

Sustainable self compacting acid and sulphate resistance RAC by two stage mixing approaches

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Abstract. In this research article, acid resistance, sulphate resistance and sorptivity of self compacted concrete (SCC) prepared from C&D waste have been discussed. To improve the above properties of self compacted recycled aggregate concrete (SCRAC) along with mechanical and durability properties, different two stage mixing approaches (TSMA and TSMA_{sfc}) were followed. In the proposed two stage mixing approach (TSMA_{sfc}), silica fume, a proportional amount of cement and a proportional amount of water were mixed in premix stage which fills the pores and cracks of recycled aggregate concrete (RAC). The concrete specimen prepared using above mixing approaches were immersed in 1% concentration of sulphuric acid (H₂SO₄) and magnesium sulphate (MgSO₄) solution for 28, 90 and 180 days for evaluating the acid resistance of SCRAC. Experimental results concluded that the proposed two stage mixing approach (TSMA_{sfc}) is most suitable for acid resistance and sulphate resistance in terms of weight loss and strength loss due to the elimination of pores and cracks in the interfacial transition zone (ITZ). In modified two stage mixing approach, the pores and cracks of recycled concrete aggregate (RCA) were filled up and make ITZs of SCRAC stronger. Microstructure analysis was carried out to justify the reason of improvement of ITZs by electron probe micro analyser (EPMA) analysis. X-ray mapping was also done to know the presence of strength contributing elements presents in the concrete sample. It was established that SCRAC with modified mixing approach have shown improved results in terms of acid resistance, sulphate resistance, sorptivity and mechanical properties.

Keywords: recycled aggregate concrete; two stage mixing approach; interfacial transition zone; Sorptivity test; acid resistance of concrete

1. Introduction

For the development of sustainable infrastructures by recycling crushed concrete into aggregates not only implies conservation of materials but also can be used as an alternative of natural aggregates to a large extent (Yaragal and Roshan 2017). The vital dissimilarity between recycled and natural aggregate is the adherence of mortar which is highly porous in nature and is favourably susceptible to chemical attacks from external ions. Though, owing to its more porous nature, recycled aggregate concrete (RAC) is weak in terms of mechanical and durability properties (Verma and Ashish 2017). However, with the use of proper additional cementitious materials, admixtures and adopting advanced mixing approach, the mechanical and durability properties of RAC can be enhanced (Saha and Rajasekaran 2016, Yaragal *et al.* 2016, Mukharjee and Barai 2015, Rajhans *et al.* 2018 a, b, Wijayasundara *et al.* 2018, Kisku *et al.* 2017, Kapoor *et al.* 2016). The sustainability of concrete production is enhanced by producing self-compacting concrete (SCC) which embodies a satisfactory level of behavioural characteristics like filling ability, passing ability and resistance to segregation (Dinakar *et al.* 2008, Aslani *et al.* 2018, Dinakar *et al.* 2013, Boudali *et al.*

2016).

It has been reported in many literatures (Karakurt and Topcu 2011, Al-Salami and Salem 2010, Dehwah 2007, Sahmaran *et al.* 2007, Santhanam *et al.* 2006, Bellmann *et al.* 2006, Chindaprasirt *et al.* 2004, Santhanam *et al.* 2002) that the concrete got deteriorated due to the attack of sulphate ions. The reduction in strength of concrete structures occurs due to exposure of sulphate ion resources such as soils, groundwater, seawater, river water and due to wastes of industries that contains a large concentration of sulphate ions. The sulphate containing salts reacts with the cement/concrete and converts the hydration products into gypsum, ettringite and thaumasite. Further affects the primary strength-providing product calcium silicate hydrate (C-S-H) gel to cause decalcification and destabilization. In addition, acidic environments such as sewage system, chemical, paper industries and thermal power plant are required to convey a large amount of acidic and alkaline solutions from one point to other with the use of conveyance concrete pipe. Thus, the strength of such conveyance concrete pipes is utmost importance because the reason of deterioration of concrete pipes to the exposure against the attacks of acid and their fumes. The sulphur dioxide that is let out in the atmosphere due to industrial emission attacks the structural concrete and degrades the concrete strength. Sulphuric acid is also more vigorous and highly reactive with the free lime (Ca(OH)₂) available in the concrete and this converts to gypsum (CaSO₄.2H₂O). The hydrated product C₃A available in concrete readily reacts

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with the gypsum producing the expansive product i.e., ettringite that increases the volume of concrete causing internal pressure to weight loss and leads to the formation of cracks in the concrete. The acid resistance of concrete can be improved by careful adaptation of mix design and the mixing approach with the employment of admixtures and cementitious materials. From the studies (Freidin 1999, Torii and Kawamura 1994, Mehta and Siddique 2017) it is understood that with the use of mineral admixtures, the resistivity to acid attack is enhanced and the effective performance is obtained by the addition of silica fume.

Bulatović *et al.* (2017) investigated the results of sulphate resistance on the concrete of RCA/normal concrete aggregate (NCA). Concrete samples were immersed in 5% Na₂SO₄ or 5% MgSO₄ for 90, 180 and 365 days. Sulphate resistance was evaluated by determining the compressive strength loss and length change. Mehta and Siddique (2017) examined the sulphuric acid resistance of fly ash based geopolymer concrete in which ordinary portland cement (OPC) was added as an additional calcium source. The concrete specimens were immersed in 2% sulphuric acid solution up to the age of one year and the effects of sulphuric acid on concrete specimen was measured in terms of mass loss and compressive strength loss. It was concluded that the addition of OPC in concrete resulted in improved compressive strength but it did not show a similar effect on its resistance to sulphuric acid. Gamal *et al.* (2017) studied the carbonation depth measurements and the corrosion of portland cement concrete (PCC), sulphur resistance cement concrete (SRC) and modified sulphur concrete (MSC). Results showed that high resistivity was observed in MSC to the sewerage environment over PCC and SRC. Acharya and Patro (2016) investigated various properties such as workability, fresh density, compressive strength, flexural strength, bond strength, acid resistance, sulphate resistance and sorptivity of concrete with the potential of ferrochrome ash along with lime as a partial substitute material of cement. Aydin *et al.* (2007) investigated the effect on the mechanical properties and sulphuric acid resistance of concrete by incorporating ASTM class C fly ash. Test results were improved for the sulphuric acid resistance of steam cured concrete by introducing fly ash. Alfi *et al.* (2004) studied the characteristics of sulphate resistance cement paste using limestone and silica fume curing for 90 days. They concluded that the mechanical properties and porosity of cement with the addition of limestone and silica fume was improved. Roy *et al.* (2001) investigated the effects of chemical environment on the concrete prepared with ordinary portland cement and various replacement percentages of silica fume (SF), metakaolin (MK) and fly ash (FA). It was observed that under certain circumstances, the addition of SF, MK and FA, improved the acid resistance of concrete.

Many investigations (Choi *et al.* 2016, Elhakam *et al.* 2012, Tam *et al.* 2005, Tam and Tam 2008, Liang *et al.* 2013, Li *et al.* 2009) were carried out to improve the mechanical and durability properties of concrete by using different mixing approaches. Tam *et al.* (2005) firstly proposed a new mixing approach namely two stage mixing approach (TSMA) in which water was divided into two

stages. It was observed that TSMA resulted better compressive strength than that of normal mixing approach (NMA). Tam and Tam (2008) further optimized TSMA with the use of silica fume and cement at premix stage. It was found that the addition of silica fume and cement at premix stage improves the interfacial transition zone (ITZ) and resulting increase in the strength of RAC. Liang *et al.* (2013) proposed two mixing method i.e., mortar mixing approach (MMA) and sand enveloped mixing approach (SEMA). By using these proposed two mixing methods the harden properties of RAC were improved. Li *et al.* (2009) developed a new technique in which the surface of RCA was coated with pozzolanic powder (fly ash, silica fume and blast furnace slag) which results in better quality of RCA resulting improved properties of RAC.

Literature study reveals that there is a significant improvement in different properties of recycled aggregate concrete by using advanced mixing approaches (Rajhans *et al.* 2018 a, b). However, the acid resistance of recycled aggregate concrete prepared using two stage mixing approaches are rarely available in open literature. In this context, the present study is on evaluation of different properties of SCRAC along with acid resistance and sorptivity. The sulphuric acid environment in concrete is developed in different applications like sewage, mining, chemical and paper industries etc. The magnesium sulphate environment is created when the structure is exposed to external sulphate attack comes from soils, groundwater, and sea water, etc. The sulphuric acid resistance and sulphate resistance of SCRAC using different replacement percentage of RCA were considered and this was experimented by immersing in 1% concentration of H₂SO₄ or MgSO₄ solution up to 28, 90 and 180 days. The parameters like strength loss and mass loss were calculated to observe the adverse effects of acids on SCRAC. Microstructure analysis was carried out to justify the reason for improvement of ITZs by electron probe micro analyser (EPMA) analysis. Elemental mapping was done to confirm the presence of strength contributing elements present in the concrete samples.

2. Materials and methods

2.1 Materials

To evaluate the influence of different parameters on acid and sulphate resistance of concrete, the following materials were used:

Cementitious material: Ordinary portland cement (OPC) of 43 grade conforming the IS: 8112- (1989), fly ash and silica fume were used as cementitious materials. The properties of cement, fly ash and silica fume are available in Rajhans *et al.* (2018 b).

Water: The potable water was used for mixing in the production of concrete. For maintaining the concentration of H₂SO₄ and MgSO₄ distilled water was used.

Admixture: AT-CARPOL 800 high range polycarboxylate ether superplasticizer was used to maintain the workability of self compacting concrete in

Table 1 Mix proportion of SCC with different percentage of RCA using Nan Su method of mix design

% of RCA	Mix designation	Cement (kg/m ³)	Water (kg/m ³)	Coarse aggregate		Fine aggregate (kg/m ³)	Fly ash (kg/m ³)	SP (kg/m ³)
				Virgin (kg/m ³)	Recycled (kg/m ³)			
R0	SCVAC	325	182	830	-	930	138	4.5
R20	SCRAC20	325	182	664	154	930	138	4.5
R40	SCRAC40	325	182	498	308	930	138	4.5
R60	SCRAC60	325	182	332	462	930	138	4.5
R100	SCRAC100	325	182	-	770	930	138	4.5

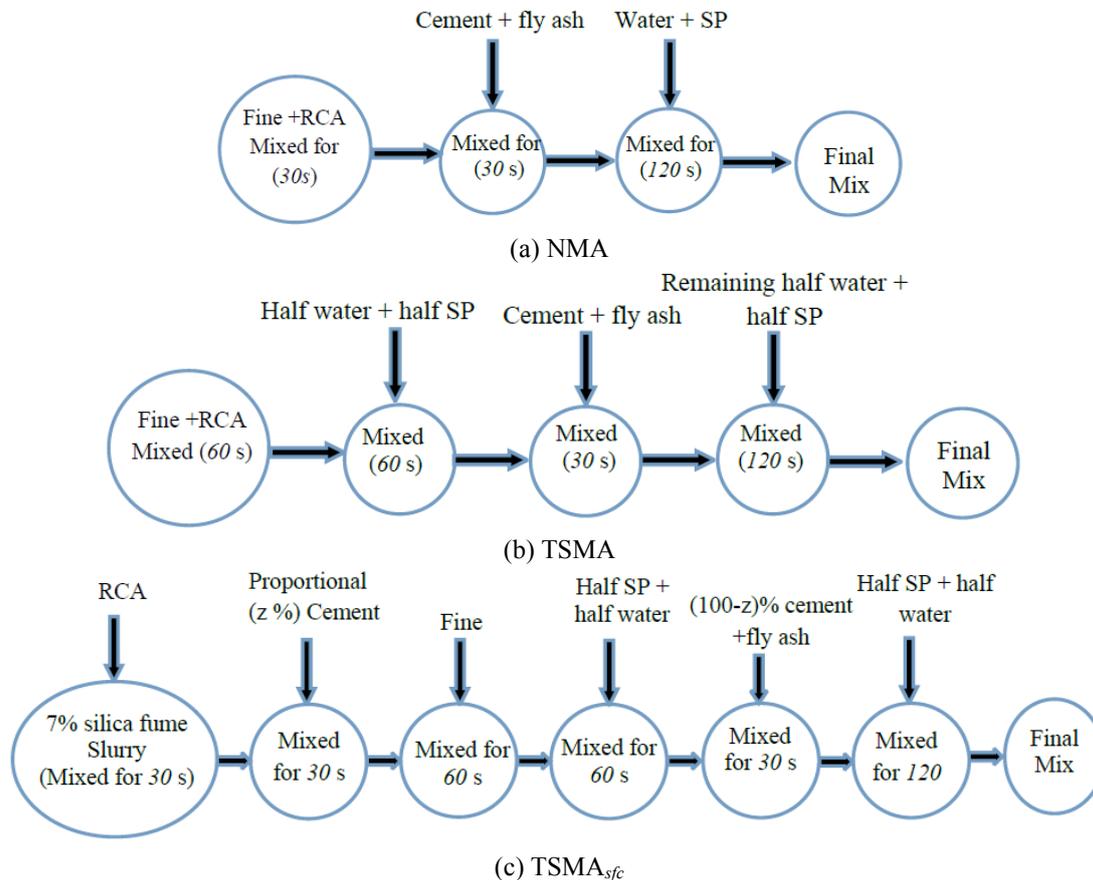


Fig. 1 Flow diagram of different mixing approaches used in present study

all concrete mixes. The superplasticizer conforms the IS: 9103 (1999) (amendment No/2 august 2007). The colour of superplasticizer is light brown and solid content is 39%.

2.2 Characterisation of aggregates

Virgin and recycled aggregates were used as coarse aggregates. Limestone was used for producing virgin aggregate and river sand was used as fine aggregates. The fineness modulus of fine aggregate is 2.45 confirming to zone II as per IS 383 (1970). The source of recycled aggregate was a demolished 30 years old building at IIT (ISM) Dhanbad, India campus. Physical and mechanical characteristics of both virgin and recycled aggregates are already reported in the previous study (Rajhans *et al.* 2018b) by the present authors. The physical properties of RCA were different from the properties of virgin concrete aggregate (VCA) because old mortar is adhered to the

surface of recycled aggregate and this attached mortar is light porous in nature. The bulk density of RCA was also lower than the VCA because of the above mentioned reason. The particles size distribution of the aggregates is available in previous study of the same author (Rajhans *et al.* 2018b). It was observed that the crushing value of RCA was higher than VCA. The crushing test and impact test were also conducted as per IS 2386 (1963) (Part IV).

2.3 Mixture proportion

The mix proportions of M30 grade of SCC have obtained by following Nan Su mix design (2001) method and the obtained proportion are presented in Table 1. Five SCC mixes were prepared with different percentage of recycled aggregate i.e., 0%, 20%, 40%, 60% and 100%. The mixes are referred as SCVAC, SCRAC-20, SCRAC-40, SCRAC-60 and SCRAC-100 for 0% RCA, 20% RCA, 40% RCA, 60% RCA and 100% RCA, respectively. All the

aggregates were kept into saturated surface dry (SSD) conditions before mixing. The proportions of all the constituents were obtained after satisfying the required fresh properties test values.

2.4 Mixing approaches

In this study, three different mixing approaches are used namely; normal mixing approach (NMA), two stage mixing approach (TSMA) and two stage mixing approach (*silica fume, fly ash and cement*) (TSMA_{sfc}).

2.4.1 Normal mixing approach (NMA)

The process of mixing was conducted by mixing coarse and fine aggregate for 30 seconds, then cement and fly ash were mixed for further 30 seconds. Lastly, for the next 120 seconds of mixing, water and SP were mixed thoroughly. The flow diagram of mixing stage is shown in Fig. 1(a).

2.4.2 Two stage mixing approach (TSMA)

In the first stage of mixing, fine and coarse were mixed for 60 seconds. After that, 50% of water and 50% of SP were poured in the mixing machine and mixed for another 60 seconds. Then cementitious materials i.e., cement and fly ash were added and mixed for another 30 seconds. Lastly, at second stage of mixing, rest of the water was added with SP and mixed for another 120 seconds. The flow diagram is shown in Fig. 1(b).

2.4.3 Two stage mixing approach (*silica fume, fly ash and cement*) (TSMA_{sfc})

The proposed mixing approach is a modified two stage mixing approach (TSMA_{sfc}). Initially, the fresh properties of SCRAC were investigated experimentally by partially replacing fly ash with a certain percentage of silica fume. The fresh properties of SCRAC were evaluated through conducting several trials by replacing 2%, 4%, 6%, 7%, and 8% of fly ash with silica fume. The optimum percentage of silica fume was found to be 7% after performing the workability tests for above percentage of replacement of fly ash with silica fume. In the premix stage of TSMA_{sfc}, the obtained optimum percentage of silica fume which was 7% was added to the proportional amount of water, cement and the percentage of RCA to make silica fume-cement slurry. Fine aggregate, fly ash, SP, remaining cement and rest of the water were added according to TSMA. The complete stages of modified two stage mixing approach are presented in Fig. 1(c).

2.5 Test specimen

Cubes of size 150 mm were prepared for determining the compressive strength, acid resistance, and sulphate resistance. Cylinders of 100 mm diameter and 200 mm long were prepared, and specimens of 100 mm diameter with 50 mm thick were saw cut for the test of sorptivity.

2.6 Curing procedure

The specimens were covered with plastic sheets after casting in a mould and kept for 24 h at room temperature of



Fig. 2 Concrete cubes prepared by using different mixing approaches immersed in 1% of sulphuric acid solution for curing

27°C. After 24 hours of casting, the specimens were taken out from mould and immersed in water tank at 27°C±2°C temperature for curing till the testing at different ages.

2.7 Fresh properties test

To attain the fresh properties of SCC, different tests were performed as per EN standards (2002) guidelines. The tests i.e., slump flow test, T_{500} , V -funnel and J -ring were conducted for various replacement percentage of RCA, for each of the three mixing approaches.

2.8 Acid resistance test procedure

Acid resistance test was conducted by immersing the 28 days water cured cubes in 1% concentration solution of sulphuric acid (H_2SO_4) as shown in Fig. 2. The test specimen was immersed in 1% percentage of the sulphuric acid solution (pH=1) because this concentration of sulphuric acid is associated with the concentration of sulphuric acid that found in sewers in the process of deterioration (Meyer and Ledbetter 1970). After the curing of certain days in sulphuric acid, the cubes were weighed and the measured weight was compared with the same before immersion for evaluating the weight loss. The weight loss (WL) of acid cured cubes was the difference in the weight of control specimen and the weight of acid cured cubes of same age and same mix. Similarly, the strength losses of cube were evaluated after following the same process as above. The percentage of strength loss (SL) is expressed as given below.

$$SL(\%) = \frac{f_{c1} - f_{c2}}{f_{c1}} \times 100 \quad (1)$$

where, f_{c1} represents 28 days compressive strength of control specimens and f_{c2} is the compressive strength of the specimen after immersing in 1% concentration of sulphuric acid (H_2SO_4) solution for 28 days. The percentage of weight loss (WL) is expressed as

$$WL(\%) = \frac{w_1 - w_2}{w_1} \times 100 \quad (2)$$

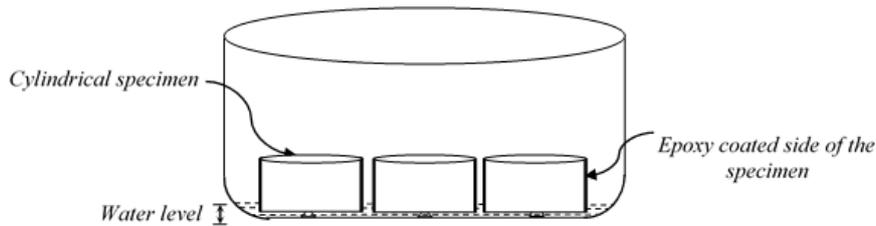


Fig. 3 General arrangements of sorptivity test set up for concrete samples

where, w_1 and w_2 are the weight of the specimens (in grams) before and after 28 days of immersion, respectively.

2.9 Sulphate resistance test procedure

In sulphate resistance test, magnesium sulphate ($MgSO_4$) was used to measure the strength loss and weight loss of concrete. Magnesium sulphate is more hazardous than other sulphate and showed more adverse effect on cement paste than other sulphate (Torii and Kawamura 1994). Magnesium sulphate reacts with calcium hydroxide and hydrated calcium silicates which makes concrete weaker. The effect of 1% concentration of magnesium sulphate on concrete was examined by immersing the sample on magnesium sulphate solution after 28 days of curing. The weight loss and strength loss were calculated in same manner as performed for acid resistance test.

2.10 Sorptivity test procedure

Sorptivity test for SCRAC was performed to measure entrapped water into the pores due to the capillary action. A sample of 100 mm diameter and 50 mm thick concrete disc sample were taken from a standard cylinder of 100 mm diameter and 200 mm length. The side face of the specimen was covered by epoxy to prevent the entry of water from side faces. The specimens were kept in oven at a temperature of $105 \pm 5^\circ C$ for 24 hours and then the specimens were taken out, and cooled at room temperature. The initial weight of the sample was weighed at the atmospheric temperature. The sample was placed in water filled tray in such a manner that only the bottom 5 mm of the specimen was under water as per ASTM C 1585-11 (2004) guidelines and the general arrangement of sorptivity set up is illustrated in Fig. 3. The weight of the specimen was taken at the time interval of 9, 16, 25, 36, 49, 64, 81, 100, 121, 144, 169, 196, 225, 256, 289, 324 and 361 minutes. The difference in the initial weight of the sample and weight of the sample after water absorption is noted as water absorption or sorptivity. The relation between sorptivity and the time is defined as given below.

$$i = s\sqrt{t} \quad (3)$$

where, i = The cumulative water absorption per unit area of inflow surface i.e., g/mm^2 ; t = time in minutes; s = Sorptivity of concrete obtained from a linear regression of ' i ' versus square root of time i.e., $(10^{-4} g/mm^2/min^{1/2})$.

2.11 Electron probe micro analyser (EPMA) for microstructural studies

Microstructural analysis was carried on backscattered secondary electron (BSE) images in order to evaluate the quality of interfacial transition zone (ITZ) of the concrete. This BSE images were taken by using electron probe micro analyser (EPMA). In this image, brighter regions represent the compounds having higher mean atomic number and those having lesser mean atomic number are characterised as darker. Therefore, BSE images were used to understand the hydrated (C-S-H) gel and unhydrated (CH) parts of cement mortar as unhydrated products appears brighter than the hydrated products, and pores appear dark in BSE image (Kjellsen *et al.* 2003, Sahu *et al.* 2004).

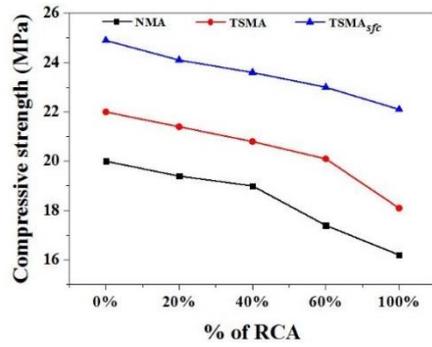
3. Results and discussion

3.1 Fresh concrete properties

Fresh properties of SCRAC using $TSMA_{sfc}$ with 100% RCA are evaluated by V -funnel, slump cone and J -ring apparatus and the results are available in the research publications of the present authors (Rajhans *et al.* 2018b). In modified mixing approach $TSMA_{sfc}$, different replacement percentage of recycled aggregate are used along with the replacement of fly ash content with different percentage of silica fume i.e., 2%, 4%, 6%, 7% and 8%. Due to the fact that, silica fume has large specific surface area and is having very fine particle size; the concrete prepared with silica fume enhances the requirement of water. From the obtained experimental results of fresh properties, it has been established by the present investigators that upto 7% of silica fume, the fresh properties of SCRAC are within the EFNARC (2002) guidelines. The fresh properties test results (slump flow diameter, T_{500} , V -funnel and J -ring) of SCRAC with different replacement percentage of RCA using different mixing approaches (NMA, TSMA and $TSMA_{sfc}$ with 7% silica fume) are reported in previous study (Rajhans *et al.* 2018b). Slump flow diameter fulfils the EFNARC (2002) guidelines for all mixing approaches i.e., NMA, TSMA and $TSMA_{sfc}$. T_{500} is in the range of EFNARC (2002) guidelines for all mixing approaches. EFNARC (2002) recommends the maximum height difference of concrete outside and inside of the J -ring is 10 mm and it is seen that the value of J -ring is in the permissible range of guidelines. The value of V -funnel is also in the range of EFNARC (2002).

3.2 Hardened concrete properties

Compressive strength: Compressive strength of concrete



$$NMA: y = -0.0399x + 20.154$$

$$R^2 = 0.9639$$

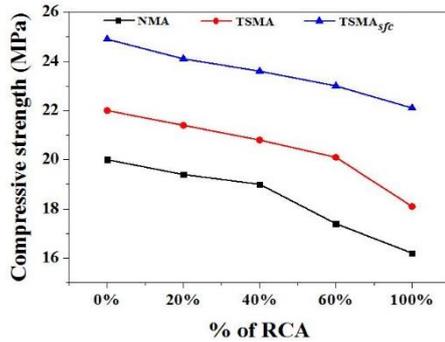
$$TSMA: y = -0.0388x + 22.186$$

$$R^2 = 0.982$$

$$TSMA_{sfc}: y = -0.0275x + 24.75$$

$$R^2 = 0.9879$$

(a) 7 days



$$NMA: y = -0.0932x + 25.203$$

$$R^2 = 0.9664$$

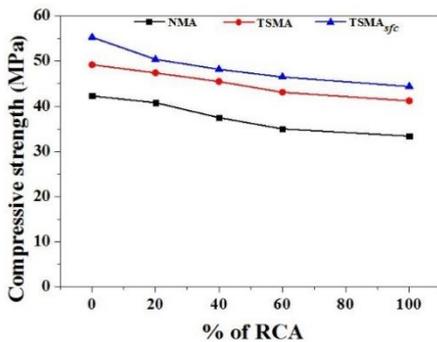
$$TSMA: y = -0.1158x + 27.976$$

$$R^2 = 0.9494$$

$$TSMA_{sfc}: y = -0.1112x + 33.214$$

$$R^2 = 0.9862$$

(b) 28 days



$$NMA: y = -0.0924x + 43.804$$

$$R^2 = 0.9368$$

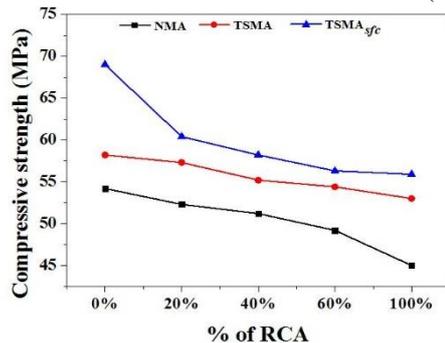
$$TSMA: y = -0.0824x + 48.904$$

$$R^2 = 0.9722$$

$$TSMA_{sfc}: y = -0.1022x + 53.458$$

$$R^2 = 0.8878$$

(c) 90 days



$$NMA: y = -0.0908x + 54.376$$

$$R^2 = 0.9905$$

$$TSMA: y = -0.0538x + 57.986$$

$$R^2 = 0.951$$

$$TSMA_{sfc}: y = -0.0856x + 62.907$$

$$R^2 = 0.8003$$

(d) 180 days

Fig. 4 Compressive strength of SCRAC for different mixing approaches at different curing ages

with various replacement percentage of VCA with RCA pertaining to mixes SCVAC, SCRAC-20, SCRAC-40, SCRAC-60, and SCRAC-100 using different mixing approaches (NMA, TSMA and TSMA_{sfc}) at an interval of 7, 28, 90, and 180 days are presented in Fig. 4. It is observed that the improvement in compressive strength is noticed with the use of proposed mixing approaches. The compressive strength of concrete prepared with TSMA is

improved because in this mixing approach, the cement and half water were mixed in the first stage and makes cement slurry which fills the pores and cracks present in the surface of RCA (Tam *et al.* 2005). The old mortar adhered on the RCA has lots of pores and cracks, and these pores and cracks make old ITZ weaker. Compressive strength depends on strengthening of ITZ and this ITZ gets stronger after filling the pores, and cracks. In TSMA_{sfc}, a silica fume-

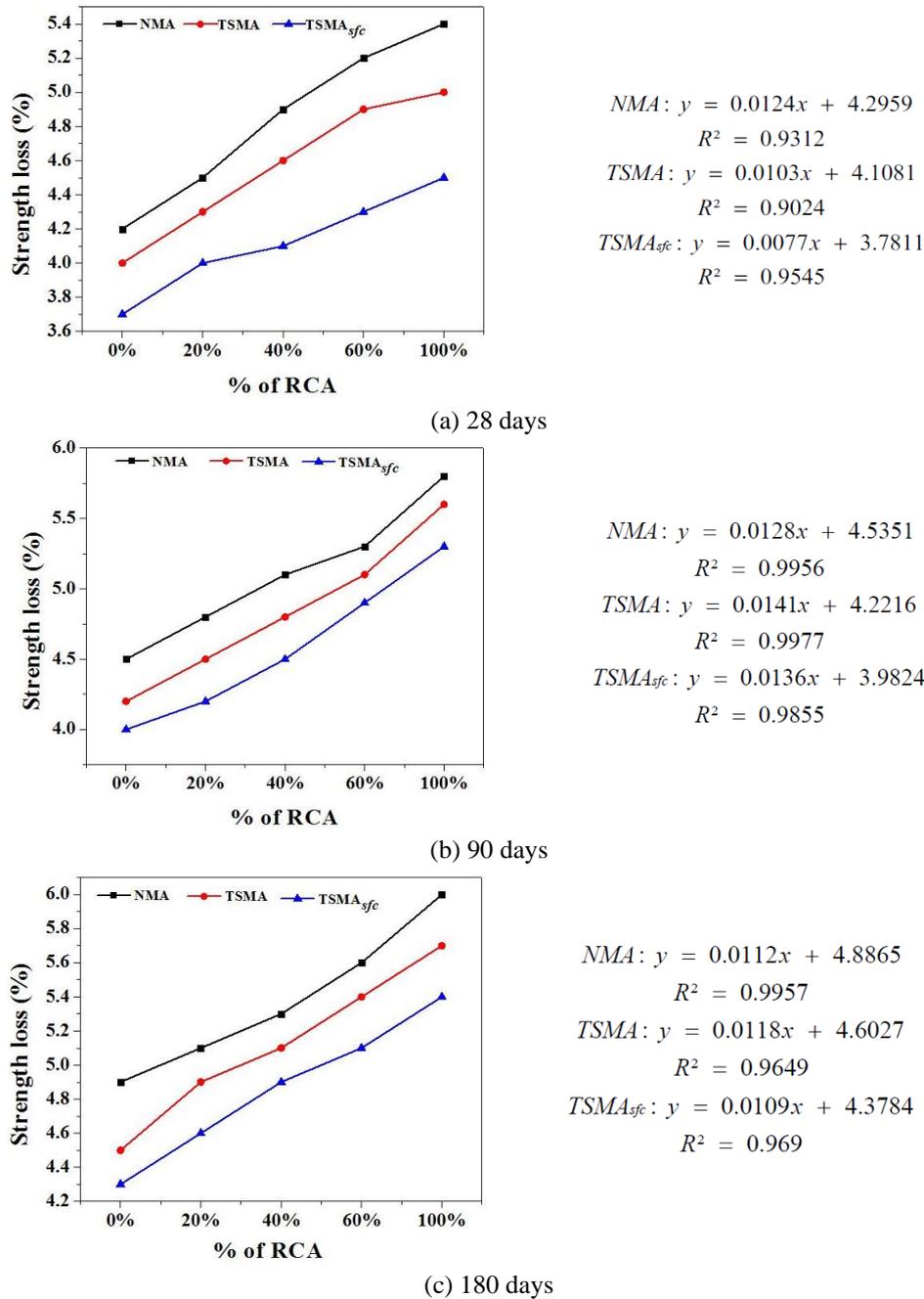
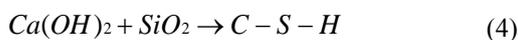


Fig. 5 Strength loss of concrete samples immersed in 1% concentration of sulphuric acid for different curing ages with various replacement percentage of RCA content

cement slurry is formed which fills the pores and cracks as silica fume and cement are mixed in the premix stage of mixing. Silica fume has very high amorphous and extreme fine silicon dioxide content. Silica fume is very reactive pozzolanic material. When portland cement in concrete reacts with water, calcium hydroxide is formed. The silica presents in silica fume further reacts with calcium hydroxide to form additional binder material called calcium silicate hydrate. The chemical reaction is as given below.



This calcium silicate hydrate is very similar to calcium silicate hydrate formed from hydrated cement (Siddique and

Khan 2011). This additional binder improves the mechanical properties of concrete prepared with $TSMA_{sfc}$. Concrete prepared with silica fume shows better mechanical properties because silica fume acts as filler due to its extreme fine particle size which fills the pores. Improved compressive strength for 100% replacement of RCA in SCRAC using TSMA is 17.64% at the age of 28 days in comparison to 100% replacement of RCA in SCRAC using NMA. Similarly, the compressive strength is increased with proposed mixing approach $TSMA_{sfc}$ at 28 days of curing for SCRAC-100 is 39.07% in comparison to SCRAC-100 using NMA. Concrete prepared with $TSMA_{sfc}$ mixing approach gives better compressive strength results. All the

experiments result are analysed for all cases and a perfect statistical correlation is formed as the value of R^2 is close to 1 in most of the cases. It is also perceived from Fig. 4 that at the age of 180 days the concrete prepared with all three mixing approaches show that the difference in compressive strength is reduced than that of the concrete at the age of 28 and 56 days. The compressive strength of concrete prepared with SCRAC mixes increases with increase in curing days for all the mixing approaches. This enhancement of compressive strength is observed due to the involvement of fly ash and silica fume. Silica fume and fly ash both is the pozzolanic material and undergo pozzolanic reactions at later stages (Cohen and Mather 1991).

3.3 Acid resistance

Acid resistance of SCRAC is observed in terms of strength loss and weight loss having different percentages of RCA. For evaluating the acid resistance of SCRAC, concrete cubes were prepared using different mixing approaches and were immersed in 1% concentration of sulphuric acid solution for 28 days, 90 days and 180 days as discussed earlier.

Strength loss due to sulphuric acid attack: Sulphuric acid was originated by sulphate-reducing bacteria (SRB) and micro-organisms which were presented in the sludge layer of sewer pipes. These bacteria took sulphates as a source of oxygen and released sulphide, this sulphide reacted with dissolved hydrogen presented in wastewater to form dissolved hydrogen sulphide and hydrogen sulphide ions. This dissolved hydrogen sulphide was evaporated and discharged through the sewer pipes, and oxidised into sulphuric acid by the action of aerobic bacteria (*Thiobacillus*) that was present in the apex of sewer pipes. This sulphuric acid reacted with the hydration products of concrete matrix. With this reaction, two compounds were formed i.e., gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) and ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$). The chemical reactions are as given below.



Due to the larger volume of gypsum and ettringite, expansion of volume took place resulted internal pressure, crack formation, weakening of the concrete wall aggregate and thinning of the concrete pipes. This leads to the concrete pipe losses its mechanical strength. For evaluating the strength loss of SCRAC for above sulphuric acid environment, the strength of concrete samples of different mixing approaches are determined after immersing in 1% concentration of sulphuric acid solution for 28, 90 and 180 days and the results are presented in Fig. 5. It is observed that the strength is negatively affected with the increase in percentage of RCA. The strength loss of concrete is accounted due to the old mortar paste attached on the surface of RCA. This attached mortar paste has lots of minute cracks and pore which shows high absorption property of RCA. Due to this high absorption, more

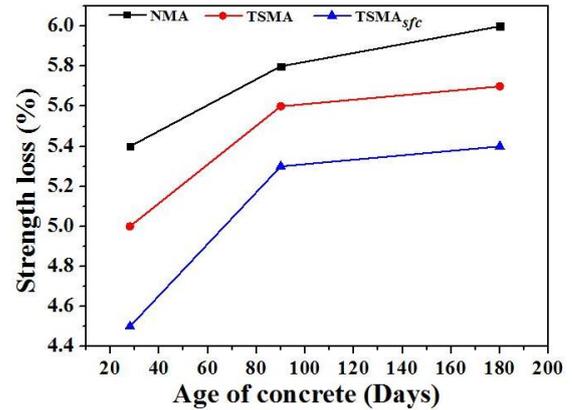
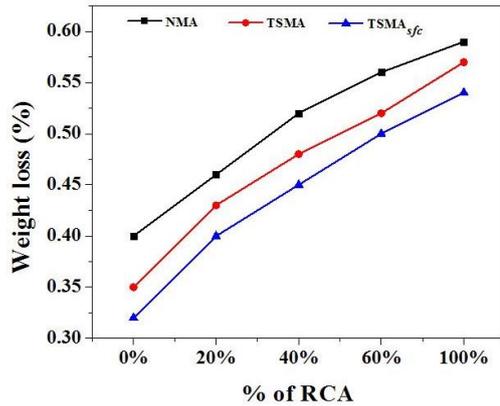


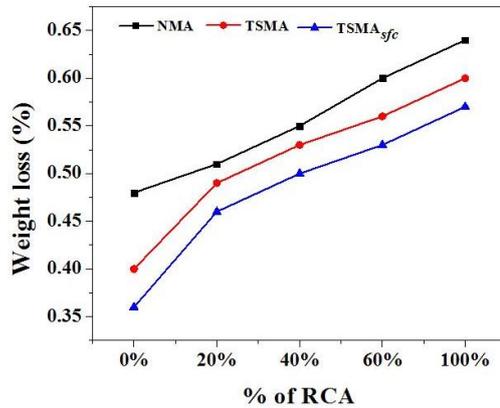
Fig. 6 Strength loss of concrete samples prepared using all three mixing approaches having 100% RCA for different curing periods

sulphuric acid is absorbed and gypsum is formed resulting loss in strength due to the weakening of ITZ. Two stage mixing approach is an attempt to reduce the cracks and pores in RCA and this also controls the loss of strength in SCRAC. From the experimental results of Fig. 5(a), it is observed that the strength loss of SCRAC with 100% RCA in acid curing (1% concentration of H_2SO_4) is 5.4%, 5% and 4.5% for NMA, TSMA and TSMA_{sfc}, respectively. It is concluded that because of TSMA and TSMA_{sfc}, there is an improvement of 7.40%, and 16%, respectively, than that of NMA. It is noticed that after 90 days and 180 days of acid curing of concrete prepared with TSMA_{sfc}, there is a loss of strength of 15% and 16%, respectively than the concrete of 28 days curing. Fig. 6 presents the loss of strength of concrete for SCRAC mix with curing age having 100% RCA and prepared with three different mixing approaches. It is noticed that rate of strength loss is noticeable during initial 90 days of curing (Bulatović *et al.* 2017). The rate of strength loss of SCRAC after 90 days of acid curing is reduced due to the later stage pozzolanic reactions between silica content and free lime available in the cement. The rate of strength loss is minimum in modified mixing approach (TSMA_{sfc}) due to the presence of silica fume and the formation of more C-S-H gel. Strength losses are observed to be minimum in the concrete prepared with TSMA_{sfc} as compared to all other mixing approaches. Data analysis is done for all the mixing approaches having different percentage of RCA and with different curing ages. The (R^2) value for the experiments result are analysed for all cases and a perfect statistical correlation is found as the value of R^2 is close to 1 in most of the cases.

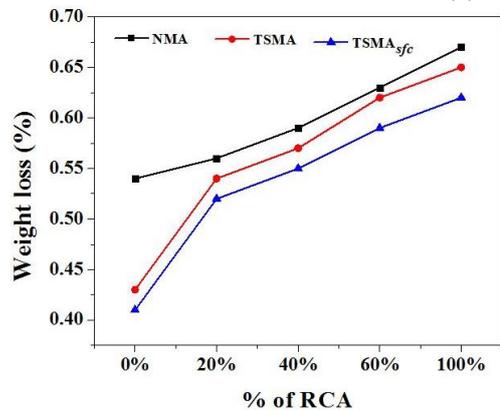
Weight loss due to sulphuric acid attack: The cubes immersed in 1% concentration of H_2SO_4 solution showed weight loss due to the reaction between sulphuric acid and free lime ($\text{Ca}(\text{OH})_2$) presented in cement paste leading to the formation of gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$). The more adverse effect was encountered when the above gypsum reacted with calcium aluminate presented in cement paste. From the reaction of these two compounds, a less soluble reacting products ettringite ($3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot 3\text{CaSO}_4 \cdot 32\text{H}_2\text{O}$) was formed. Both the above products (gypsum and ettringite) were less dense than that of the high dense hydrated



(a) 28 days



(b) 90 days



(c) 180 days

Fig. 7 Weight loss of concrete samples immersed in 1% concentration of sulphuric acid for different curing ages with various replacement percentage of RCA content

products of cement resulting softening (decrease in density) of concrete. The decreased in density of concrete leads to decrease in mass of concrete and this decrease in density of concrete was also occurred due to the sulphur acid-cement paste reaction. For evaluating the weight loss of SCRAC for above sulphuric acid environment, the weight of concrete samples of different mixing approaches are evaluated after immersing in 1% concentration of sulphuric acid solution for 28, 90 and 180 days, and the results are presented in Fig. 7. The weight loss of concrete prepared with NMA after 28 days curing is 0.44%, 0.50%, 0.55%, 0.6% and 0.635% for replacement of RCA by 0%, 20%, 40%, 60% and 100%, respectively. This weight loss is controlled after using

modified mixing approach and the minimum weight loss is observed in the case of two stage mixing approach (TSMA_{sfc}). The weight loss of concrete prepared with TSMA_{sfc} is 0.36%, 0.43%, 0.47%, 0.52% and 0.56% for replacement of RCA by 0%, 20%, 40%, 60%, and 100%, respectively. It is concluded that two stage mixing approach (TSMA_{sfc}) is least affected by sulphuric acid attack.

3.4 Sulphate resistance

Strength loss due to magnesium sulphate attack: Magnesium sulphate solution affected the cement paste more severely than other sulphate solution because of the

$$NMA: y = 0.0019x + 0.4599$$

$$R^2 = 0.9282$$

$$TSMA: y = 0.002x + 0.4146$$

$$R^2 = 0.9701$$

$$TSMA_{sfc}: y = 0.002x + 0.3815$$

$$R^2 = 0.9426$$

$$NMA: y = 0.0017x + 0.4826$$

$$R^2 = 0.9745$$

$$TSMA: y = 0.0019x + 0.4336$$

$$R^2 = 0.8893$$

$$TSMA_{sfc}: y = 0.0019x + 0.3984$$

$$R^2 = 0.8716$$

$$NMA: y = 0.0014x + 0.5382$$

$$R^2 = 0.9855$$

$$TSMA: y = 0.0021x + 0.4716$$

$$R^2 = 0.8589$$

$$TSMA_{sfc}: y = 0.0019x + 0.453$$

$$R^2 = 0.8412$$

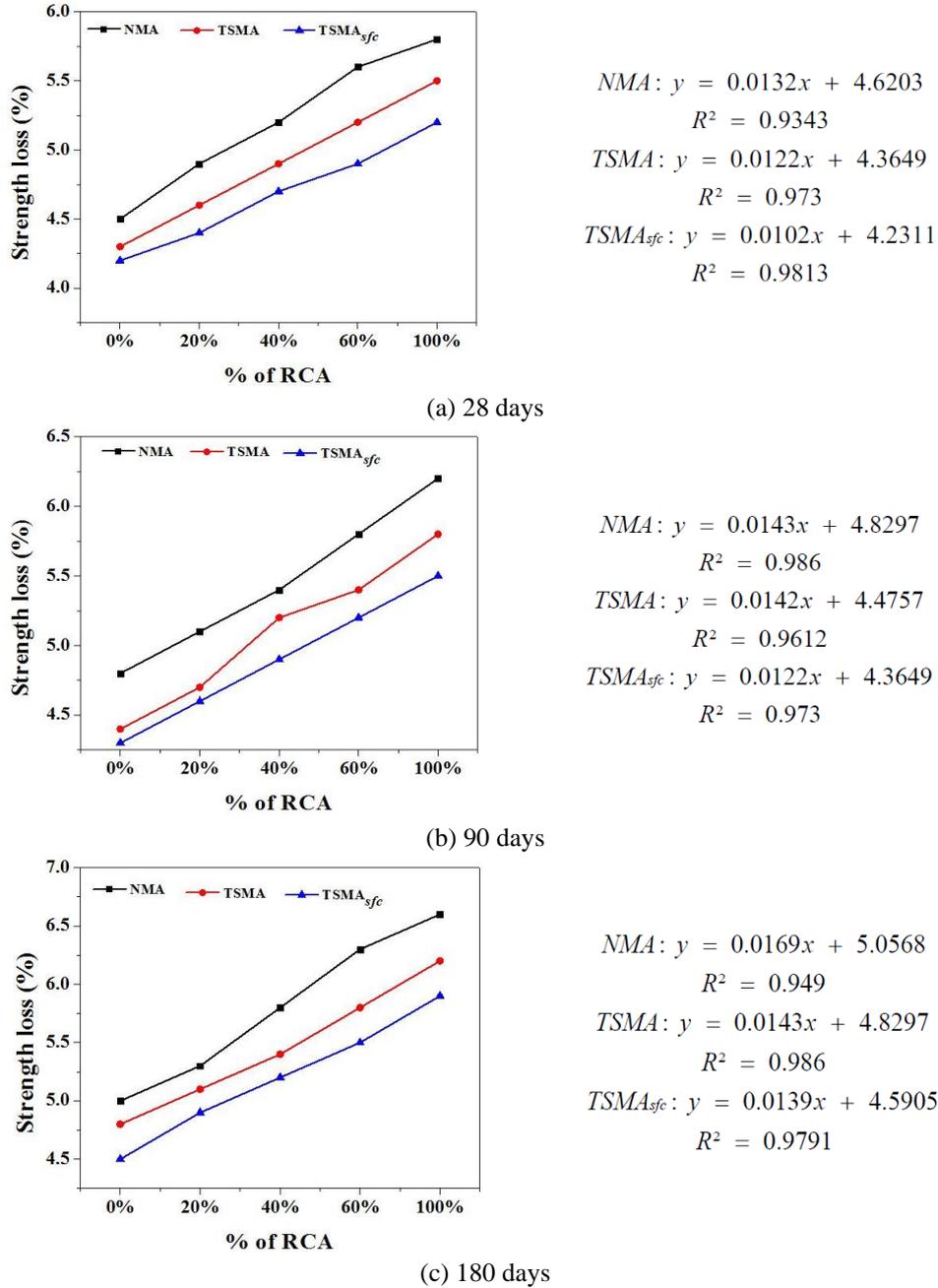
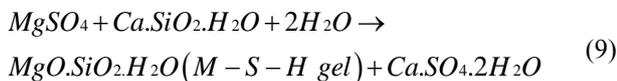
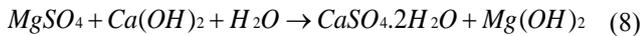


Fig. 8 Strength loss of SCRAC after cured for different days in 1% magnesium sulphate solution

formation of low dissoluble magnesium hydroxide. This magnesium hydroxide showed high aggressiveness nature against C-S-H gel. The reaction of magnesium ions with the calcium hydroxide, calcium silicates and C-S-H gel resulted magnesium hydroxide, gypsum, and ettringite, respectively. The formations of gypsum, ettringite and magnesium hydroxide make concrete weak. The chemical reactions are as given below.



For evaluating the strength loss of SCRAC for above

magnesium sulphate solution, the strength of concrete samples of different mixing approaches were evaluated after immersing in 1% concentration of magnesium sulphate solution for 28, 90, and 180 days, and the results are presented in Fig. 8. From the experimental result presented in Fig. 8(a), it is observed that the strength loss of SCRAC with 100% RCA in $MgSO_4$ curing (1% concentration of $MgSO_4$) is 5.8%, 5.5% and 5.2% for NMA, TSMA and $TSMA_{sfc}$, respectively. It is concluded that because of TSMA and $TSMA_{sfc}$ there is a reduction in strength loss by 5% and 10%, respectively, than that of NMA. Resistance to sulphate attack is better in concrete made with proposed two stage mixing approach ($TSMA_{sfc}$) than that of NMA. It is observed that the occurrence of magnesium sulphate solution severely affects the concrete

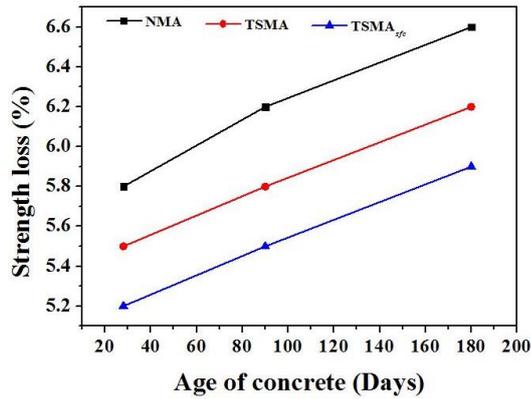
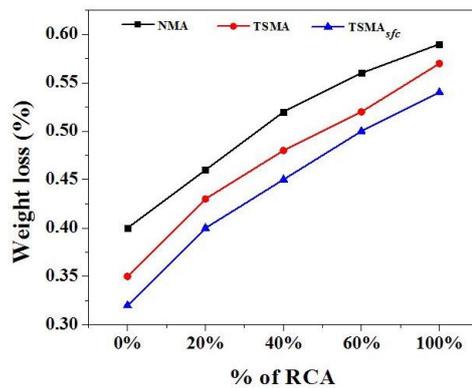
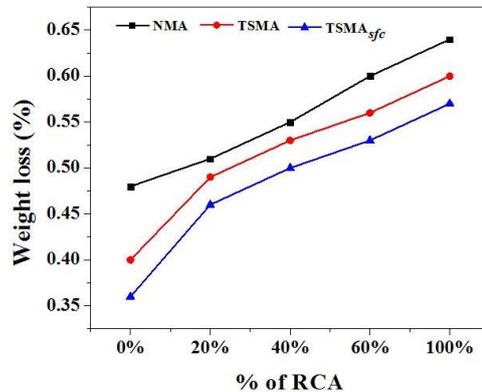


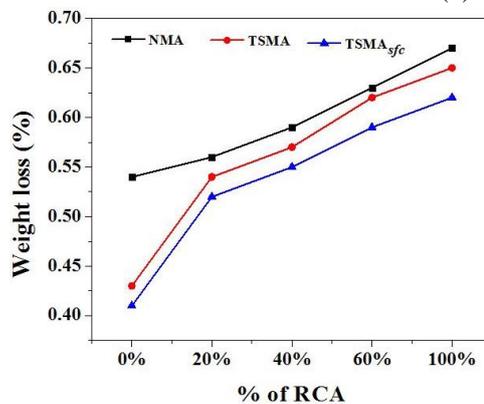
Fig. 9 Strength loss of SCRAC using different mixing approaches using 100% RCA



(a) 28 days



(b) 90 days



(c) 180 days

Fig. 10 Percentage of weight loss of SCRAC using different mixing approaches after curing for different days in 1% magnesium sulphate solution

prepared with NMA and affects less to the concrete prepared with TSMA_{sfc} because of the presence of silica fume. Previously, Wang *et al.* (2017) reported that the concrete containing fly ash and silica fume resulted in significant improvements in the resistance of concrete against the sulphate attack.

Fig. 9 presents the loss of strength of concrete of SCRAC mix with curing age having 100% RCA and prepared with three different mixing approaches. It is shown that the strength loss of concrete increases with the age of 28, 90 and 180 days because of the formation of M-S-H gel. All the experiment results are analysed and a relation is obtained between strength loss and days of curing of concrete. For all the cases a perfect statistical

$$NMA: y = 0.0019x + 0.4599$$

$$R^2 = 0.9282$$

$$TSMA: y = 0.002x + 0.4146$$

$$R^2 = 0.9701$$

$$TSMA_{sfc}: y = 0.002x + 0.3815$$

$$R^2 = 0.9426$$

$$NMA: y = 0.0017x + 0.4826$$

$$R^2 = 0.9745$$

$$TSMA: y = 0.0019x + 0.4336$$

$$R^2 = 0.8893$$

$$TSMA_{sfc}: y = 0.0019x + 0.3984$$

$$R^2 = 0.8716$$

$$NMA: y = 0.0014x + 0.5382$$

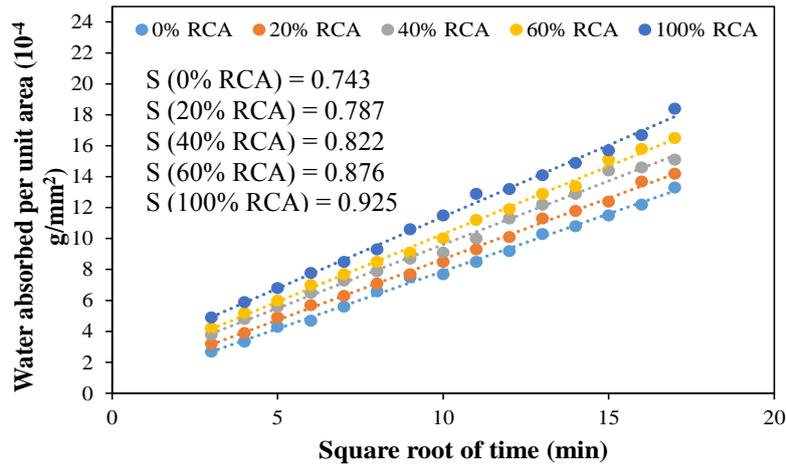
$$R^2 = 0.9855$$

$$TSMA: y = 0.0021x + 0.4716$$

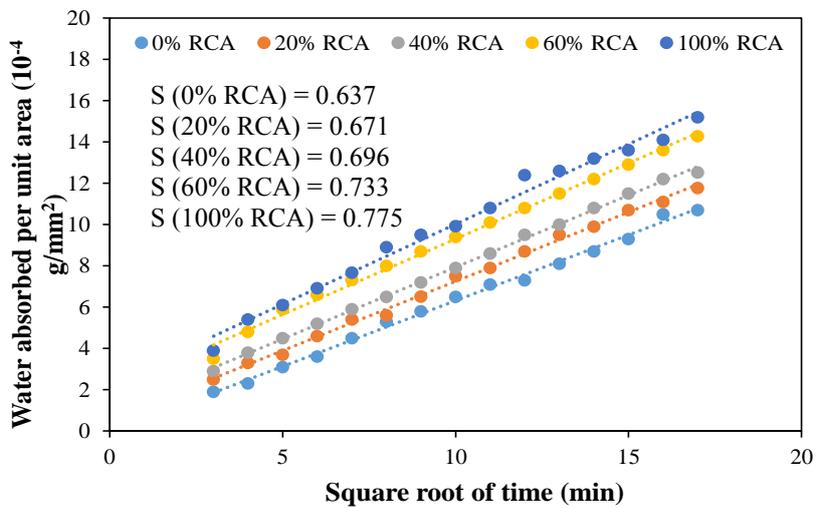
$$R^2 = 0.8589$$

$$TSMA_{sfc}: y = 0.0019x + 0.453$$

$$R^2 = 0.8412$$



(a) NMA



(b) TSMA

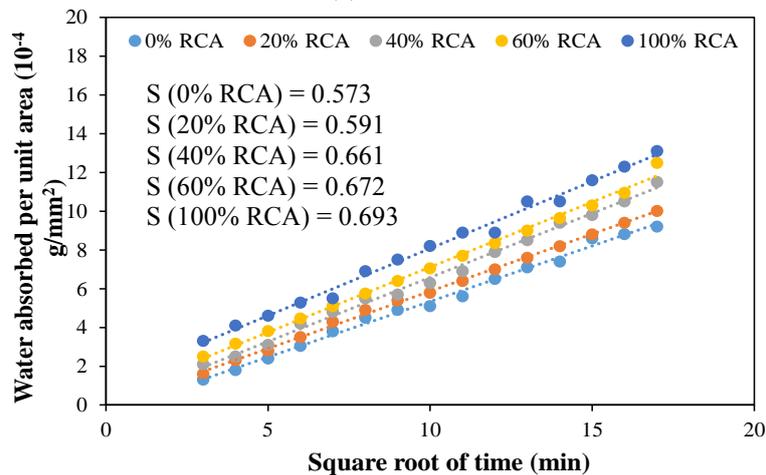
(c) TSMA_{sfc}

Fig. 11 Sorptivity test results of SCRAC using mixing approaches after 28 days of curing.

correlation is formed as the value of R^2 is close to unit.

Weight loss due to magnesium sulphate attack: Figure 10 illustrates the weight loss of SCRAC prepared using NMA, TSMA and TSMA_{sfc} with the partial replacement percentage of RCA immersed in 1% concentration of magnesium sulphate solution for 28, 90, and 180 days. It is

observed from Fig. 10 that the weight loss of concrete prepared with NMA after 28 days curing is 0.4%, 0.46%, 0.52%, 0.56% and 0.59% for replacement of RCA by 0%, 20%, 40%, 60% and 100%, respectively. Similar results were concluded by Limbachiya *et al.* (2012) when the concrete was prepared with RCA and immersed in sulphate

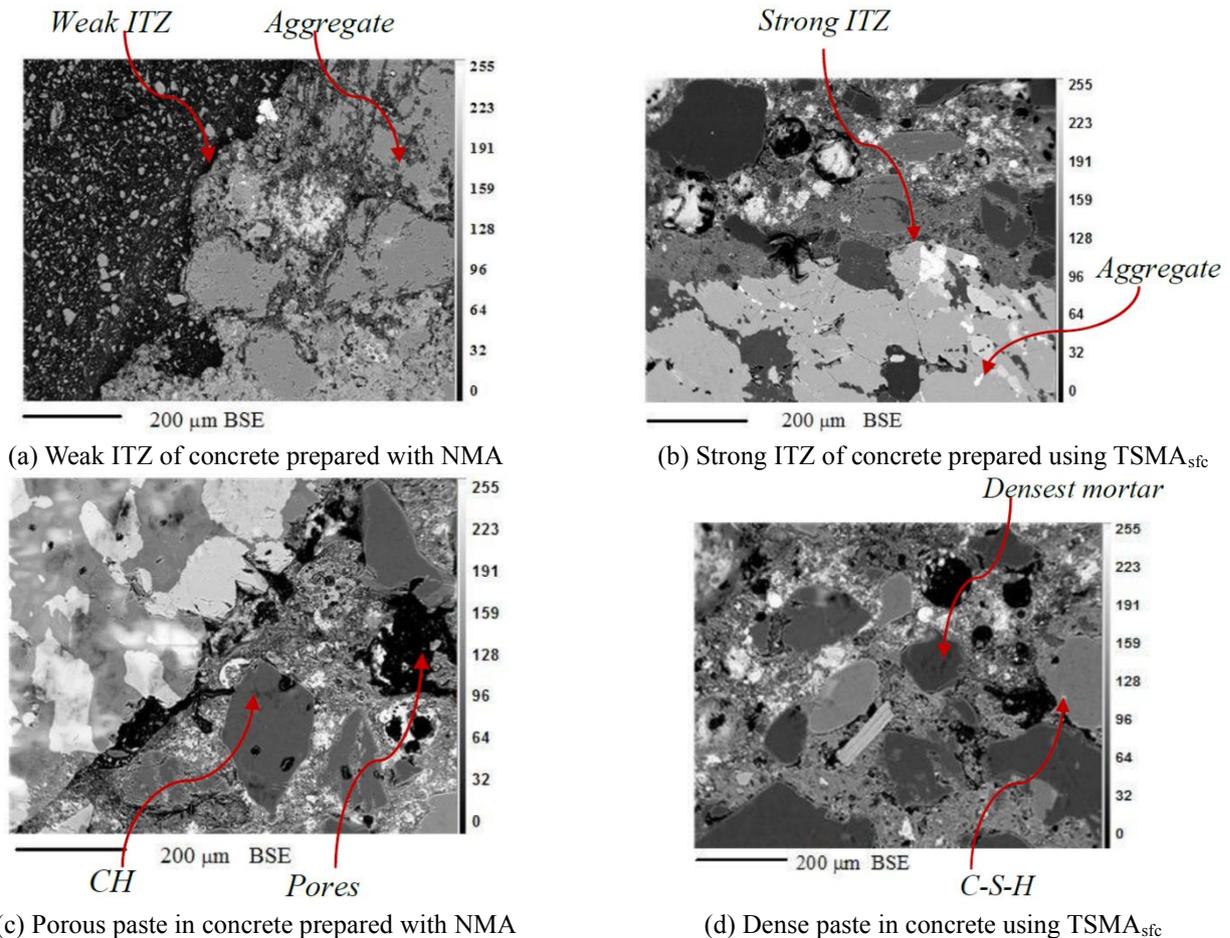


Fig. 12 EPMA of ITZ and pores of concrete samples after curing in sulphuric solution for 180 days

solution. This weight loss is controlled by using modified mixing approach and the minimum weight loss is observed in the case of two stage mixing approach (TSMA_{sfc}). The weight loss of concrete prepared with TSMA_{sfc} is 0.32%, 0.4%, 0.45%, 0.5% and 0.54% for replacement of RCA by 0%, 20%, 40%, 60%, and 100%, respectively. It is concluded that two stage mixing approach (TSMA_{sfc}) is least affected by magnesium sulphate attack.

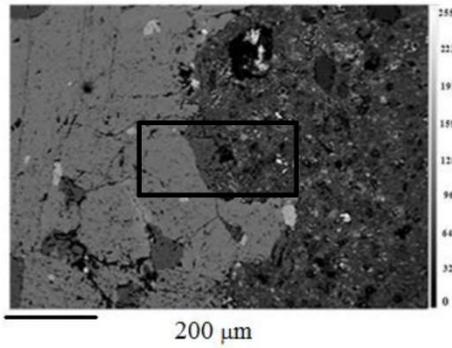
3.5 Sorptivity of SCRAC

The cumulative amount of water absorbed per unit cross sectional area of concrete specimen prepared using all three mixing approaches are presented in Fig. 11. All SCRAC mixes show an increase in ingress of water along with an increase in time and with the increase in the percentage of RCA. The ingress of water using NMA with 100% replacement of RCA shows almost the highest amount of water absorbed. In TSMA, it is seen that the water absorbed per unit area by the concrete specimen is less because the pores and cracks are controlled by two stage mixing approach. After using modified mixing approach TSMA_{sfc} the water ingress is further controlled. This is because in TSMA_{sfc} the silica fume gives a better effect on reduction of water absorption of concrete than that of fly ash. Moreover, silica fume undergoes pozzolanic reactions and this pozzolanic reaction enhances the homogeneity of SCRAC

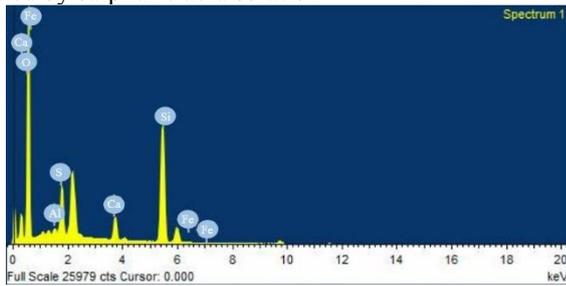
structure resulting reduction sorptivity. It is observed from Table 2 that the sorptivity (S) is minimum for TSMA_{sfc} than that of other two mixing approaches. The sorptivity (10^{-4} g/mm²/min^{1/2}) is 0.573 and 0.693 for replacement of RCA by 0% and 100%, respectively. The sorptivity (S) for TSMA_{sfc} is seen to be minimum for 0% RCA and maximum for 100% RCA. After using TSMA_{sfc}, the sorptivity is reduced by 25% in comparison to NMA for the 100% replacement of RCA.

3.6 Microstructures

The self compacting recycled aggregate concrete (SCRAC) test samples (100% replacement of RCA) are prepared by using NMA and TSMA_{sfc}. These samples are immersed in sulphuric acid solution and magnesium sulphate solution for 180 days, and the EPMA study is done for microstructural analysis. It is observed from Fig. 12 that the specimen prepared with NMA is highly permeable as a large number of voids and microcracks are presented. These microcracks further create weak links in the microstructure. However, because of the modified mixing approach i.e., TSMA_{sfc} the voids and cracks are significantly reduced. The silica fume in TSMA_{sfc} reacts with compounds of cement results calcium silicate hydrate (C-S-H). This C-S-H gel improves the strength properties significantly. The specimen immersed in the sulphuric acid solution is found to be



(a) Sample area for X-ray mapping of ITZ of concrete treated by sulphuric acid solution

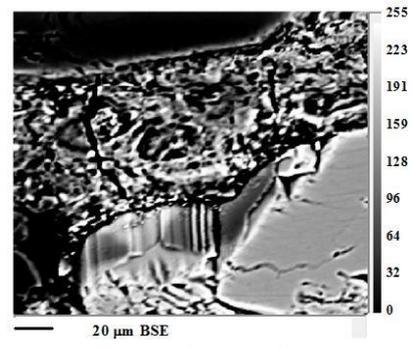


(b) X-ray mapping of the ITZ of concrete cured by sulphuric acid solution

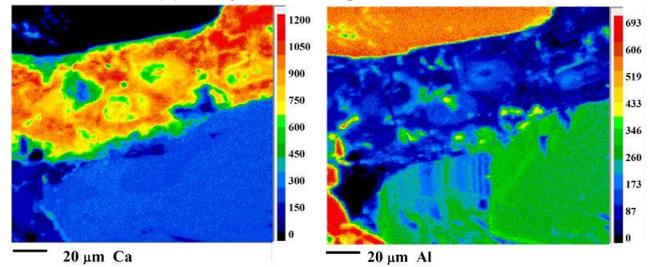
Fig. 13 X-ray mapping of concrete for analysing the compound presents in the ITZ of concrete after treated by sulphuric acid solution

deteriorated. In general, the degradation in strength due to acid exposure mainly occurred due to the reaction between calcium and sulphuric acid. The sulphur based compounds formed during this reaction are responsible for the mass loss as well as strength loss. In case of TSMA_{sf}, the strength contributing products with strong Al-O, Si-O and Ca-O bond that are less affected by acid exposure are formed. Hence, there are less cracks and pores observed in TSMA_{sf} and there is an increase in the resistance to ingress harmful ions.

Microstructural analysis also reveals that the concrete prepared with TSMA_{sf} is least affected by H₂SO₄ solution. This is because of the minimum presence of pores resulting minimum formation of CaSO₄.2H₂O. A rectangular area in ITZ of concrete sample with TSMA_{sf} is taken for X-ray mapping as shown in Fig. 13(a). It is seen from Fig. 13(b) that the X-ray mapping confirms the presence of sulphur, silica and calcium components in the spectrum of TSMA_{sf}. For confirming the above fact, a backscattered secondary electron (BSE) image is taken at the typical junction of cement paste adhered to the surface of aggregate as shown in Fig. 14(a). Fig. 14(b), (c), (d) reflect the significant presence of Ca, Si and Al, respectively, in the cement paste which fills the pores and these are the strength contributing elements. These above elements are maximum in TSMA_{sf} because of the addition of silica fume in the premix stage of concrete. However, it is observed that the sulphur content is minimum in the concrete prepared with TSMA_{sf} because the pores in concrete are already filled up by the silica fume during the two stage mixing approach as silica fume is a pore filler material.

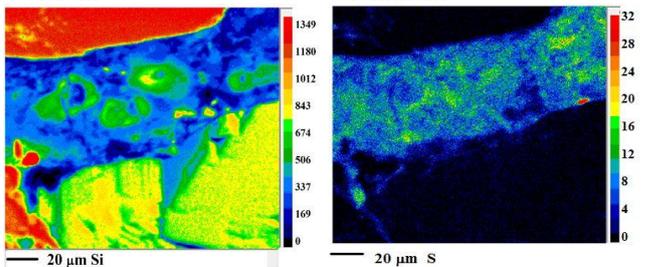


(a) Greyscale image of concrete



(b) Elemental mapping of calcium

(d) Elemental mapping of aluminum



(c) Elemental mapping of silica

(e) Elemental mapping of sulphur

Fig. 14 Elemental mapping of different elements of concrete samples

4. Conclusions

Due to the growing need of consistent development of civic amenities to sustain the growth of exponentially increasing human population, demolition of existing civil structure is taking place rampantly. Such demolitions generate a huge quantity of C&D waste which is generally dumped at open land, posing environmental threat. Concrete aggregate precipitated after recycling these waste materials can be used in the production of concrete. In this study, sustainability of self compacting concrete with reference to acid and sulphate resistance has been evaluated. For obtaining the proper mix proportion of SCRAC, Nan Su mix design method is used. A number of trials was conducted with admixtures like fly ash and silica fume. With the proper mix proportion, the workability of SCRAC (flowability, passing ability, segregation to resistance) satisfied the parameters as per EFNARC guidelines (2002). In the proposed modified two stage mixing approach (TSMA_{sf}), a silica fume-cement slurry was prepared and premixed with RCA resulting stronger ITZ and producing concrete with better strength and durability properties along with acid and sulphate

resistance. After adopting modified two stage mixing approach (TSMA_{sfc}) using 100% RCA, the compressive strength of SCRAC were increased by 39.07% in comparison to NMA. Strength loss and weight loss were also controlled by 16% and 11.81%, respectively, in comparison to NMA after immersing in 1% concentration of sulphuric acid solution. Immersion in 1% concentration of magnesium sulphate solution resulted strength loss and weight loss by 10% and 8.4%, respectively. The sorptivity of SCRAC prepared using TSMA_{sfc} was reduced by 25% than that of concrete prepared with NMA. EPMA petrography revealed that the weak pores and cracks present in the ITZ of concrete prepared using NMA got eliminated because of TSMA_{sfc} and the ITZ becomes stronger. Elemental mapping of concrete made up of TSMA_{sfc} showed the presence of strength contributing elements like silica, calcium and aluminium. This strength contributing elements counterbalanced the adverse effect of sulphuric acid and magnesium sulphate. From all the above analysis, it was concluded that the SCRAC obtained from 100% RCA using TSMA_{sfc} gave significant improvement in mechanical and sulphate resistance properties. Hence, this SCRAC can be used in most applications of concrete including sulphate environment also.

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