INFLUENCE OF CALCINED ZEOLITE ON PHYSICAL-MECHANICAL PROPERTIES OF SELF COMPACTING CONCRETE CONTAINING GLASS POWDER

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The influence of glass powder as fine aggregate when used in combination with calcined zeolite for the betterment of the workability and strength improvement of self compacting concrete was analyzed in the study. Initially the fresh state properties such as the passing ability, flowing ability and the filling capacity of the SCC were examined. Then the mechanical strength of the zeolite- glass powder containing self compacting concrete was investigated using compression, flexural and split tensile tests. The durability of the SCC was assessed by the water absorption, porosity, chloride permeability and sorptivity tests. The durability and strength results were validated with microstructure analysis such as XRD and scanning electron microscopy studies. Furthermore the Alkali – silica reactivity of the concrete was characterized to analyze the reactivity of the glass aggregates. The results show that the SCC with 30% of glass powder as fine aggregate replacement and 20% of cement replacement by zeolite showed overall better performance. Thus the use of calcined zeolite can reduce the expansiveness in concrete caused by reactive glass powder aggregates and is also an eco-friendly option to minimize the cement utilization thereby reducing the global warming.

Keywords: *Durability, Glass powder, Mechanical strength, Microstructure, Natural Zeolite and Self compacting concrete*

1. Introduction

One of the upcoming technologies in the concrete field is the self compacting concrete (SCC) that provides many benefits for the engineers such as ease of filling in complex areas, better surface finish, complex reinforcement, easy placement and better durability [1].Several researchers have ventured into this field due to the attractive characteristics of SCC by the incorporation of many new materials and the scientists have extended their knowledge beyond innovation levels [2]. To revolutionize the concrete technology and to extend the boundary of their applications of concrete, SCC was introduced globally by use of non conventional materials [3]. Among the various ingredients used in concrete, fine aggregate plays an important role in improving the characteristics of the concrete [4]. Due to the global demand of concrete, the natural sand consumption is found to be high. Sand mining causes severe ground water depletion which results in major environmental problems and many other ecological problems have also been reported due to the rapid extraction of natural sand which makes it essential to find a suitable replacement for natural sand [5]. Hence the alternative material must satisfy the properties provided by the natural sand. Glass powders are generally non water absorbing in nature hence their usage as fine aggregates in self compacting concrete can yield high performance and workable self compacting concretes [6-9]. However the alkali silica reaction and crack formations are the most common threats faced by the concrete when glass aggregates are used [10-12]. The chemical reaction of the glass aggregates with the alkali hydroxides present in the pore solution of concrete results in expansive gel formation known as ASR gels [13]. The calcium Silicate hydrate gel formation from by reaction of glass aggregates with the alkali present in pore solution causes expansion of the concrete [14-16]. The ASR gels appear around the aggregates or sometimes within the aggregates thus expanding into the concrete matrix disrupting their structure [17]. The increased absorption of OH^- ions by the gels may also cause a significant increase in the stresses of concrete due to high swelling pressure [18]. However the alkali silica reaction is a long term process and may take several years to develop the significant effects in concrete which makes their characterization a time consuming study [19]. Accelerated ASR test is one method that is employed to synthetically create ASR gels [20]. Some studies tried to quantify and qualitatively analyse the ASR gel formations by modifying the reactivity levels and the alkali levels of the aggregates in concrete [21]. Several studies also tried to elucidate the expansion of ASR gel when it is exposed to the alkali solution [22]. On similar lines the present study tries to examine the ASR expansion of the concrete when glass aggregates are used in combination with calcined zeolite.

Zeolites are alumino-silicate in nature and when they are used in concrete can improve their strength and durability [23,24]. The mitigations of alkali silica reaction are one of the most important properties of zeolite. The utilization of zeolite which is a pozzolanic material reduces the calcium

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hydroxide present in concrete by transforming them into CSH gels [25]. Hence their usage in concrete in combination with glass powders can yield significant results. The use of zeolites in natural form may function as internal curing agent but they affect the workability of concrete due to their high water demand [26, 27]. Self compacting concretes are mainly knows for their high fluidity and workability and these are important properties for use in practical applications [28]. Therefore in this present study an attempt is done to utilize the zeolite in its calcined state in self compacting concrete, as partial replacement of cement and glass powder, as partial replacement of fine aggregate.

The present study mainly focuses on the key areas such as strength and durability characteristics of the SCC. The possibility of producing good quality SCC by the cement replacement with zeolite at calcined state is attempted to attain better resistance towards alkali silica reaction and sulphate attack in self compacting concrete.

2. Research Significance

The study is based on the intention of producing self compacting concrete with increased strength and durability by using zeolite and glass powder. Usage of glass powder is common in self compacting concretes but insufficient data and guidelines regarding their usage in combination with zeolites have prevented its usage in self compacting concrete. Complete analysis of the properties of the concrete is essential when new combinations are to be added in the self compacting concrete. Developing the perception of using glass powder as a fine aggregate replacement with calcined zeolite as cement replacement is handled in this research by the measurement of workability, strength and durability of the self compacting concretes.

3. Materials

In the present work, the commercially available Ordinary Portland cement (OPC 53) from Coromandel cement is used as main binder. Table 1 depicts the chemical composition of cement, glass powder and zeolite used in this study. The crushed hard granite stones of nominal size 12mm are utilized as coarse aggregate for the self compacting concretes. The finely ground glass powder was used as fine aggregate replacement and calcined synthetic zeolite was used as cement replacement. The naphthalene based superplasticizer of commercial grade is used as water reducing agent.

Table 1

Chemical composition of Raw materials

Oxide (%)	**Cement**	**Glass powder**	**Natural zeolite**
SiO_2	21.25	71.2	63.87
Al_2O_3	5.37	0.98	11.47
Fe_2O_3	4.28	0.15	0.215
CaO	63.3	8.7	2.37
MgO	1.84	3.7	1
SO_3	2.7	< 0.05	-
Na_2O	-	12.81	6.81
K_2O	-	0.35	0.94
Cl_2	0.003	< 0.005	-
LOI	1.26	0.92	11.97

3.1. Calcination of Zeolite

The calcination of zeolite was carried out in a muffle furnace at a temperature of about 800^0C for a period of 8 hours and then stored in air tight container until mixing of concrete is done [29-31]. The temperature of 800^0C was chosen from

Table 2

Mix proportion of self compacting concrete series

Mix Id	**Binder (%)**		**Fine aggregate (%)**		**Coarse aggregate (%)**
	Cement	**Calcined Zeolite**	**River Sand**	**Glass Powder**	
SCM	100	-	100	-	100
SCGM1	95	5	92.5	7.5	100
SCGM2	90	10	85	15	100
SCGM3	85	15	77.5	22.5	100
SCGM4	80	20	70	30	100
SCGM5	75	25	62.5	37.5	100
SCGM6	70	30	55	45	100

the previously established research works that state that the structural modification of the zeolite crystals takes place at that temperature [32].The XRD pattern of the natural zeolite and the calcined zeolite showed Clinoptilolite peaks as the major crystalline compound present in zeolite. No other crystalline peaks were observed, indicating that no new compounds are formed due to calcination and only thermal collapse of the lattice framework has occurred due to calcination.

4. Mix Proportion

In the present research work, the self compacting concrete mixes of M30 grade were produced using EFNARC guidelines. The control concrete mix was produced with the cement content at 400 kg/m^3 of concrete and the proportion of the aggregates used for the self compacting concrete were 50% for coarse aggregate and 50% for fine aggregate. The coarse aggregate to concrete volume was kept as 0.32. The basic proportion was modified by the partial replacement of cement with calcined zeolite and sand with glass powder at various percentages (as are shown in Table 2). Totally 7 mixes were produced by modifying their replacement levels of cement from 0% to 30% and for river sand at 0% to 45% with 5 and 7.5 increment levels. Throughout the concrete mixes the super-plasticizer dosage was fixed at 2.5% and the water / binder ratio was maintained as 0.4 constantly.

5. Experiments

The workability of the fresh self compacting concrete mixes were investigated using J-ring, L box, slump flow, T50 and V funnel test as per the procedure stated in EFNARC 2002. The compressive strength of the concretes at ages of 14, 28 and 90 days were determined on cubic specimens of sizes 150 x 150 x 150 mm according to the standard IS 516-1959. The flexural strength of the beams of sizes 100 x 100 x 500 mm was determined at 7, 28, 90 and 180 days as per the code IS 516:1959 . The split tensile strength was determined on cylindrical specimen of sizes150 mm diameter x 300 mm high as per IS 5816:1999 at the age of 7, 28, 90 and 180 days. The water absorption test was determined as per ASTM C 642. The porosity test was performed in 150 mm cubic concrete specimens as per the saturation methods mentioned in EN 1936-2006 and sorptivity of the 100 mm size cubic specimens were tested. The rapid chloride penetration test was performed in the self compacting concrete mix as per the procedure stated in the ASTM C 1202 on cylindrical specimen of sizes 50±2 mm the height and 100 ± 2mm diameter; the amount of chloride ions through the concrete specimens is measured. The experiment to characterize alkali silica reaction was done as per the procedure stated in ASTM C 1260 using the self compacting concrete prisms of sizes (75x75x300 mm) [33] and measuring their length changes at various ages. The accelerated ASR expansion test was done by conditioning the concrete specimens in 1M NaOH solution for respective time periods at a temperature of 80^{0}C [34]. All the specimens were subjected to water curing for 28 days and maintained at room temperature until the testing is done. The testing of the specimens was done in triplicates and the average forms the final result.

6. Results and discussions

6.1. Fresh state concretes

The fresh state self compacting concretes containing variant proportions of calcined zeolite showed a significant improvement in the flowing ability, filling ability, segregation resistance and passing ability as are shown in Table 3. The use of zeolite in calcined state in self compacting

Table 3

Fresh state characteristics of self compacting concrete mixes

Mix Id	Fresh state characteristics				
	Slump flow (mm)	T50 Slump flow (sec)	V-Funnel (sec)	J-Ring (mm)	L-Box
SCM	712	2.65	8.1	7.86	0.88
SCGM1	716	2.61	7.9	7.92	0.9
SCGM2	721	2.55	7.4	8.34	0.93
SCGM3	724	2.51	7.1	8.65	0.95
SCGM4	729	2.49	6.9	8.91	0.96
SCGM5	736	2.45	6.7	9.24	0.97
SCGM6	739	2.43	6.6	9.31	0.97

concretes showed improvement in the workability due to the combined additions with glass powder and modifications in the pore structure aided by calcination. The external surface area is reduced and less amounts of water are absorbed by the zeolite leading to improving workability. The internal pore reduction is also caused by the calcined zeolite. The calcinations effect in the zeolite also causes a change in the exchangeable ions that can take up the water. The modification of the crystal structure due to calcination also modified the number of micro pores that can absorb water thereby causing less disruption to the flowing ability of the concrete. Moreover the molecular sieves of zeolite can also hold excess waters that are present in concrete due to the increasing substitution of glass powder thereby resisting to bleeding and segregation. This change in the cationic structure of zeolite also minimizes the water that zeolite can hold up in their internal structure thereby minimizing the imbibed water providing water for mixing.

6.2. Compressing strength

The improved strength of the calcined zeolite containing self compacting concrete containing also glass powder as is shown in Figure 1 may be due to the destabilization of the internal pores of zeolite that improved the denseness of the concrete. The glass powder substitution also well occupied the inter layer space of the concrete leading to improved strength at all ages. The pozzolanic reactivity of zeolite and glass powder has also caused the formation of hydrates CSH gels that improved the strength of the concrete. The water demanded by the self compacting concretes to develop the strength is also much reduced due to the inclusion of zeolite that can provide additional water for the transformation of CH into CSH due to their internal curing ability [35]. The reduction in the amount of pores present in zeolite due to calcination effect and glass powder substitution that prevented particle agglomeration in the self compacting concrete may also be the reason for the improved strength. The pores filling characteristics as exhibited by glass powder which at later ages showed pozzolanic activity also improved the strength of concrete at later ages. The decrement in the amounts of water absorbed by the zeolite in combination with glass powder effectively reduced the amounts of free evaporable water in the concrete which may be the main reason for the improvement in the mechanical strength properties of the self compacting concretes.

6.3. Flexural Strength

The self compacting concrete containing glass powder and calcined zeolite showed increased flexural strength at all ages when compared to the control concrete as is shown in Figure 2. 30% of glass powder together with 20% calcined zeolite is considered optimum for attaining the target flexural strength. The self compacting concrete containing

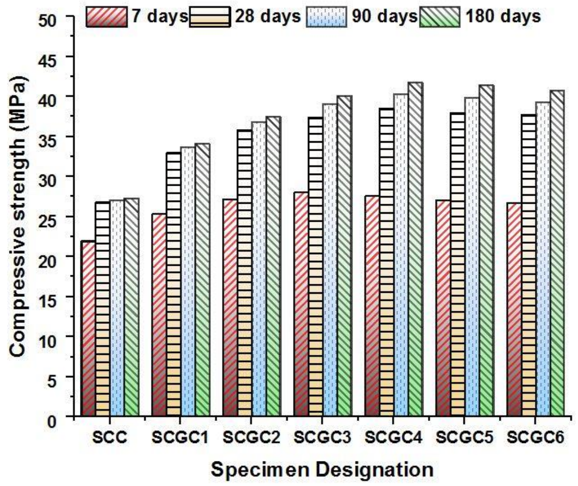

Fig. 1 - Compressive strength of SCC mixes at various ages.

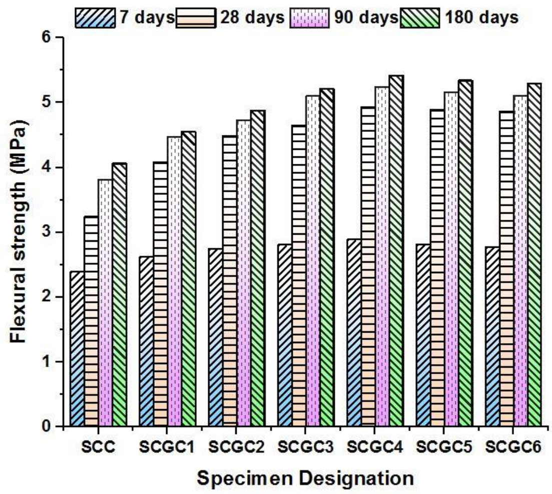

Fig. 2 - Flexural strength of SCC mixes at various ages.

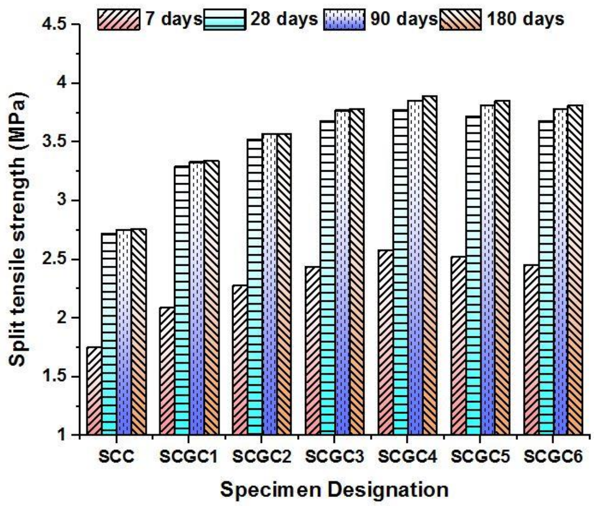

Fig. 3 - Split tensile strength of SCC mixes at various ages

calcined zeolite also resulted in higher flexural strength than control self compacting concrete even at high percentage substitutions of glass powder as fine aggregate. The addition of glass powder and calcined zeolite had a significant filler effect in self compacting concrete. The calcined zeolite effect in combination with the glass powder has a positive impact on improving the flexural behavior at all percentage substitution levels of zeolite in the self compacting concrete. By the use of calcined zeolite in the concrete, the strength improvement was found throughout the increasing replacement level in combination with the glass powder. The zeolite replacement of 30% caused strengthening of the bond in concrete which makes it less vulnerable to the effect of flexural failure.

6.4. Split tensile strength

Figure 3 depicts the cumulative effect of calcined zeolite and glass powder in improving the split tensile strength of the concrete. The curing days caused an inclined slope in improving the split tensile strength of the self compacting concrete. The brittleness caused by the glass powder has been eliminated by the addition of the calcined zeolite and has resulted in improved strength. Increased splitting tensile strength was observed with increased glass powder substitution at all ages. Reduction in bleeding and shrinkage has been resulted with the raise in the interfacial transition zone strength.

6.5. UPV (ultrasonic pulse velocity)

The ultrasonic pulse velocity showed an improved pattern for the self compacting concrete. In Figure 4 it is evident that the velocity of self compacting concrete mixes was improved throughout all the increasing percentage levels of replacement by glass powder and zeolite. This is mainly due to the usage of calcined zeolite that increased the denseness and reduced the pores present in concrete. The improvement in velocity of the concrete resulted in better pore structure which makes all the concrete fall under 'good' category. The self compacting concrete with better strength and durability are thus confirmed with their reduced travel time and increased ultrasound velocities.

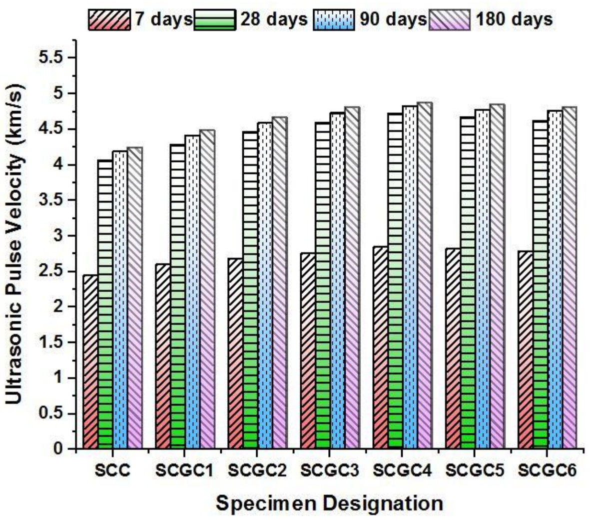

Fig. 4 - UPV of SCC mixes at various ages.

The durability of self compacting concretes have to be carefully observed since they are highly flowable and are mainly used as internal filling agents in congested reinforcements. When the durability of self compacting concrete is low then it affects the reinforcement which is an integral part of any concrete structure.

6.6. Water absorption

The values of water absorption of the self compacting concrete containing glass powder and calcined zeolite in comparison with the control concrete at all ages is shown in Figure 5. When calcined zeolite and glass powder is included, the concrete specimens resulted in reduced water absorption at all ages of concrete. The lower water absorption values of SCC concretes were mainly due to the combined effect of calcined zeolite and glass powder. The reduction in water absorption leads to the reduced porosity of the self-compacting concrete. Calcined zeolite has the property of filling pores and the fine glass powder has inter-granular filling capacity that causes the self compacting concrete to be dense with minimal water accessible pores.

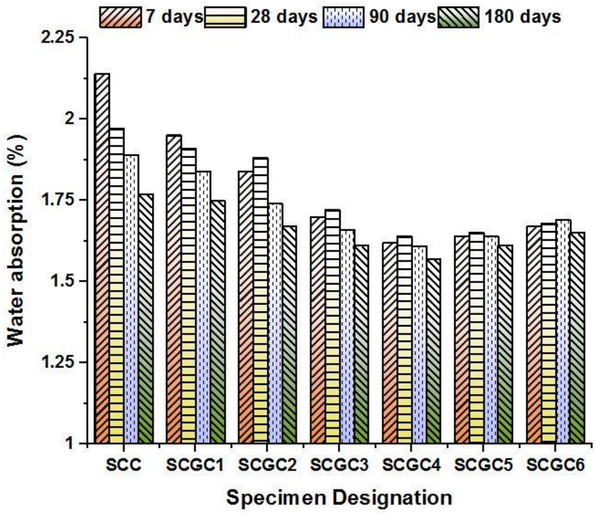

Fig. 5 - Water absorption of SCC mixes at various ages.

6.7. Porosity

The usage of calcined zeolite in the concrete showed a positive impact on porosity of self compacting concrete which is evident in Figure 6. Zeolite in its normal state is highly porous and water absorbed by saturation technique of the concretes is increased to a considerable extent [36]. Whereas the use of zeolite in calcined state yielded less number of pores in the concrete which easily blocks the continuous channels through which water can pass and reduces the cavities present in concrete that function as reservoirs in which water molecules can reside. The calcined zeolite modifies the pore structure and the voids present in concrete are found to be drastically reduced due to the modification in the crystal structure.

30% replacement of fine aggregate by glass powder showed least porosity value when compared to all the other concretes. The calcined zeolite effect resulted in complete reduction of pores in the concrete throughout their increasing level of replacement but the values slightly increased beyond certain threshold levels. This may be due to the increased fineness content in the concrete at higher substitution levels of glass powders. Thus the combined usage of calcined zeolite and glass powder helps in improving the porosity of the concrete which in turn increased the strength of self compacting concrete.

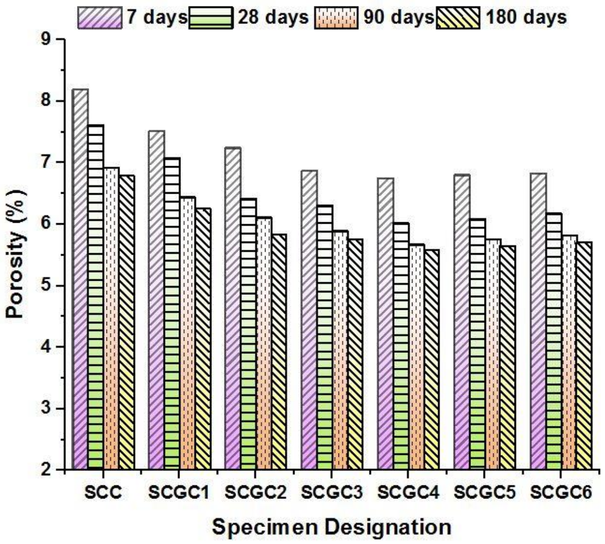

Fig. 6 - Porosity of SCC mixes at various ages.

6.8. Sorptivity

The capillary water absorption is mainly an inherent surface property and is dependent on the capillary pores present in the concrete. The capillary uptake of water was reduced in the concrete due to the effect of calcined zeolite inclusion indicating the thermal effect that modified the pore structure of zeolite thereby reducing the capillary pores present in the concrete. A significant reduction in the sorptivity values were observed due to additions of calcined zeolite in combination with glass powder in the concrete as is shown in Figure 7. Glass powders are generally hydrophobic in nature and hence reduce the amounts of waters that can be absorbed through capillarity by the concrete [37]. The pozzolanic reactivity of calcined zeolite is also generally much higher which has the ability to transform CH crystals into CSH gels that possess pore plugging properties and reduces the capillary water channels.

6.9. RCPT (rapid chloride permeability test)

The self compacting concrete specimens were subjected to rapid chloride permeability test at 28 days, 90 days and 180 days and the data were shown in Figure 8. During the test, transportation of certain quantity of ions is caused by the presence of OH^- ions and they act as a comfortable electrolyte

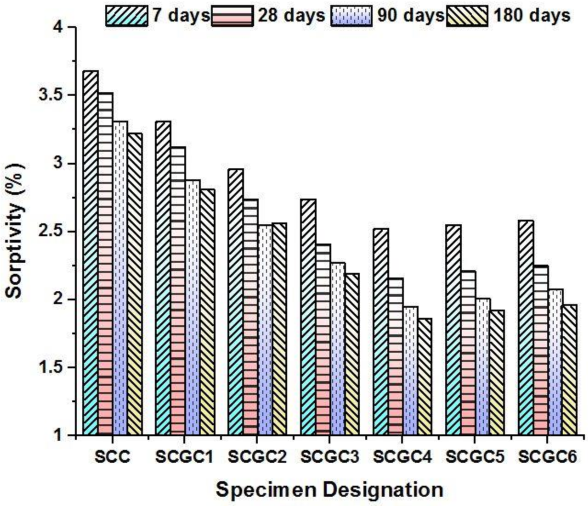

Fig. 7 - Sorptivity of SCC mixes at various ages.

for chloride. All the concretes exhibited a gradual reduction of charge passed at various ages thereby categorizing all the self compacting concretes under 'good' grade since the charge passed values were within 100-1000 coulombs [37]. The reduced passage of charge through the self compacting concrete indicates more dense concrete thus improving the resistance to chloride ions. The combined effect of calcined zeolite and glass powder has a major impact on preventing the penetration of ions through the self compacting concrete even at increased levels of replacement.

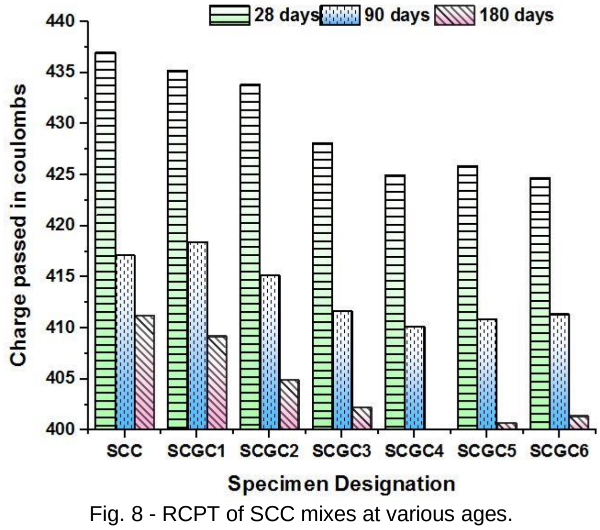

Fig. 8 - RCPT of SCC mixes at various ages.

6.10. ASR Concrete prism test

The categorization of the aggregates as per ASTM C 1260 is presented in Figure 9 based on the values of expansion obtained. It can be seen that the addition of calcined zeolite in the concrete has been found to mitigate the ASR expansion at all ages. The concretes containing calcined zeolites were found to possess minimal expansion values and they fall either under non-reactive or potentially active category. Minimal increase of expansion ratio values were found for the self compacting concretes with increasing

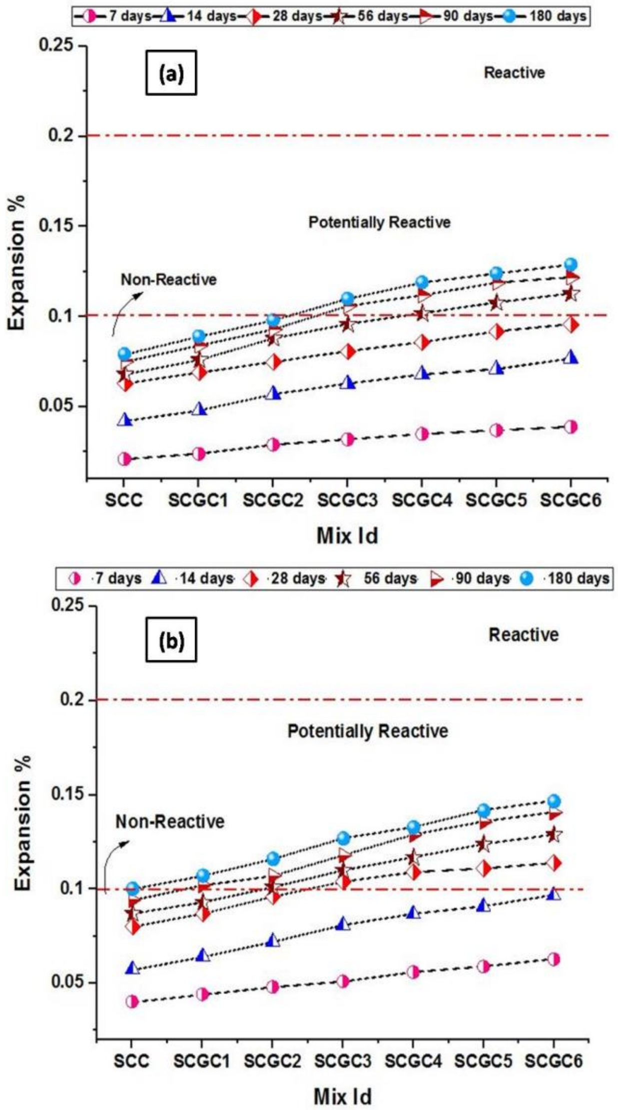

Fig. 9 - Expansion percentages of (a) SCC mixes at various ages (b) NaOH conditioned SCC mixes at various ages.

glass powder content. The expansion ratio for concrete containing 45% glass powder and 30 % calcined zeolite was 0.12% at normal curing conditions and after subjected to NaOH conditioning it was found to be 0.15% after 180 days. NaOH conditioned concretes also showed enhanced resistance to expansion due to the pozzolanic action of calcined zeolite. This zeolite can form additional CSH gels including alkalis of the concrete. The reduction of the alkali in pore solution of the concrete increases the durability of concrete by effectively suppressing the ASR expansion.

7. Conclusions

The main highlights of the conclusions that can be arrived from the experiments conducted on the self compacting concretes containing calcined zeolite as cement replacement and glass powder as fine aggregate replacement can be stated as follows:

- The influence of calcined zeolite admixtures was complemented by the addition of glass powder which resulted in increased slump values due to their hydrophobic nature and glassy surface texture. The increased specific surface of zeolite showed reduced availability of free water. Thus the combination of zeolite and glass powder in the self compacting concretes helps in improvement of the workability of the self compacting concrete.
- Zeolite with the pozzolanic glass powder contributed to the strength increment for the self compacting concretes. Dense microstructure of the self compacting concrete is obtained by the calcined zeolite and glass powder and its filling ability has resulted in an increased compressive strength.
- The self compacting concrete containing glass powder and zeolite showed higher flexural strength than control concrete even at high percentage substitutions. The addition of glass powder and calcined zeolite acted as significant filler in self compacting concretes. The brittleness caused by the glass powder has been eliminated by the calcined zeolite and has resulted in improved strength.
- Lesser values of water absorption, porosity and sorptivity and expansion ratio have been recorded when the self compacting concrete containing glass powder and calcined zeolite is compared to the control at all ages. Calcined zeolite tends to have the properties of filling pores and the fine glass powder has inter granular filling that shows lesser pores and that causes the self compacting concrete to be dense.

Thus it can be finally concluded that the use of glass powder partially substituting the fine aggregate and cement partially replaced with calcined zeolite showed a positive influence on the strength and the durability properties.

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