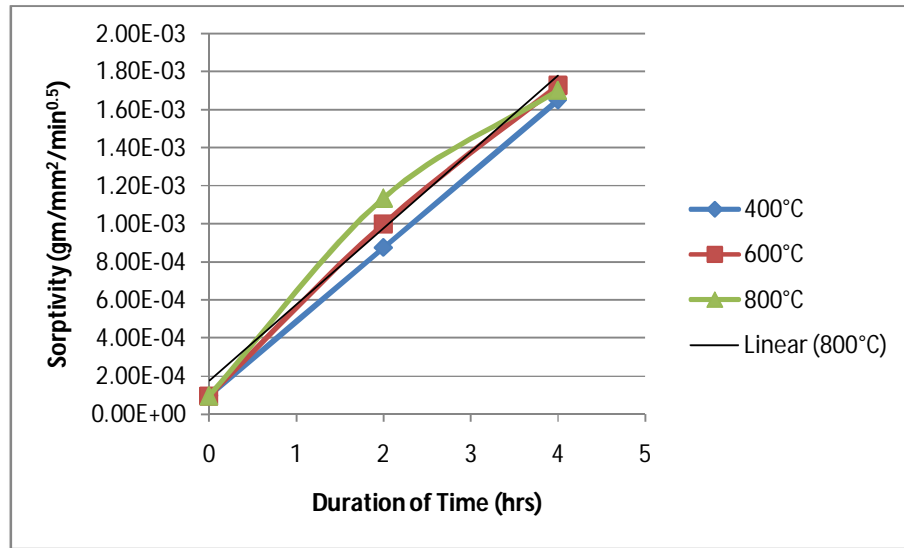


Fig.4.3.4.2(c).
Sorptivity vs. Duration of heating for AASF(Slag:Fly ash=70:30) mortar

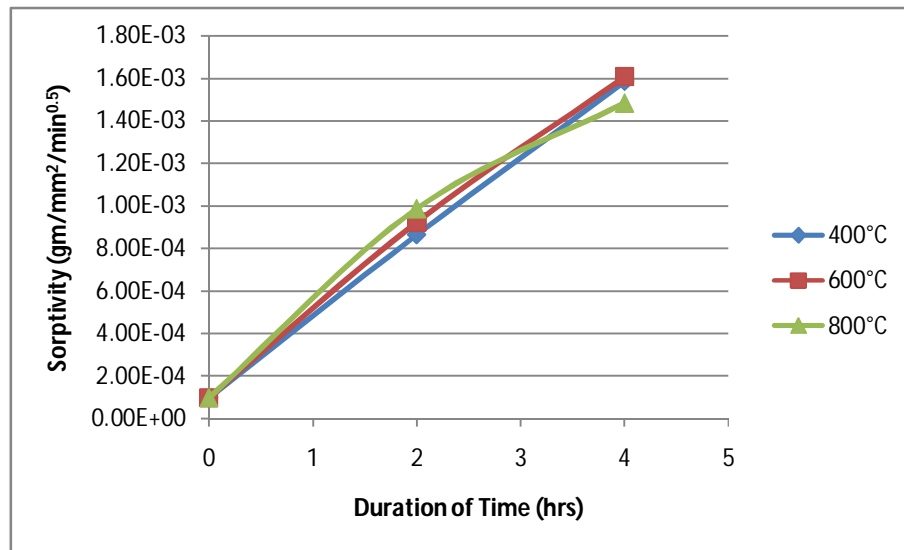


Fig.4.3.4.2(b)
Sorptivity vs. Duration of heating for AASF(Slag:Fly ash=60:40) mortar

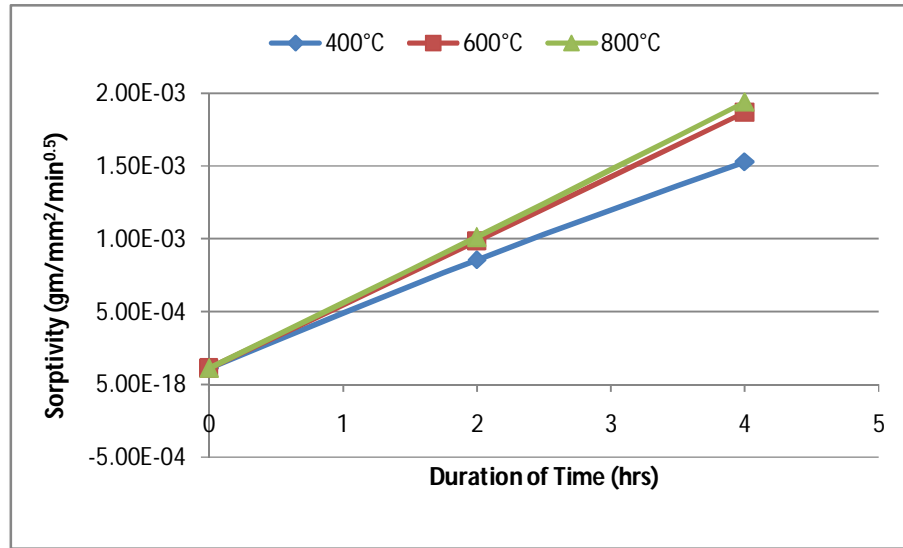


Fig.4.3.4.2(b)
Sorptivity vs. Duration of Time for CSC mortar

It has been observed that for AAS mortar specimens, as temperature increases sorptivity increases initially at faster rate upto 400°C. Then the increment is relatively slower in between 400°C and 600°C, thereafter i.e. above 600°C Sorptivity again increases at faster rate. This is due to the fact that as temperature increases the sand particles expand and the bond between the C-S-H paste loosens and cracks occur thereby increasing water absorption. At higher temperature C-S-H gets completely disintegrated. The normal slag cement mortar also behaves in a similar fashion.

When Alkali Activated GGBS is partially replaced by Fly Ash and exposed to high temperature it has been observed that the blended verity gives lower Sorptivity value at higher temperatures due to the formation of sodium alumino-silicate phases: albite, carnegieite and nepheline[52] which melt at higher temperature. Thus blended AAC gives better performance when exposed to high temperature.

4.3.5. Compressive Strength.

4.3.5.1. Compressive strength vs. Temperature Levels.

Table 4.3.5.1.
Compressive Strength vs. Temperature Levels (2hrs & 4hrs duration of heating) for different mortar types

TEMPERATURE (°C)	28 days Compressive strength (Mpa)									
	AAS		AAC(80:20)		AAC(70:30)		AAC(60:40)		Slag Cement(CSC)	
	Exposure									
	2hr	4hr	2hr	4hr	2hr	4hr	2hr	4hr	2hr	4hr
Unexposed Temp	56.5		42.3		38.5		35.6		32.8	
400	22.5	8.5	25.4	12.2	22	10.2	25.6	8.2	15.5	7.2
600	10	0*	12.2	4.2	12	6.2	13.2	5.5	3.6	0.4
800	2	0*	5.5	0*	4	2.4	5.4	3.2	0*	0*
	*could not be determined									

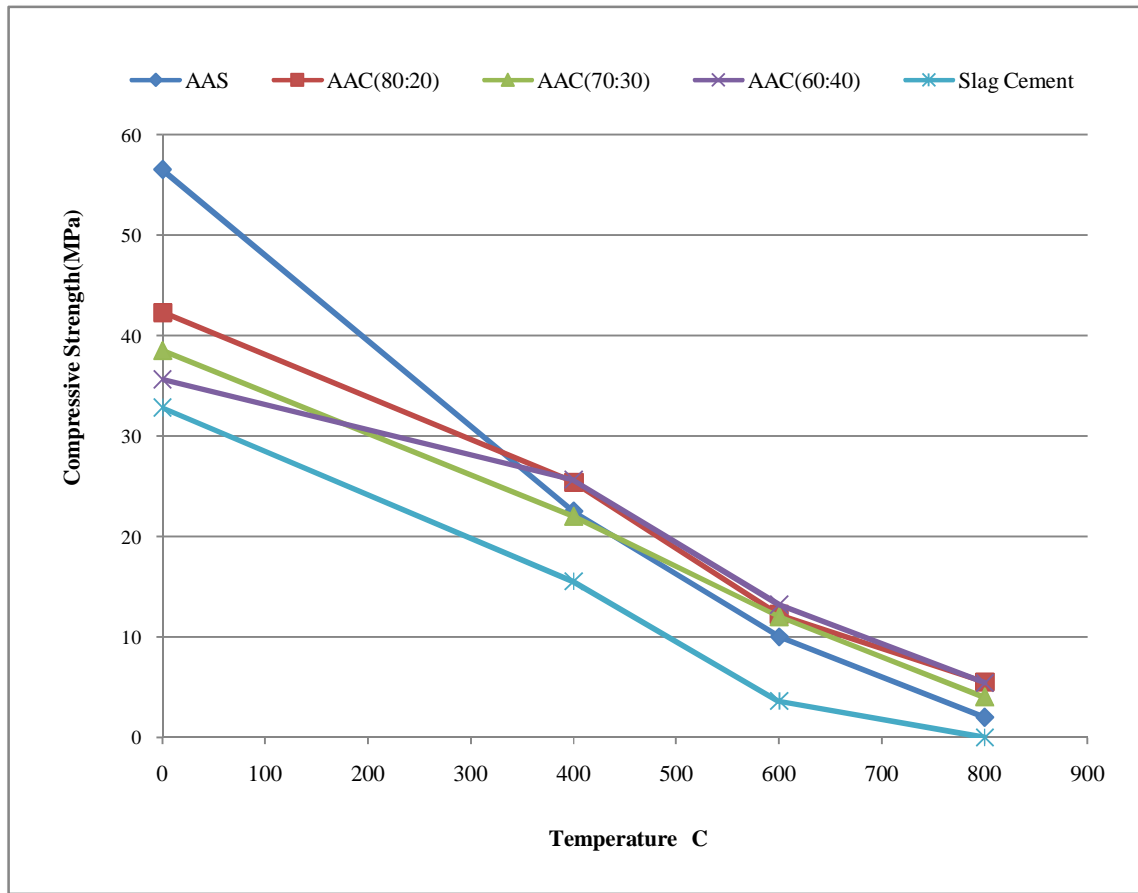


Fig. 4.3.5.1(a).
Compressive Strength vs. Temperature Levels (2hrs duration of heating) for different mortar types.

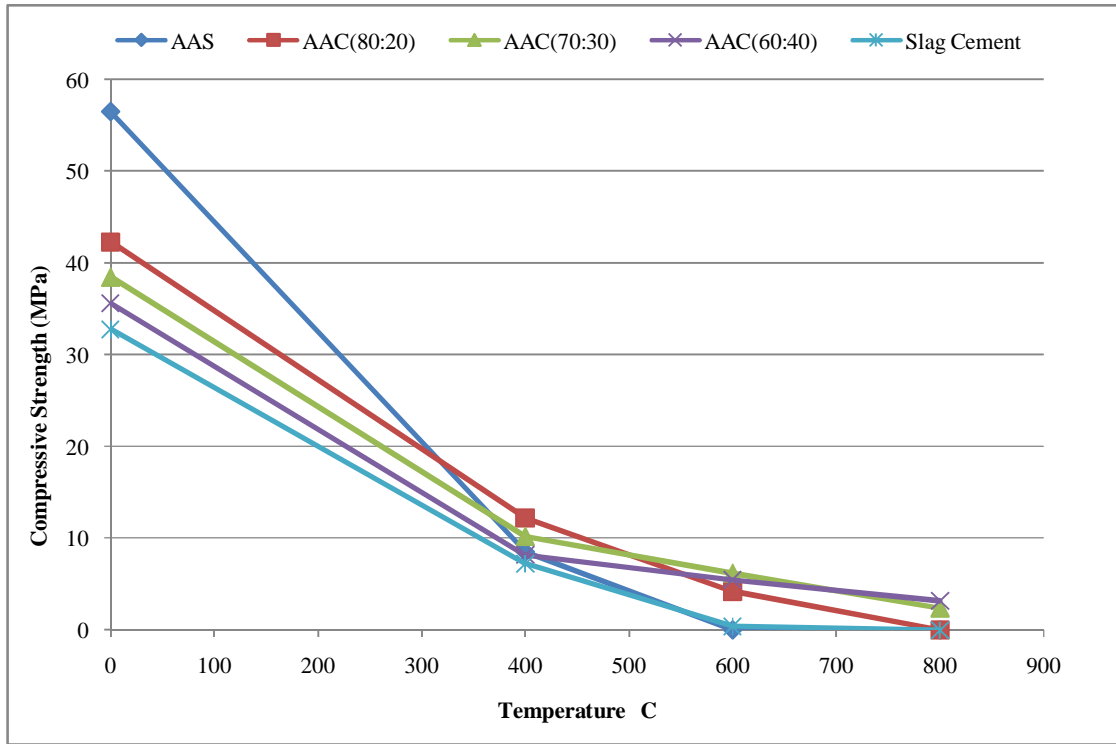


Fig. 4.3.5.1(b)

Compressive Strength vs. Temperature Levels (4hrs duration of heating) for different mortar types

4.3.5.2. Compressive Strength vs. Duration of Heating.

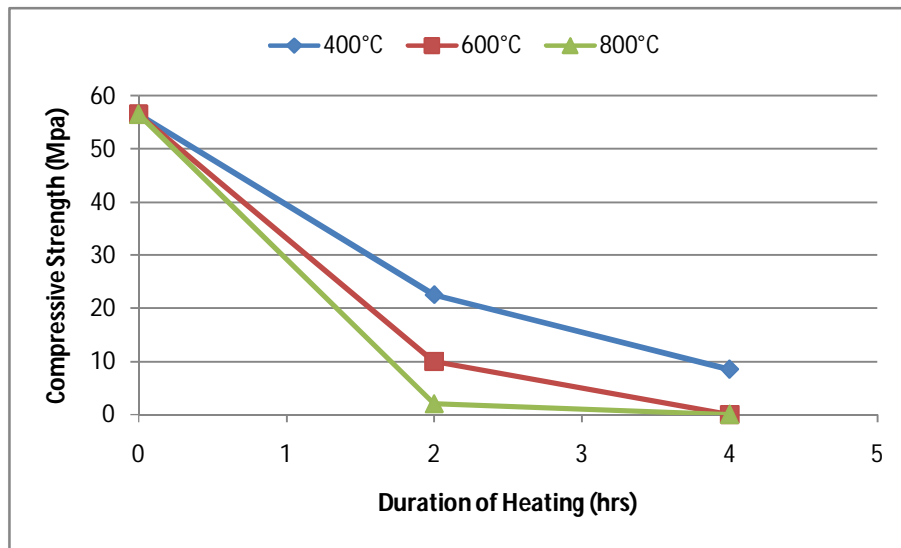


Fig. 4.3.5.2(a)

Compressive Strength vs. Duration of heating for AAGBS mortar

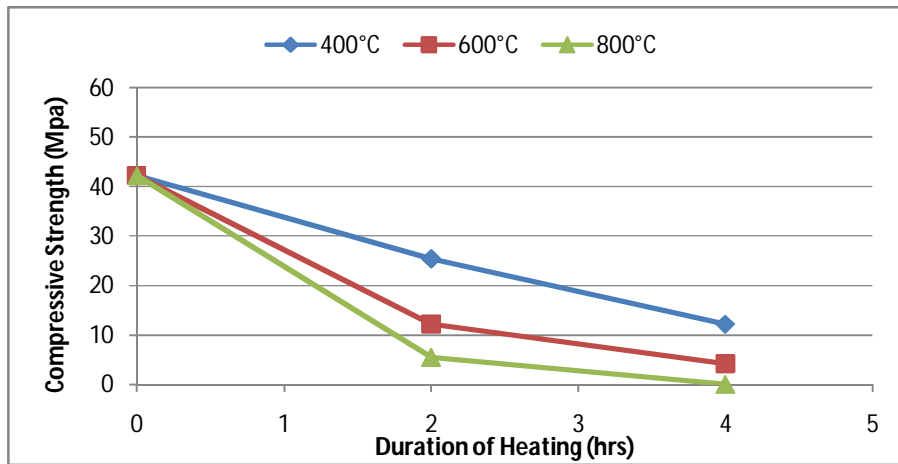


Fig. 4.3.5.2(b). Compressive Strength vs. Duration of heating. for AASF(Slag:Fly ash=80:20) mortar

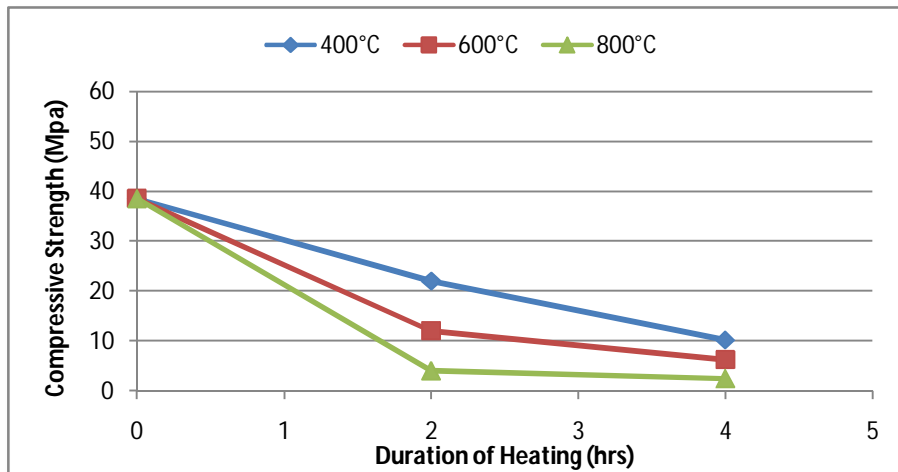


Fig. 4.3.5.2(c). Compressive Strength vs. Duration of heating. for AASF(Slag:Fly ash=70:30) mortar

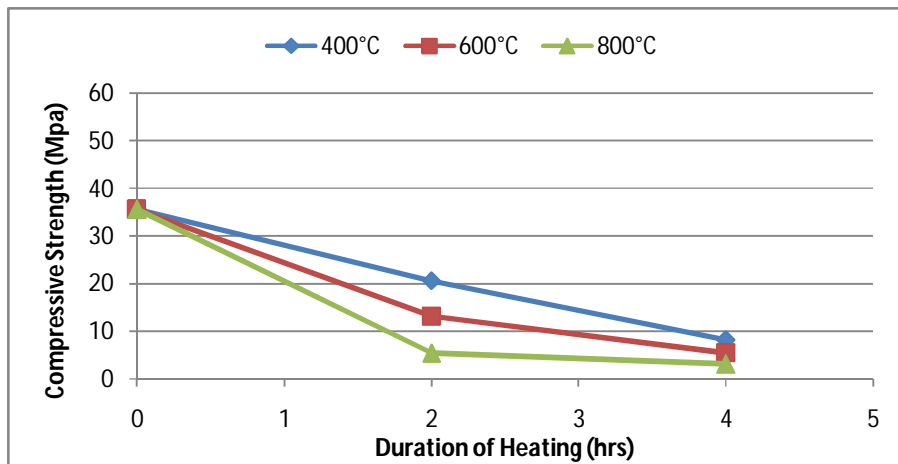


Fig. 4.3.5.2(d). Compressive Strength vs. Duration of heating. for AASF(Slag:Fly ash=60:40) mortar

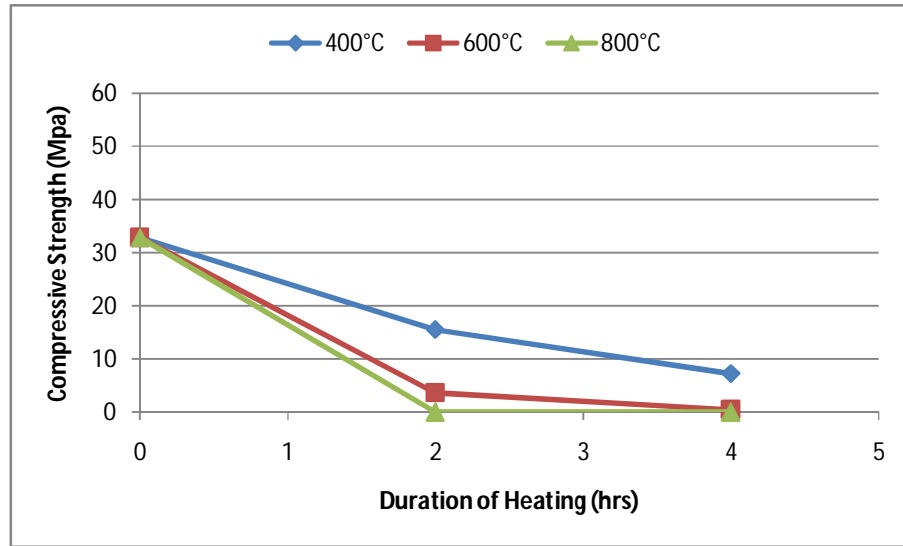


Fig. 4.3.5.2(e).

Compressive Strength vs. Duration of heating. for CSC mortar

Residual Compressive strength for AAS on exposure to elevated temperature was very low and similar results were observed for specimens out of conventional slag cement mortar. It may be said that the hydration product and heating effect and reaction products are similar in both the cases. But when GGBS is replaced by Fly Ash polymeric products of sodium aluminosilicates are formed with high melting points giving higher residual strength as compared with AAS and Slag Cement mix. A 8.98 % increase in residual compressive strength was observed for AASF(Slag:Fly ash=60:40) over AAGGBS mortar specimens when exposed to 800°C for a duration of 4hrs which may be due to the formation of products with ceramic like properties.

4.3.6. Split tensile strength

4.3.6.1. Split Tensile Strength vs. Temperature Levels.

Table 4.3.6.1. Split Tensile Strength vs. Temperature Levels (2hrs & 4hrs duration of heating) for different mortar types

Temperature (°C)	28 days-Split tensile strength (Mpa)									
	AAS		AAC(80:20)		AAC(70:30)		AAC(60:40)		Slag Cement	
	Exposure									
	2hr	4hr	2hr	4hr	2hr	4hr	2hr	4hr	2hr	4hr
Unexposed Temp.	5.2		4.8		5.6		5.3		2.35	
400	2.1	0.25	2.4	1.5	2.8	1.8	2.2	1.02	1.85	1.12
600	1.2	0.12	1.65	0.8	1.8	1.1	1.35	0.55	0.15	0.05
800	0.15	0*	1.1	0*	1.25	0.25	0.8	0*	0*	0*
	*could not be determined									

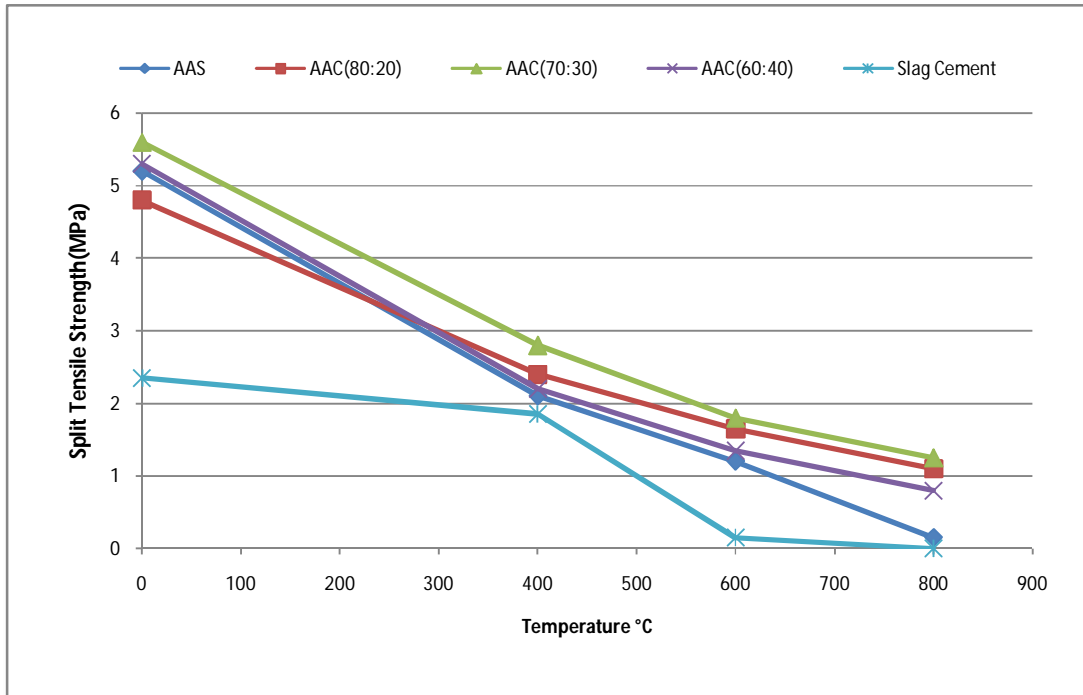


Fig. 4.3.6.1(a)

Split Tensile Strength vs. Temperature Levels (2hrs duration of heating) for different mortar types.

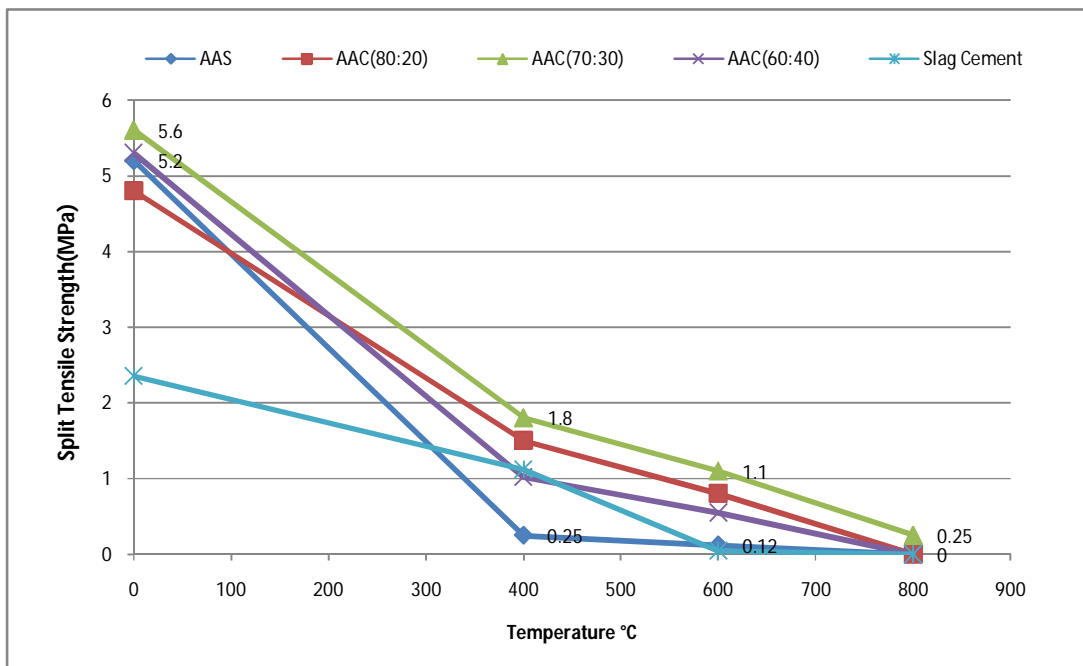


Fig. 4.3.6.1(b).

Split Tensile Strength vs. Temperature Levels (4hr duration of heating) for different mortar types

4.3.6.2. Split Tensile Strength vs. Duration of heating.

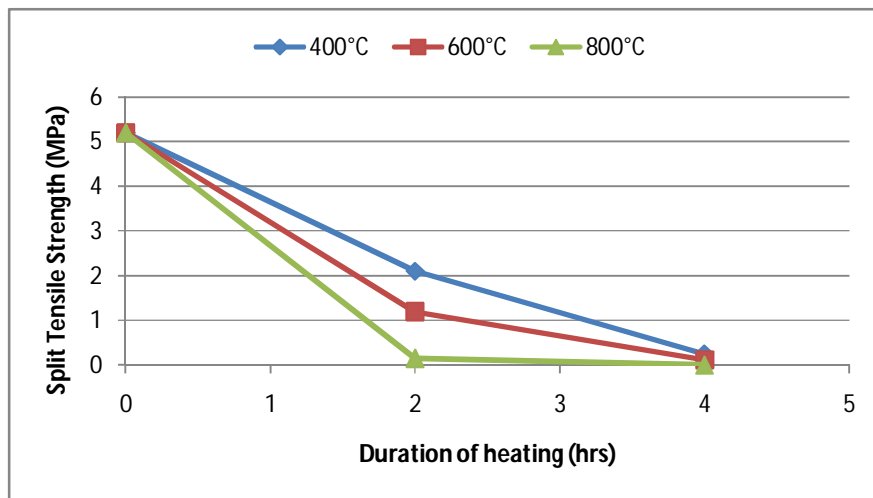


Fig. 4.3.6.2(a)

Split Tensile Strength vs. Duration of heating for AAGGBS mortar.

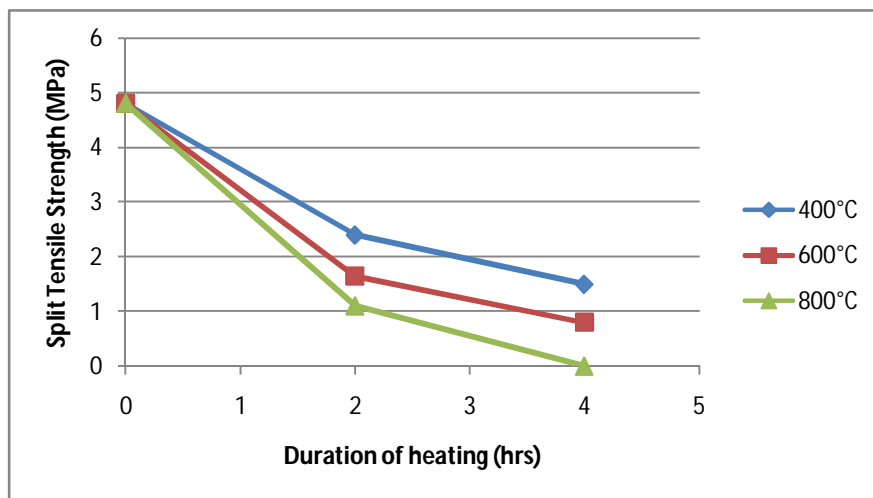


Fig. 4.3.6.2(b)

Split Tensile Strength vs. Duration of heating for AASF(Slag:Fly ash=80:20) mortar

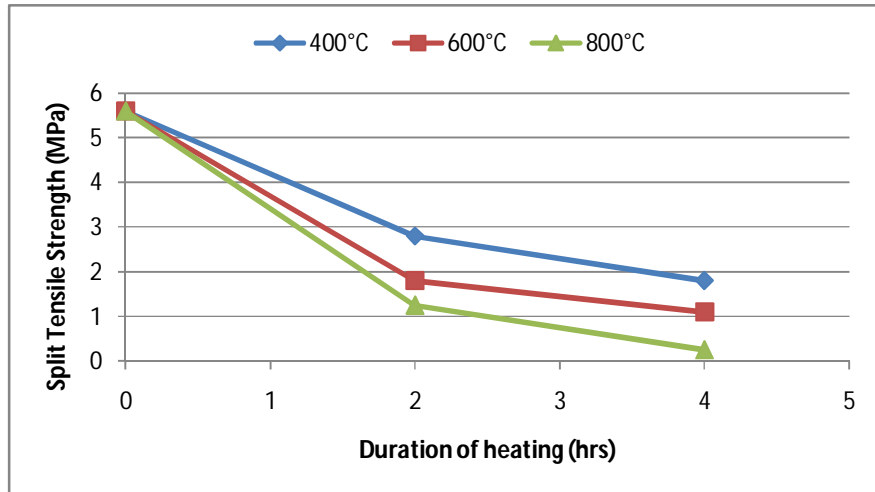


Fig. 4.3.6.2(c). Split Tensile Strength vs. Duration of heating for AASF(Slag:Fly ash =70:30) mortar

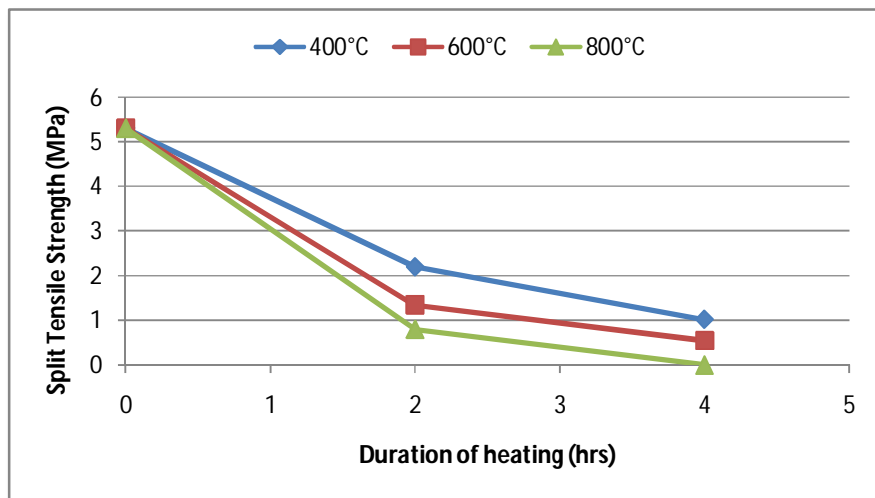


Fig. 4.3.6.2(d) Split Tensile Strength vs. Duration of heating for AASF(Slag:Fly ash=60:40) mortar

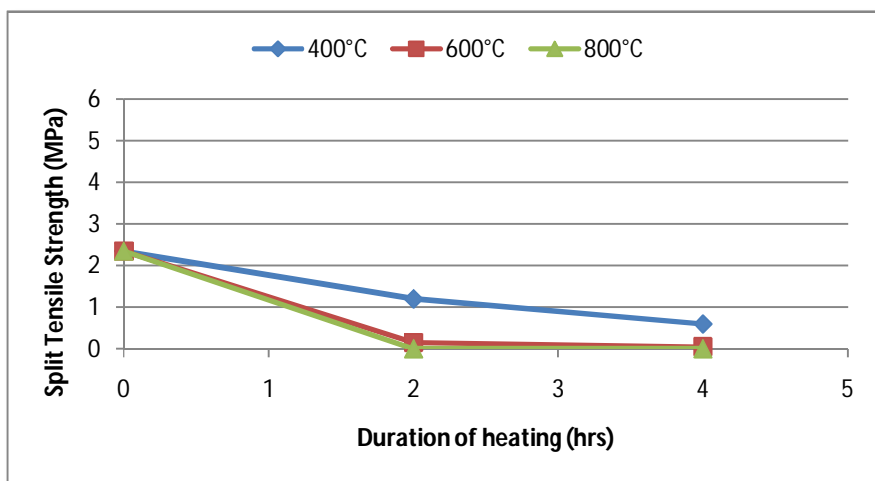


Fig. 4.3.6.2(e) Split Tensile Strength vs. Duration of heating for CSC mortar

Similar to residual compressive strength split tensile strength on exposure to elevated temperature shows rapid reduction of strength at elevated temperature. Mortar specimens of GGBS mixed with Fly ash(AASF) shows less reduction of split tensile strength than AAGGBS mortars. A split tensile strength of 0.25Mpa is found at 800°C for AASF(slag:fly ash=70:30) where we found a total collapse of sample for AAGGBS mortar samples. This may be explained due to the presence of Fly Ash, different polymeric phases of sodium aluminosilicates are formed which have long chain like molecular structure giving tensile strength to the samples.

4.3.7. Ultrasonic Pulse Velocity Test.

4.3.7.1. Ultrasonic Pulse Velocity values vs. Temperature Levels.

Table 4.3.7.1.
UPV vs. Temperature Levels Temperature Levels (2hrs & 4hrs duration of heating) for different mortar types

TEMP.(°C)	UPV (in Km/s)									
	AAS		AASF(80:20)		AASF(70:30)		AASF(60:40)		Slag Cement	
	2hrs	4hrs	2hrs	4hrs	2hrs	4hrs	2hrs	4hrs	2hrs	4hrs
0	5.4	5.4	4.2	4.2	3.6	3.6	3.3	3.3	3.1	3.1
400	2.1	1.2	2.5	1.2	2.1	1.1	2.4	1.2	1.5	1.1
600	1.2	0.8	1.6	1.1	1.3	1.2	1.5	0.6	1.0	1.2
800	1.0	1.2	1.1	0.5	1.2	0.6	1.5	2.4	0.5	0.5

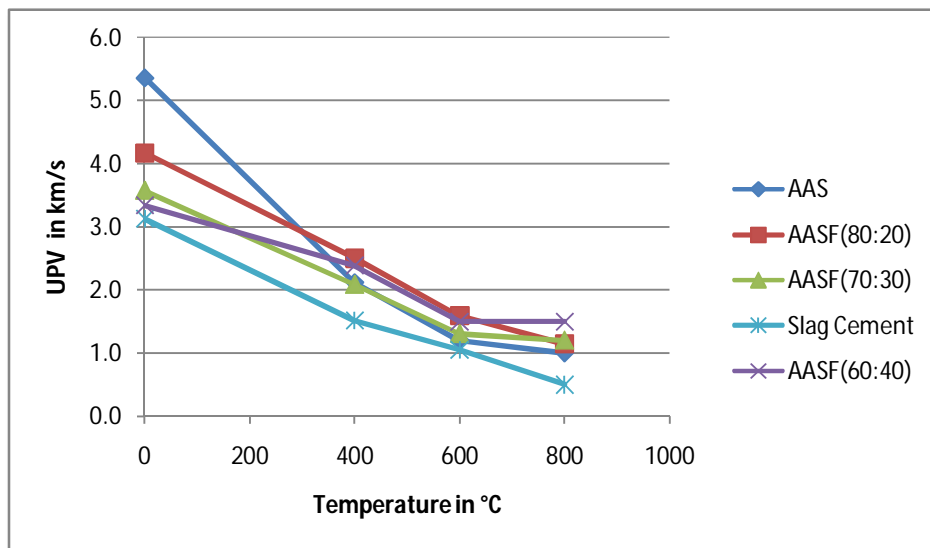


Fig. .4.3.7.1(a). UPV vs. Temperature Levels (2hrs duration of heating) for different mortar types.

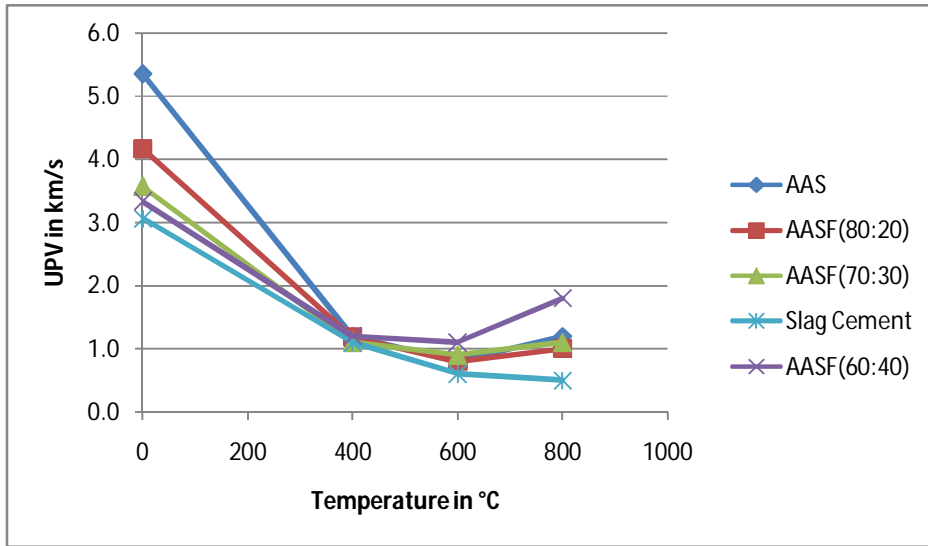


Fig.4.3.7.1(b). UPV vs. Temperature Levels (4hrs duration of heating) for different mortar types.

4.3.7.2. Ultrasonic Pulse Velocity vs. Duration of heating.

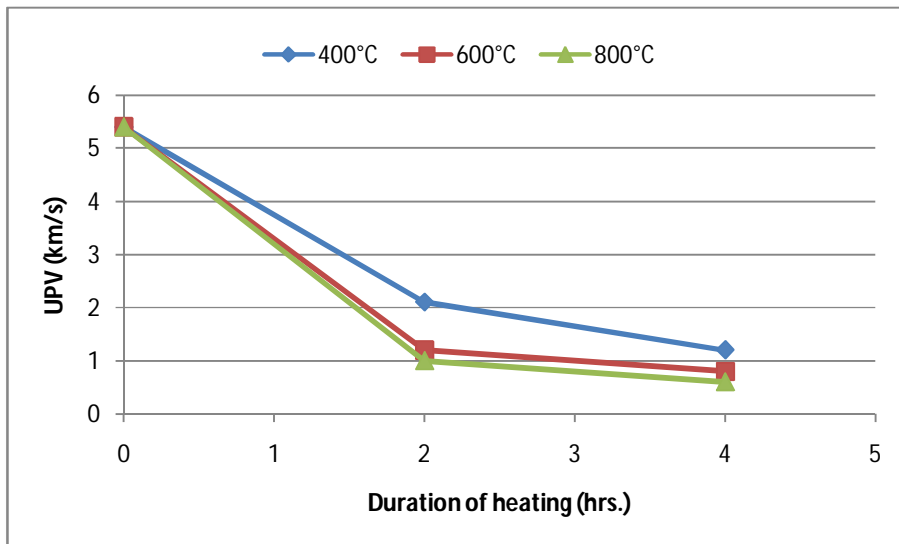


Fig.4.3.7.2.(a). UPV vs. Duration of heating for AAGGBS mortar

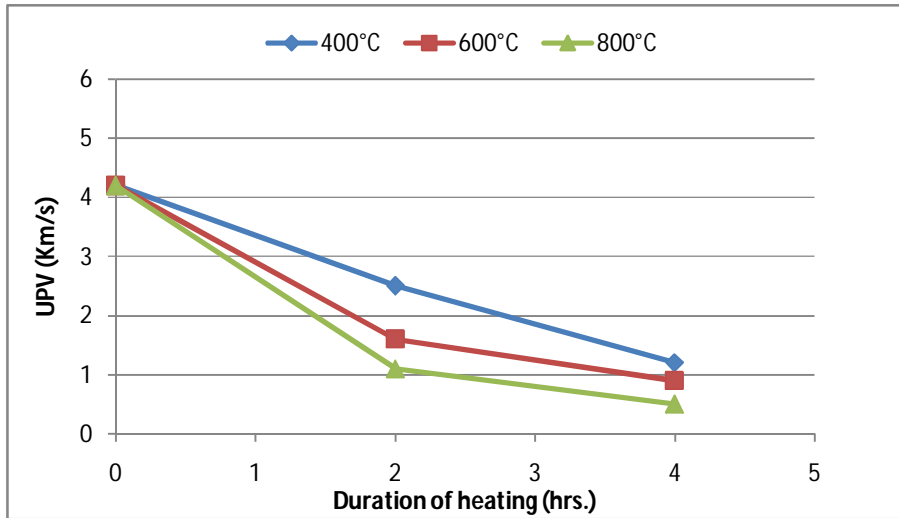


Fig.4.3.7.2.(b) UPV vs. Duration of heating for AASF (Slag:Fly Ash= 80:20) mortar.

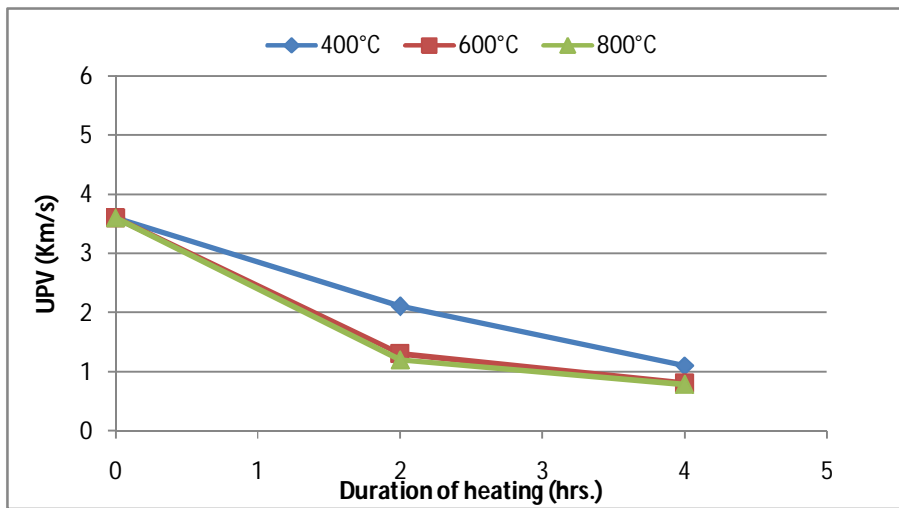


Fig.4.3.7.2.(a). UPV vs. Duration of heating for AASF (Slag:Fly Ash=70:30) mortar.

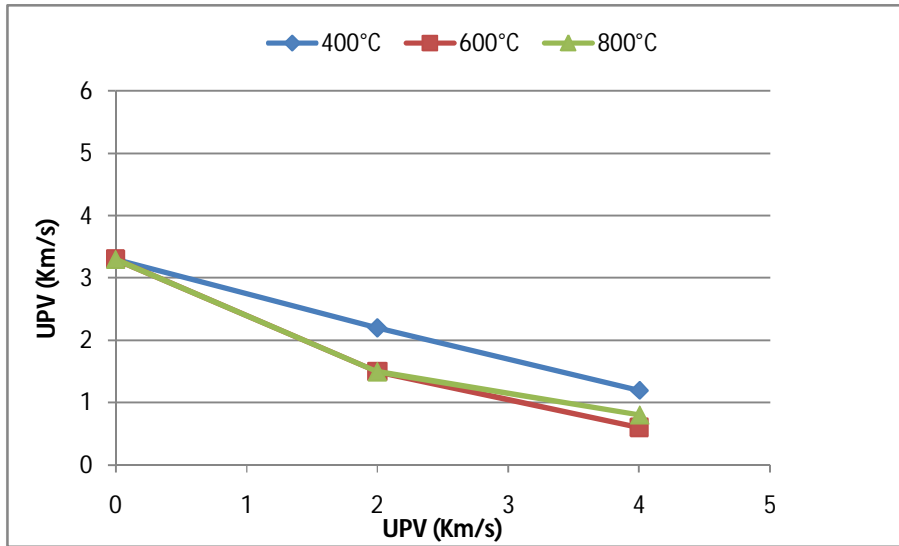


Fig.4.3.7.2.(a). UPV vs. Duration of heating for AASF (Slag:Fly Ash= 60:40) mortar

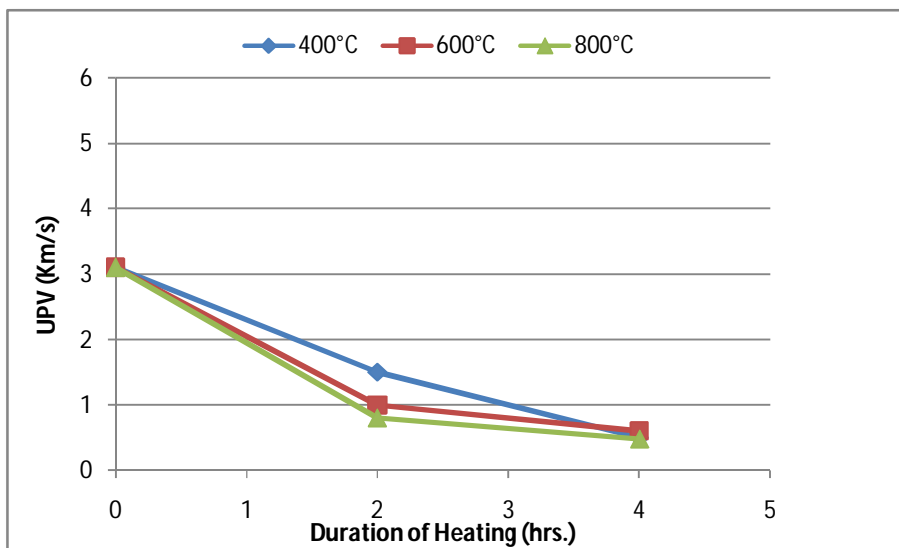


Fig.4.3.7.2.(a). UPV vs. Duration of heating for CSC mortar

The UPV values are with confirmation to compressive strength. There is drastic change in UPV values initially both in terms of temperature levels and duration of heating. The rate of decrease is less for AASF (60:40) mortar specimens for higher temperature levels and duration of heating. This may be due the fact the hydrated products fuses at high temperature with the formation of different phases of sodium aluminosilicates of reduced pore size.

4.4. Micro-structural Study.

4.4.1. SEM of Alkali Activated GGBS mortar.

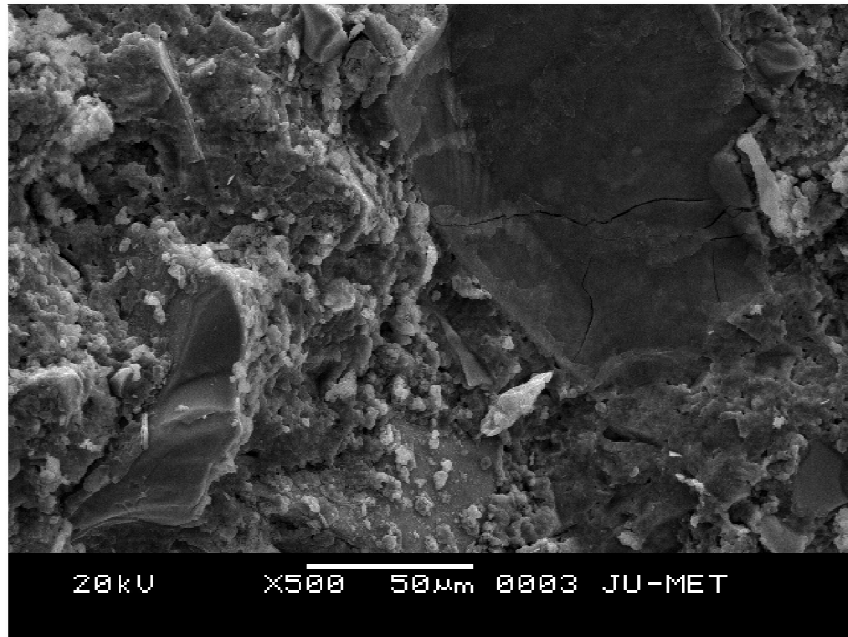


Fig.4.4.1.1(a). SEM image (500 X) of Alkali activated slag with Admixture

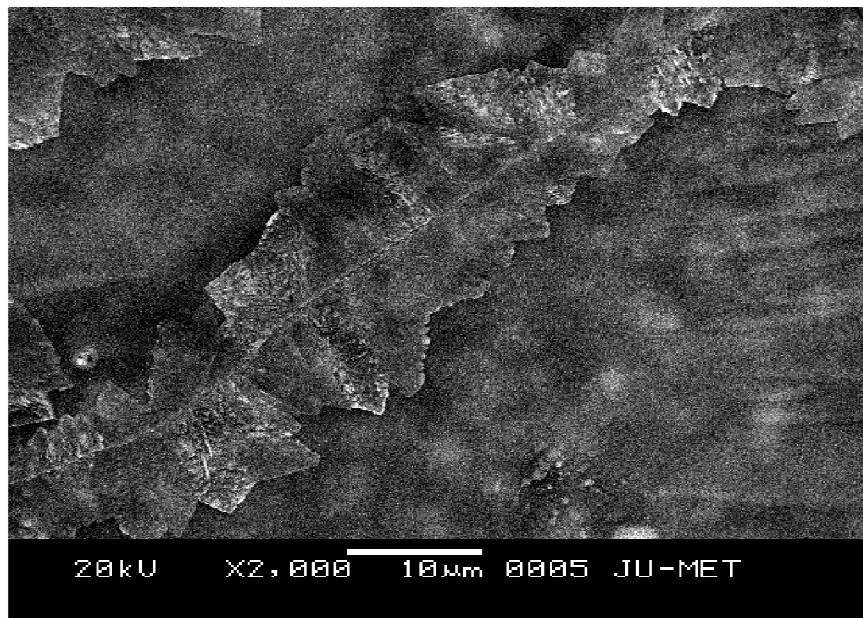


Fig.4.4.1.1(b). SEM image(2000 X) of Alkali activated slag with Admixture

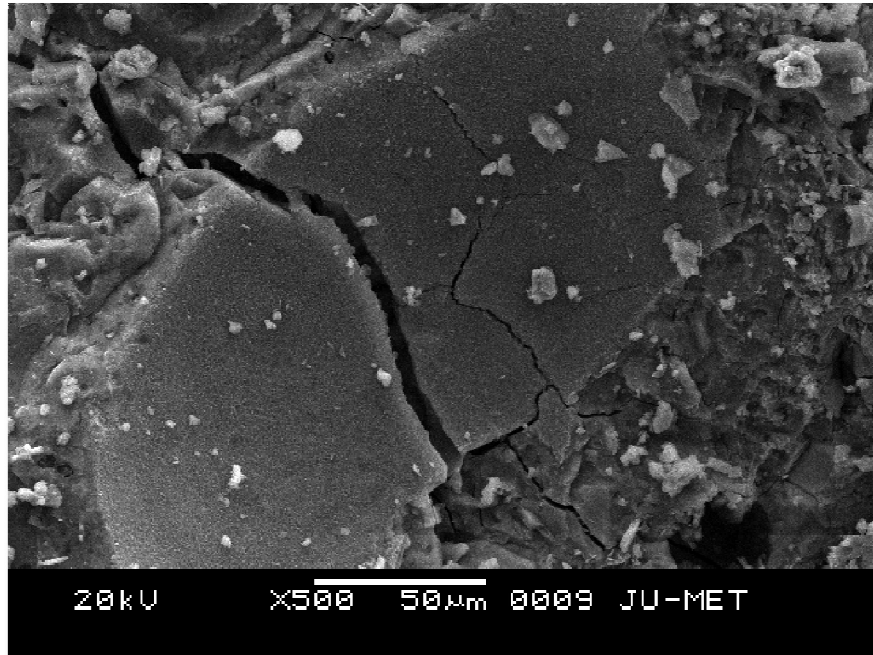


Fig.4.4.1.1(c).SEM image (500 X) of Alkali activated slag without Admixture



Fig.4.4.1.1(d).SEM (2000 X) image of Alkali activated slag without Admixture

The SEM images reveal that the shrinkage cracks are more prominent for specimens not prepared with admixtures, which resulted in lower compressive strength than the samples prepared with Admixtures. Enlarged images shows a marked difference in texture. The unreacted slag is very less for both the cases. The surface is smoother for the specimen

prepared with admixtures. However fern like structure can be seen for the samples prepared with admixtures this may due to the fact of unreacted admixture due to the presence of alkali.

SEM images of Normal Slag Cement mortar.

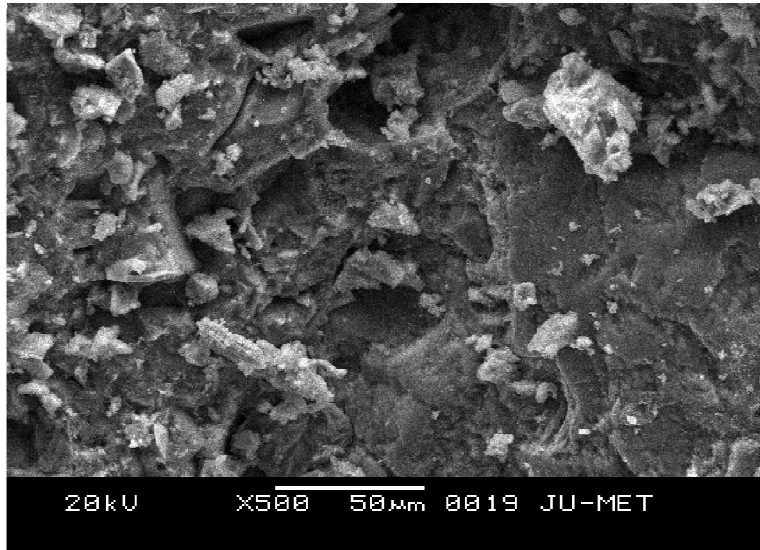


Fig.4.4.2.1(a).SEM image (500 X) of Normal Slag Cement with Admixture

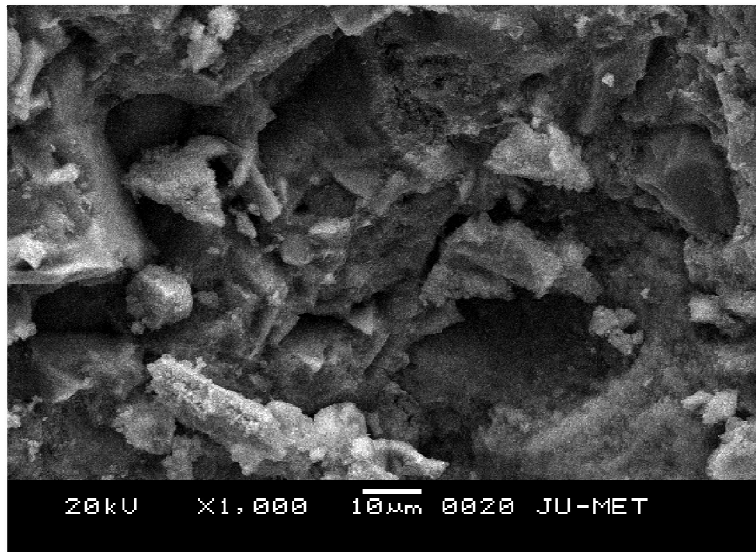


Fig.4.4.2.1(b).SEM image (1000 X) of Normal Slag Cement with Admixture

For normal slag cement mortar sample the SEM images show large amount of unreacted C-H phases. The C-S-H has not been fully developed to give a dense matrix.

4.4.2. SEM of Alkali Activated GGBS mortar exposed to high temperature.

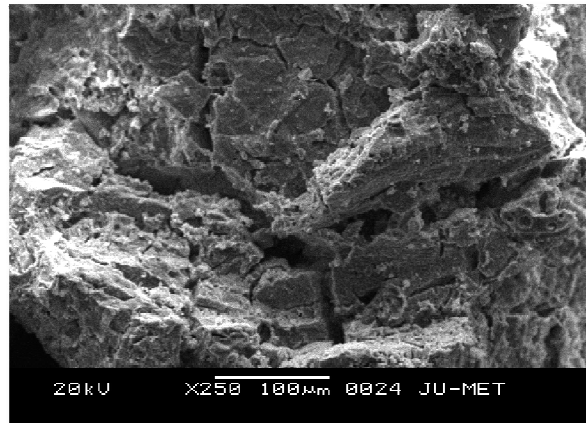


Fig.4.4.2.1 (a) SEM images. (250X) of Alkali Activated GGBS mortar exposed to 800°C for 2hrs

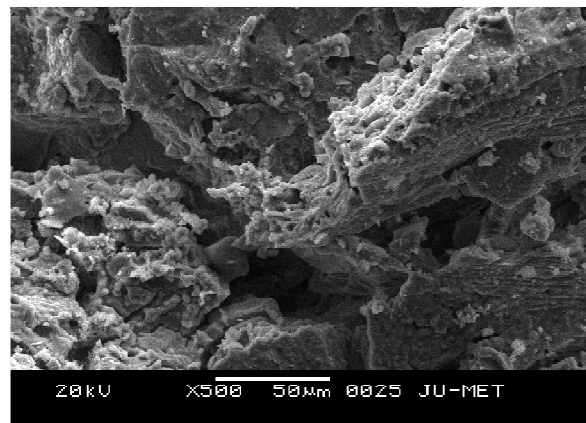


Fig.4.4.2.1 (b) SEM images. (500X) of Alkali Activated GGBS mortar exposed to 800°C 2hrs

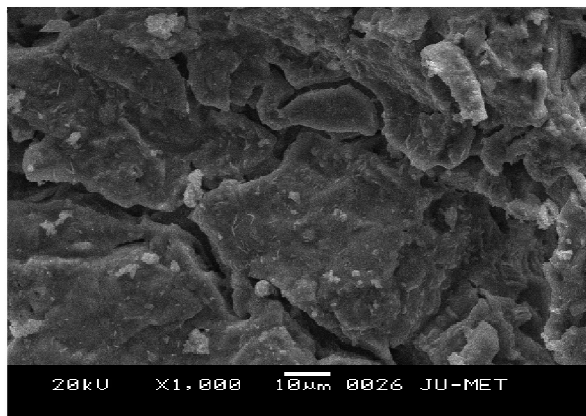


Fig.4.4.2.1(c). SEM images (1000X) of Alkali Activated GGBS mortar exposed to 800°C

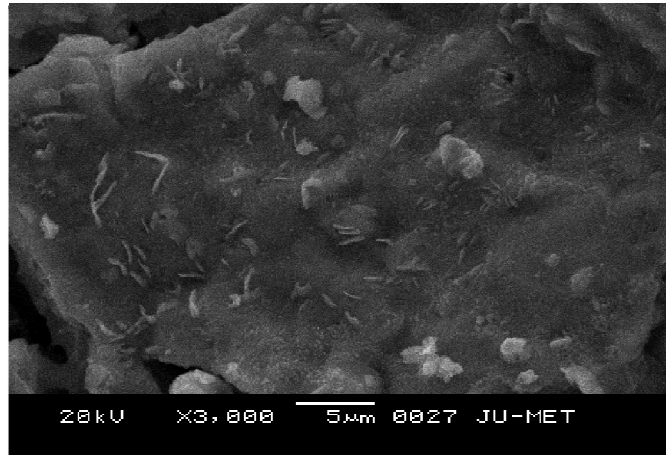


Fig.4.4.2.1(d).SEM (3000X) images of Alkali Activated GGBS mortar exposed to 800°C

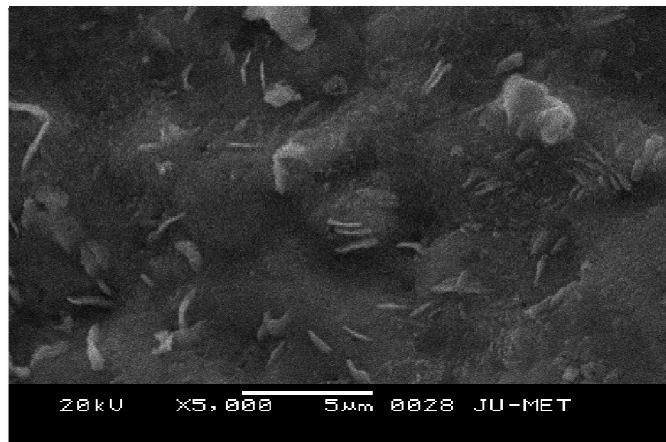


Fig.4.4.2.1(c).SEM (5000X) images of Alkali Activated GGBS mortar exposed to 800°C

The exposure to high temperature shows complete deterioration of C-S-H matrix. The above figures clearly demonstrate the difference between the structure of unexposed specimen with the exposed specimen. In Alkali activate slag mortar specimens a great number of microcracks are observed, which were caused by very high drying shrinkage. It is one of the typical characteristics of alkali activated slag reported by previous studies ^[26-27]. These microcracks get reduced on application of admixture. When the temperature increases, the micro cracks became more significant, this can be verified from the SEM images of specimens. Both the microcracks propagation and the change of microstructure can also be observed. Compared with the reference matrix, the fracture surface of specimens after exposure to 800 °C for 4 hrs, a significant change of matrix morphology can be found. The reason may contribute to the decomposition of C-S-H gels and the formation of crystalline phase “Gelhenite” with the chemical formula of $\text{Ca}_2\text{Al}_2\text{SiO}_7$ ^[27]

5. Conclusion

The primary aim of this present study is to appreciate the engineering properties of Alkali activated mortars. Engineering properties like Workability, Water sorptivity, Compressive strength, Split Tensile Strength. Water absorption, Apparent porosity, Ultrasonic pulse velocity, Microstructure of AAGGBS have been studied in presence and absence of Admixtures. Mortars were then exposed to different temperature levels with varying duration of heating. Finally the properties are compared with that of unexposed specimens.

Performance of AAGGBS exposed to high temperature has not always shown encouraging performance. GGBS was partially replaced by different proportion of Fly Ash and some improvement in performance was noticed.

Certain broad references can be made as follows:

1. Improvements in strength and workability of AAGBS have been found on application of admixture.
2. The effect of admixture on mortar mix was noticed better at lower Na_2O concentration and at higher water/binder ratio. An increase of compressive strength and split tensile strength have been found 31.62% & 44.4% respectively in presence of admixture (refer Table 4.2.5.1 and 4.2.6.1).
3. Maximum increase of flow value of 21.21 % has been found for AAGGBS mortar in presence of admixture (refer Table 4.2.1.1).
4. Maximum reduction of water absorption of 51.54%; apparent porosity of 48.39% and sorptivity of 40% (refer Table 4.2.2.1; 4.2.3.1 and 4.2.4.1) was found for AAGBS mortar specimens with admixtures.
5. Increased loss of weight was noticed and the rate of weight loss increases with the increase of temperature level and duration of heating.[Refer Fig.4.3-1.1(a) & 1.2(b)]
6. Increase of water absorption, apparent porosity and sorptivity was noticed for specimen exposed to high temperature and the rate of increase is more for higher temperature level.
7. Residual Compressive strength of AAGGBS specimens exposed to elevated temperature was found very low (refer Table 4.3.5).Practically almost 100% loss of compressive strength have been noticed.

8. Similar results were observed for specimens out of conventional slag cement mortar. It may be said that the hydration product and the products formed due to heating effect may be similar.
9. Increase of both Water Absorption and Porosity was observed for blended (Fly ash + GGBS) alkali activated mortars with the increase of fly ash content [refer Fig.4.2.8.1(a)].
10. The compressive strength of alkali activated blended (Fly ash + GGBS) mortar was found to be lesser than AAGGBS mortar but greater than conventional Portland slag cement mortar (refer fig. 4.2.8.2).It may be concluded the C-S-H formed in AAGGBS mortar specimens are more and stronger than the blended variety.
11. Split tensile strength for AASF however was found greater than AGGBS(Refer fig. 4.2.8.2) this may be due to formation three-dimensional polymeric chain and ring structure consisting of Si-O-Al-O bonds.
12. The inclusion of fly ash to the alkali activated system improves the refractory properties. A 8.98 % increase in residual compressive strength was observed for AASF(Slag:Fly ash=60:40) over AAGGBS mortar specimens when exposed to 800°C for a duration of 4hrs.(refer table 4.3.5.1)
13. A decrease in water absorption, apparent porosity and sorptivity was also observed for AASF (Slag:Fly ash=60:40) mortar specimens exposed to temperature level of 800°C for heating duration of 4hrs .
14. Alkali activated blended mortars (GGBS+Fly ash) with higher fly ash proportion may be used in various engineering works exposed to optimum temperature.

Scope of Future Study:

The following areas may be explored:

1. More study on durability of the present work like long term sorptivity, effect of acids, chloride penetration, alkali aggregate reaction, carbonation, etc. may be carried out as future study.
2. The same alkali activated system may be studied for concrete mix.
3. More study on microstructure is required for better understanding of products formed in Alkali activated composites.

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Appendix-I

Composition of materials

1. Ground Granulated Blast Furnace Slag.

It was sourced from Durgapur Steel Plant, Durgapur, India

Chemical composition of fly ash by XRF

Chemical composition	SiO ₂	CaO	Al ₂ O ₃	MgO	Fe ₂ O ₃	SO ₃	Na ₂ O	K ₂ O	TiO ₂	MnO	LOI*
Percentage	28.5	37.5	19.5	7.2	0.33	0.4	0.6	0.3	1.25	0.35	0.8

2. Fly-Ash

It was sourced from Kolaghat Thermal Power Plant, Kolaghat, India.

Chemical composition of fly ash by XRF (Source: *Kalyan Kr. Mandal, JU, et.al*)

Chemical composition	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	TiO ₂	CaO	MgO	K ₂ O	Na ₂ O	SO ₃	P ₂ O ₅	LOI*
Percentage	56.01	29.8	3.58	1.75	2.36	0.3	0.73	0.61	Nil	0.44	0.40

*Loss on Ignition.

3. Sodium Silicate Solution:

Manufactured by LOBA Chemie Pvt. Ltd, 107 Wodehouse Road, Jahangir villa, Mumbai

Specification:

Assay of Na₂O=7.5%-8.5%

Assay of SiO₂=25-28%

Density=1.39gm/cc

pH = 11.2

4. Sodium Hydroxide pellets:

Manufactured by: Merck Specialties Pvt. Ltd. Shivsagar Estate, Worli, Mumbai.

Assay of NaOH=97%

5. Admixtures

Superplasticizer: Sika, Viscocrete 2004NS;

Retarder: Fosroc, Conplast R

6. Portland Slag Cement:

Manufactured by: JK Cement, Gotan, Rajasthan

Test Results obtained from the manufactures catalog:

SAMPLE TEST TYPE	RESULT OBTAINED FROM TEST
CHEMICAL TEST	
1. Magnesium oxide(Mgo)	5.28
2. Sulper tri oxide (SO ₃)	1.09
3. % Sulphide sulphur (S)	0.65
4. Loss of Ignition	1.15
5. Insoluble residue	1.11
6. Total Chloride Content	0.046
7. Alkalies % K ₂ O	1.48
8. Alkalies % Na ₂ O	0.30
PHYSICAL TEST	
A Fineness	360
B Soundness	
1. Lechatelier	1.0
2. Autoclave	0.128
C Setting Time	
1. Initial	124
2. Final	224
D Compressive Strength	
1. 3 Days (72+/- 1Hrs)	29.6
2. 7 Days (168+/- 1Hrs)	39.3
3. 28 Days (672+/- 1Hrs)	50.0

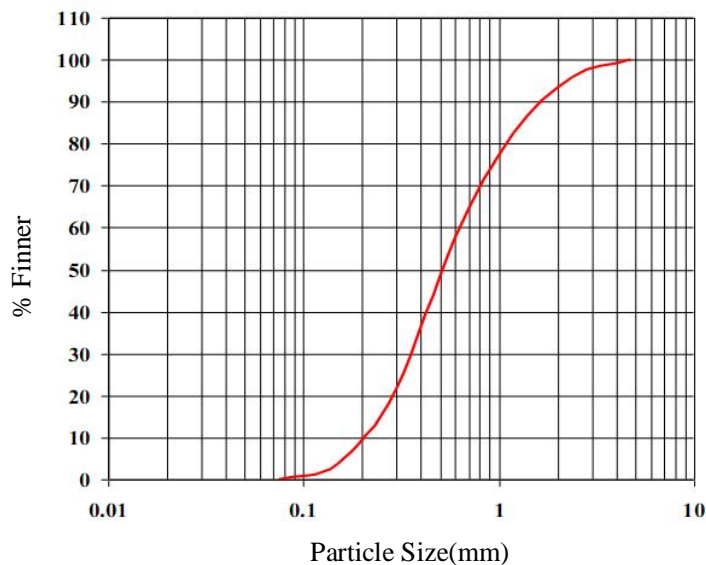
7. Sand

Fine aggregate used was river sand.

Specific gravity=2.55

Fineness modulus =2.70

From particle size distribution of the sand it falls in Zone-II, as per IS;383-1970



Appendix-II

Calculation of quantity of alkali activators

Sample Calculation for different ingredients needed for producing AAC mortar of specified parameters:

Parameters

Na₂O=6% by wt of GGBS

SiO₂O=6% by wt of GGBS

Binder/Sand in activator solution=1

Water/GGBS=0.33

Admixture=1.2% Viscocrete 2004 NS; 1.5% Conplast R

Assume:

Quantity of GGBS=1000gm

Therefore:

Na₂O required=60 gm.

SiO₂ required = 60 gm.

Water required=330 gm

Sand=1000 gm

Now.

1000gm Sodium Silicate contains 265gm of SiO₂, 80gm of Na₂O and 655gm of water

Therefore to get 60gm of SiO₂ in activator solution

Quantity of Sodium Silicate needed=265/1000 x 60=226.415 gm

Quantity of Na₂O in 226.415 gm Sodium Silicate=80/100 x 226.415=18.11 gm

Remaining Na₂O required= 60-18.11 =41.89 gm which is to obtained from Sodium Hydroxide pellets

Again,

1000gm Sodium Hydroxide in pellets from contains 775gm of Na₂O and 225gm of water.

Therefore to get 41.89 Na₂O

Quantity of NaOH pellets required=1000/775 X 41.89=54.05 gm

As NaOH pellets are 97% pure.

Actual Quantity of NaOH pellets required=55.72 gm.

Now, Water in 226.415 gm Sodium Silicate Solution=655/1000 x 226.415 =148.301 gm.

Water in 55.72gm NaOH pellets=225/1000 x 55.75 = 12.537 gm.

Extra Water to be added= 330-(148.301+12.537)= 139.162 gm

Admixture =Viscocrete2004 NS @1.2%by wt. of GGBS=12 gm ;

= Conplast R @1.5%by wt. of GGBS=15 gm

Appendix-III

Sample Calculations to get engineering properties from test results

Apparent porosity					
Alkali=		4			
Silica=		6			
Water/GGBS=		0.33			
Samp. No.	Oven Dry Wt.(A)	Saturated Surface Dry (B)	Submerged Saturated Weight (C)	Apparent porosity $n_a(\%)$ $= (B-A)/(B-C) \times 100$	Avg
AA1	272.5	284.8	78.3	5.96	6.49
AA2	270.5	284.5	74.2	6.66	
AA3	275.2	286.7	76.8	5.48	
AA4	270.6	287.7	74.5	8.02	
AA5	272.1	286.5	72.2	6.72	
AA6	271.6	284.5	73.2	6.11	

Water Absorption				
Alkali=		4		
Silica=		6		
Water/GGBS=		0.33		
Samp. No.	Oven Dry Wt.(A)	Saturated Surface Dry (B)	Water Absorption (B- A)/Ax100	Avg.
AA1	272.5	284.8	4.51	5.04
AA2	270.5	284.5	5.18	
AA3	275.2	286.7	4.18	
AA4	270.6	287.7	6.32	
AA5	272.1	286.5	5.29	
AA6	271.6	284.5	4.75	

Sorptivity						
Water/GGBS=0.33; Na ₂ O=8% ;SiO ₂ =6%						
Time(min)		Moist Weight (gm)	Increase in Wt.(gm)	Cumulative Increase in Wt.(gm)	Cumm. Inc/X- Area (gm/mm ²)	Sorptivity (gm/mm ² /min ^{0.5})
Time (min)	Sqt. Of Time,√t (min ^{0.5})					
A	B	C	D	E	F= E/(50x50)	G=F/B
0	0	277.4	0	0	0	0
1	1	277.8	0.4	0.4	0.00016	7.16E-05
5	2.236068	278.4	0.6	1	0.0004	1.79E-04
10	3.162278	278.5	0.1	1.1	0.00044	1.39E-04
20	4.472136	278.6	0.1	1.2	0.00048	1.07E-04
30	5.477226	278.7	0.1	1.3	0.00052	9.49E-05
60	7.745967	278.8	0.1	1.4	0.00056	7.23E-05
80	8.944272	278.8	0	1.4	0.00056	6.26E-05
120	10.95445	278.8	0	1.4	0.00056	5.11E-05
150	12.24745	278.8	0	1.4	0.00056	4.57E-05
180	13.41641	278.8	0	1.4	0.00056	4.17E-05
210	14.49138	278.8	0	1.4	0.00056	3.86E-05
240	15.49193	278.8	0	1.4	0.00056	3.61E-05