

Permeation of Iron slag and recycled aggregates concrete with microstructural characteristics

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Construction and demolition wastes can be used as recycled aggregates (RA) in development of concrete. Iron slag (IS) has further added a vision towards its successful replacement of fine aggregates (FA) in non-conventional concrete. This study investigates the permeation potential of structural grade concrete containing IS and RA as alternative for FA and coarse aggregates (CA) respectively. The CA have been replaced with RA up to 100% while FA has been substituted with the fixed amount of IS (i.e. 30%). After achieving of desired strength criterion, concrete mixes have been tested for resistance towards water, salts and gas permeation. Further, the effect of permeation has also been related with the scanning electron microscopic (SEM) images. The findings have revealed that both IS and RA resulted in significant variation towards water (64%), salt (-13%) and gas (47%) ingress making concrete permeable for higher replacements. However, the findings have been reversed for lower order replacements. The diverse geometry of hydration compounds as noted in SEM images has also confirmed the potential utilization of IS and RA in the development of non-conventional concrete.

Keywords: Concrete, Iron slag, Permeation, Recycled aggregates, Microstructure

1 Introduction

Concrete is a frequently utilized global building material and has never been claimed as green material because of its higher consumption of natural resources. By the time, many improvements have made over the years, but in past few years the emphasis is on converting it into environmentally sympathetic¹. Because of the enormous volume requirements for manufacturing of concrete, the usage of natural aggregates has also been particularly extracted from the natural resources through mining. Concrete manufacturing will reach around 2.5 tonnes in year 2025 and global consumption of natural aggregates will correspondingly rise to 12.5 billion tonnes². In India the annual consumption of natural aggregates has crossed the mark of 3330 metric tonnes³. Extraction of natural aggregates result in land degradation, air pollution, deforestation, wastelands, etc. The environmental impacts and over-exploitation of natural resources are not only associated with the construction industry but also leads to production of demolition and construction wastes (C&D)⁴.

Almost 3 billion tonnes (BT) of construction and demolition (C&D) wastes have been generated around

the world out of which Indian continent accounts for around 530 million tonnes (MT)⁵. C&D wastes have variety of environmental consequences, including the usage of landfill space, illegal dumping, river and lake siltation, and the waste of precious resources⁶. Literature has indicated that such wastes may be eliminated primarily in two ways: by lowering waste production and by repurposing/recycling for other applications⁷. Demolished concrete processed through crushing and grading can be replicated to natural coarse aggregate (NCA) in order to form recycled concrete aggregates (RA) and are viable for replacement of NCA in production of concrete. The RA based concrete mainly comprises aggregate phase, mortar phase and interfacial transition zones (ITZs)⁸⁻¹³.

The amount of coal and mineral industrial wastes is regularly increasing year by year which is dumped into open vicinity causing variety of environmental consequences, including usage of landfill, illegal dumping, river and lake siltation, and the waste of precious resources. In order to curb the preceding concerns, wastes like coal bottom ash, copper slag, steel slag, iron slag, etc. have been used as aggregates in concrete manufacturing by many researchers^{3,7,14,15}. Iron slag is one such byproduct of the iron and steel manufacturing industry that can be used as a

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replacement for natural fine aggregate (NFA). In actual fact, it is a by-product produced during manufacturing of pig iron. Large amounts of iron slag are produced all around the world. India's steel industry produces roughly 12 MT of steel slag and 24 MT of blast furnace slag per year¹⁶. By 2030, it is predicted that slag production would be around 45 to 50 MT per year and will be a major menace to all forms of life¹⁶.

The application of iron slag (IS) in concrete is a novel move of eluding its ineffective disposal. Also, sustainability in construction/concrete sector is an utmost need of the hour and emphasis should be made to minimize the carbon footprint(s) and conservation of natural resources through recycling of wastes in concrete production^{5,17}. Durability and permeation parameters are directly associated with the concrete performance and is defined as resistance against deterioration caused by physical and chemical aggressive agents. Permeation parameters always play an important role while designing industrial waste-based concrete. The permeability and porosity of the concrete are governing factors while dealing with the durability of concrete as an impermeable and water/air tight pore structure does not offer easy admittance of external deteriorating agents.

Till date, the durability parameters of RA concrete have been explored extensively around the globe^{15,18-21}. In general, the impermeability of concrete reduces with increase in RA, eventually follows the reduction of durability in concrete²². For example, concrete with RA has higher water absorption (up to 9%) and higher porosity (up to 27%) due to adhered mortar for 25% and 100% replacement levels respectively²³. Likewise, the initial water absorption value was significantly more than base concrete with 100% of RA²⁰. Conversely, the inclusion of cementitious binders controls the rate of water absorption due to pozzolanic and pore refining actions¹⁵. In fact, the permeation characters of IS based concrete is scarcely reported in the existing literature and emphasis herein is towards investigating the aforesaid behavior for IS concrete. However, some of available literature reveals that inclusion of IS in concrete as NFA results in densification of microstructure of concrete^{24,25}. Singh and Siddique, 2016¹⁴ also reported that non-vibrated concrete containing IS as NFA quoted lower water absorption and chloride permeation compared to control concrete. Likewise, the adverse effects on water and salt permeation were noticed in form of lower chloride resistance, presence of excessive

sulphate and higher initial surface absorption (ISA) with higher percentages of RA in concrete^{15,26,27}. The carbonation resistance also decreased (up to 80% to that of control) with increase in percentage of RA. In nutshell, the permeation behaviour was found to be deteriorated with higher amount of RA while on the other hand cementitious binder control the rate of deterioration to some extent.

1.1 Significance and novelty of the investigation

Adopting the practice of sustainability and cleaner production in concrete construction is the utmost primacy. The current research focuses on utilisation of industrial wastes as replacement of aggregates in concrete. The process of recycling of industrial by-products will assuredly result in lowering of environmental impacts and will add value towards economic development. The distinguishing impact of incorporating RA on the permeation characters of concrete has been examined. Similarly, brief knowledge is available for utilization of IS in concrete. But on contrary, the potential of joint replacement of IS and RA as NFA and NCA on permeation of concrete has not been explored till date. To attain in-depth knowledge of permeation characteristics is the ultimate aim of the current investigation so as to fulfill the noticed gap by performing various permeation tests on different combinations of RA and IS based concrete. Herein, the IS-RA concrete were prepared with 30% constant replacement of NFA with IS and with variable replacement of NCA (up to 50%) with RA. The permeation characteristics have evaluated through various tests like capillary suction absorption, initial surface absorption, sulphate attack, acid attack, accelerated carbonation, water penetration and chloride ingress. In addition to the preceding, the compressive strength performance was also estimated for general comparison. The attained experimental outcomes were finally corroborated by investigating microstructural changes with the support of SEM analysis. Eventually, the proposed aim further follows the approach for the maximum utilization of IS and RA in manufacturing sustainable form of concrete. Since the said approach conserves vital natural resources, it substantially reduces the number of abovementioned wastes that are generally being impelled towards landfills for their idle disposal. Therefore, the outcomes of this study will indicate valuable information on the performance and potential benefits of using concrete in advanced

structural designs and construction. To the best of authors’ knowledge, no other existing study has considered the effect of IS and RA as NFA and NCA replacement in terms of permeation behaviour in concrete respectively. The outcomes of the current experimental investigation indicate substantial variation (s) in permeation characteristics of the developed concrete covering the existing gap in available literature. In fact, the present investigation is an attempt to achieve three-way benefits by minimising the dependency of concrete on conventional materials; minimising natural resource depletion and encouraging clean and green production in construction industry.

2 Materials and Methods

2.1 Materials

An outline of experimental work conducted in the current investigation is presented in Fig. 1. Ordinary Portland Cement (OPC-43) complying the provisions of IS: 8112-2013²⁸ and NFA complying the provisions IS:2386- Part I, 1963²⁹, IS 2386- Part III,

1963³⁰ were used. Iron slag utilized was procured from ‘Sonalika Tractors’ manufacturing industry Hoshiarpur, Punjab. Sieve analysis and physical properties of IS was obtained using IS-383-2016 (BS 1881-208:1996. 2010) NCA were sourced from Chaparkandi (near the Ravi River) in Punjab, India. Physical characteristics of NCA were evaluated using IS:2386-1963;1997 Part-III. NCA passing through 12.5 mm (mesh size) sieve and retained on 4.75 mm sieve were included for the study and the grading was performed. Recycled concrete aggregates were produced by crushing discarded concrete specimens available in structure testing laboratories of the of the authors’ institute. Initial crushing was carried with a hammer followed by secondary crushing in a jaw crusher machine. The production process of RA is elaborated in Fig. 2.

Physical properties and grain size distribution are shown in Table 1 and Fig. 3 respectively³¹. Concrete mixes were designed to investigate the influence of IS and RA on permeation properties. IS was used as NFA at a constant replacement level of 30% by

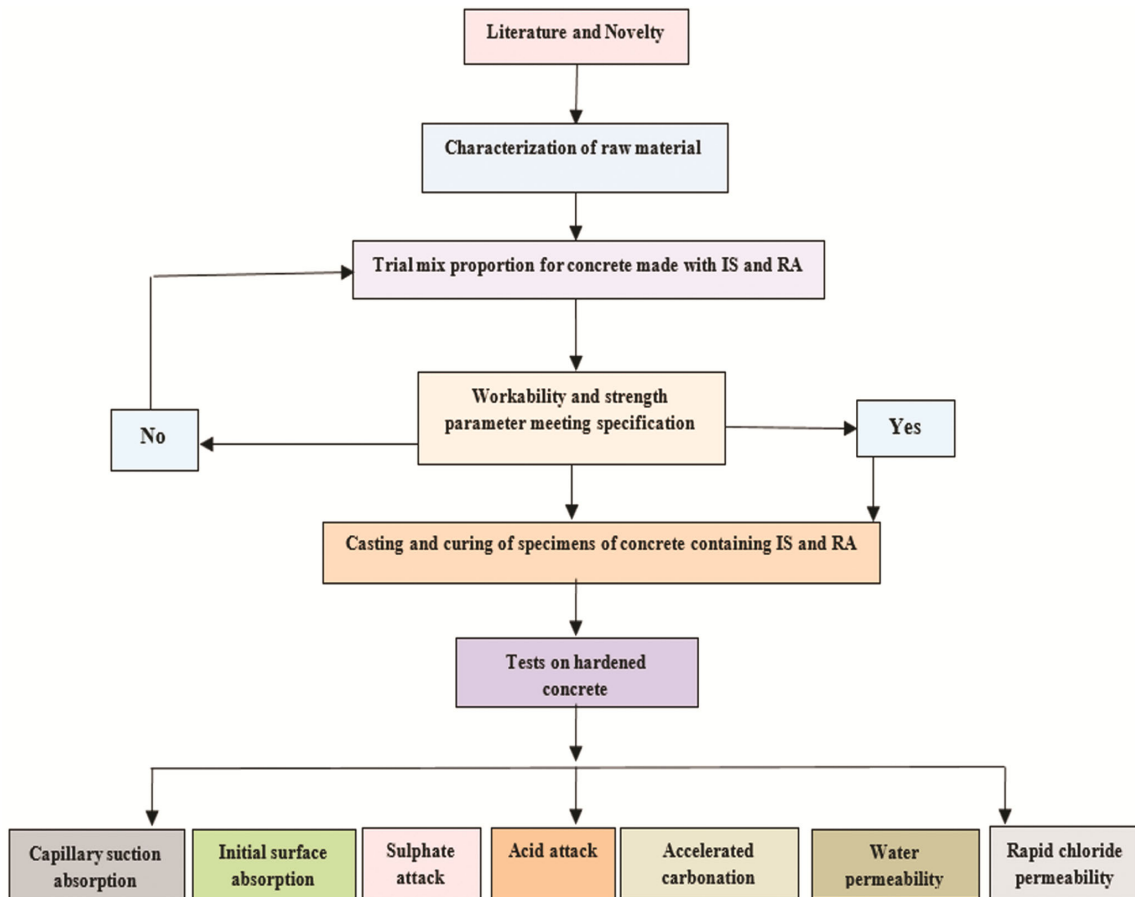


Fig. 1 — Outline of experimental work.

Table 1 — Physical characteristic of raw materials.

Property	NCA	RA	NFA	IS	Cement
Fineness Modulus (FM)	7.19	7.06	1.70	1.4	-
Specific Gravity (SG)	2.81	2.38	2.6	2.43	3.15
Water Absorption (WA)(%)	0.51	3.98	1.52	18.33	-

Table 2 — Designation of various mixes

Mix code	Mix code details
IS0RA0	100%OPC+0%RA+100%NCA+0%IS+100%NFA
IS30RA0	100%OPC+0% RA+100%NCA+30%IS+70%NFA
IS30RA25	100%OPC+25% RA+75%NCA+30%IS+70%NFA
IS30RA50	100%OPC+50% RA+50% NCA+30%IS+70%NFA
IS30RA75	100%OPC+75%RA+25% NCA+30%IS+70%NFA
IS30RA100	100%OPC+100%RA+0% NCA+30%IS+70%NFA

Table 3 — Concrete mix details (kg/m³)

Mix Code	w/c ratio	OPC (kg/m ³)	RA (%)	RA (kg/m ³)	NCA (kg/m ³)	IS (%)	IS (kg/m ³)	NFA (kg/m ³)
IS0RA0	0.48	421	0	0	1058	0	0	701
IS30RA0	0.48	421	0	0	1058	30	198.71	490.7
IS30RA25	0.48	421	25	223.97	793.5	30	198.71	490.7
IS30RA50	0.48	421	50	447.93	529	30	198.71	490.7
IS30RA75	0.48	421	75	671.9	264.5	30	198.71	490.7
IS30RA100	0.48	421	100	895.87	0	30	198.71	490.7



Fig. 2 — Manufacturing process of recycled concrete aggregate.

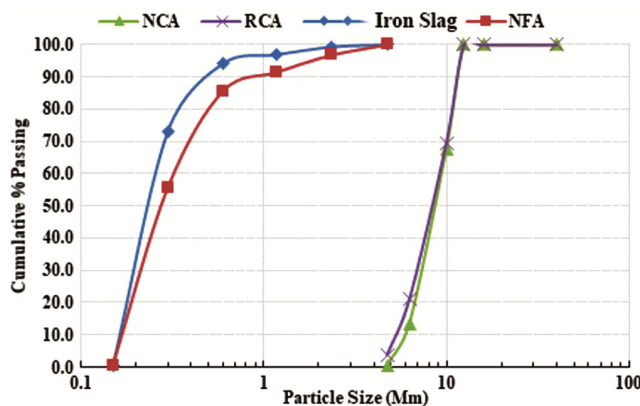


Fig. 3 — Gradation curves of aggregates.

volume. This optimum replacement level of IS was evaluated by conducting trials, while the replacement ratio of RA as NCA was varying at 0%, 25%, 50%, 75 and 100% levels. All mixes were designed to achieve the minimum target strength of 31.6 MPa i.e., M25 structural grade and for medium workability (50-75 mm slump and pump-able) complying with the

specifications of IS 10262-2019³¹. Water cement ratio ($w/c=0.48$) and cement content (421kg/m^3) were kept constant for all mixes and, saturated surface dried aggregates were used. Permeation tests were executed on all mixes after satisfying the compressive strength (CS) criteria of the M25 grade of concrete. The descriptions and details of various mixes are presented in Table 2 and Table 3 respectively.

2.2 Experimental methods

2.2.1 Capillary suction absorption tests (CSAT)

CSAT were performed to quantify the rate of 'water absorption' in concrete when only single surface of the concrete specimen is exposed for absorption to water. The increase in mass of a specimen as a function of time due to water absorption was measured. In CSAT, three specimens with of diameter 100mm and a thickness of 50mm discs were cast and conditioned according to ASTM C 1585-13 at 28 days of curing age.

2.2.2 Initial surface absorption tests (ISAT)

ISAT gives a low-pressure assessment of the concrete surface's water absorption. The volume flow can be approximated by determining the depth of 'water flow' along a capillary tube of known diameter. BS 1881-208 was used to determine the initial surface absorption (ISA) of the concrete. The concrete surface was sealed with a gasket circular cap of the area not less than 5000 mm^2 . The cap is filled with deionized water from a reservoir, and the cap's outflow is attached to a capillary tube. The rate of water absorption by concrete specimen was measured using displacement of a meniscus along the capillary tube after closing the tap of the reservoir under a pressure head of 200 mm. The measurements were noted at 10 minutes, 30 minutes, and 60 minutes³².

2.2.3 Sulphate and acid resistance tests

Sulphate and Acid Resistance Tests were performed out in accordance with standard ASTM C1012-04. After 28 days of water curing, concrete specimens were removed from the stream curing tank and were kept to dry for one day and the initial weight was noted prior to the test. After 28 days of regular water curing, the specimens (100x100x100mm) were subjected to a sulphate resistance test by submerging in a 5 percent Na_2SO_4 solution. After 28 and 90 days of immersion, the variation in weight and CS was judged. By comparing the variation in CS and weight of specimens before and after exposure, sulphate attack was evaluated. The procedure was followed for all specimens for evaluating ingress of sulphates in concrete. Acid attack tests were performed by immersing the specimens (100x 100x 100mm) in 5% H_2SO_4 solution after 28 days of water curing. The variation in weight and reduction in CS after 28 days of immersion was evaluated. The evaluation of acid attack on specimens was performed by comparing the change in CS and weight of specimens before and after exposure.

2.2.4 Accelerated carbonation tests

Accelerated carbonation frequently used to define the effect of ingress of carbon dioxide in concrete. The three essential parameters on which the carbonation phenomenon is dependent are temperature change, CO_2 concentration, and RH. In current testing, the specimens were placed in a sealed environment chamber with regulated aforesaid variables. The carbonation is severe when structures are exposed with relative humidity (RH) between

40% to 70% hence the same condition was maintained. Likewise, the temperature was kept in range from 25 degrees Celsius to 27 degrees Celsius. Throughout the testing duration, CO_2 content was kept constant at 4% to speed up the carbonation reaction. In carbonation chamber, the specimens kept under aforementioned controlled condition during desired exposure period.

2.2.5 Water penetration tests

These tests were performed on 150 mm cube specimens according to BS EN 12390-8 after cure regime of 28, and 90 days. In water permeability set up, the specimens were held at water pressure of $500 \pm 50 \text{ kPa}$ for 72 hours. After the predetermined time, the specimens were removed and split in perpendicular direction to the water-pressured face. The desired values were noted on the split face dried enough to see the water front mark. The penetration values were noted in millimetres for all tested specimens of mixes.

2.2.6 Chloride resistance test

Chloride Resistance Test on concrete were performed out in accordance with AASHTO T277 to look out the influence of chlorides that are present in environment in concrete. The test evaluates the electrical conductivity in the laboratory to offer a quick estimate of resistance to chloride ion penetration in concrete. Concrete specimens with 100 mm x 50 mm (diameter x thickness) were used. Preconditioning of the specimens was performed as specimens were coated with epoxy and placed under vacuum for three hours. Before soaking for 18 hours, the specimens in vacuum were saturated for one hour and later were placed in the test set up. Standard solutions with a 3 percent NaCl and 0.3N NaOH were used during testing. The test ran for period of six hours at 60-volt potential and the readings were taken at every 30 minutes. At completion of six hours the number of coulombs travelled through the specimens were noted and class of the specimen was categorised as per³³.

2.2.7 Qualitative microstructural analysis

Qualitative Microstructural Analysis was performed and the characteristics of IS-RA based concrete were examined using scanning electron microscopy (SEM) analyses. SEM analysis of broken concrete specimens from 100 mm cube was conducted using a make of 'ZEISS Sigma 500 VP field emission scanning electron microscope' after 28

days of curing.

3 Results and Discussion

The replacement of conventional aggregates with the industrial by-products influences the permeation behaviour and microstructure of concrete as these by-products differ from the conventional aggregates both physically and chemically.

3.1 Capillary suction absorption test

During the initial contact of unsaturated concrete with water, the capillary suction dominates the water infiltration. The absorption due to capillary rise regulates the rate at which water enters in unsaturated concrete. The slope was plotted between water absorption and the square root of contact time in order to determine the sorptivity ($\text{mm}/\sqrt{\text{s}}$) of the mixes. The IRA was noted of the first six hours whereas the slope of 1 to 7 days of data provides the secondary rate of absorption (SRA) ³⁴. The outcomes of CSAT tests revealed that the concrete prepared with RA resulted in higher capillary suction compared with CM (IS30RA0). However, inclusion of IS improves water absorption resistance of concrete. Concrete prepared with 30% IS only has 7% and 18.18% lower IRA and SRA values than nominal mix (IS0RA0) ($0.0199\text{mm}/\sqrt{\text{s}}$ IRA & $0.0011\text{mm}/\sqrt{\text{s}}$ SRA).

Similarly, concrete IS30RA25 shows higher but somewhat comparable absorption compared to CM (IS30RA0). Concrete containing 50%, 75%, and 100% RA along with IS have observed 33.51%, 64.86% and 76.75% higher IRA values than CM (IS30RA0) at 28 days curing period. These mixes also have higher SRA values of 0.0019, 0.002 and 0.0026 $\text{mm}/\sqrt{\text{s}}$ than CM ($0.0009\text{mm}/\sqrt{\text{s}}$). Figure 4(a) indicates that the addition of IS was proved to be effective in reducing water absorption values as CM shows minimum values IRA ($0.0185\text{mm}/\sqrt{\text{s}}$) and SRA ($0.0009\text{mm}/\sqrt{\text{s}}$) at 28 days curing age. It is further concluded that the use of RA caused significant increase equally in IRA and SRA, whereas the addition of IS aids in improving the water absorption resistance of concrete. Figure 4(b) indicates that capillary suction absorption of various concrete mixes follows same pattern at 90 days curing age as concrete IS30RA25, IS30RA50, IS30RA75, and IS30RA100 reports higher IRA values of 0.0183, 0.0229, 0.0294, and 0.0316 $\text{mm}/\sqrt{\text{s}}$ as compared to IS30RA0 ($0.0172\text{mm}/\sqrt{\text{s}}$).

The trends reported in the present investigation are analogous to earlier findings wherein an increase in the RA content increases the water absorption. Silva et al.,

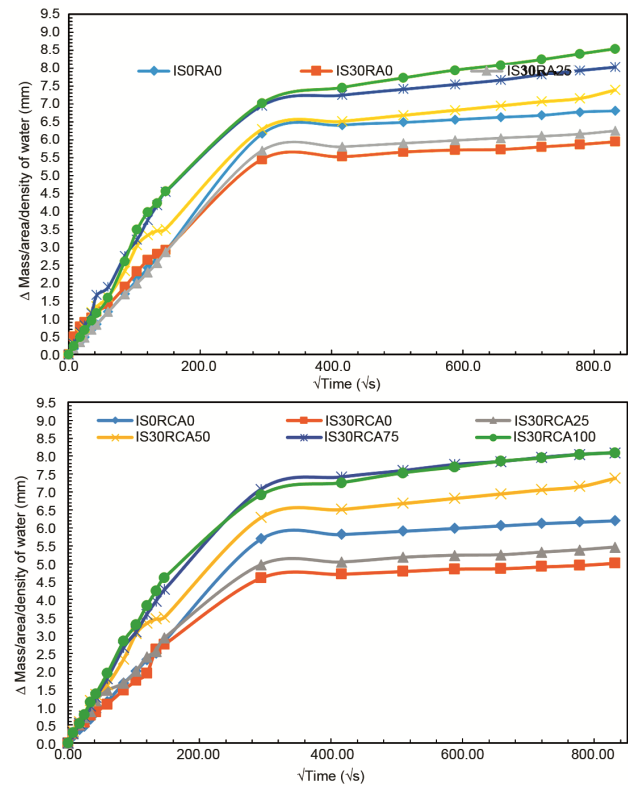


Fig. 4 — Capillary water absorption of various concrete mixes at (a) 28 days and (b) 90 days

2021²⁰ reported that 25%, 50% and 100% replacement of NCA by coarse RA resulted in a 9.1%, 12% and 27% increase in water absorption respectively. Undoubtedly, the higher water absorption in RA mixes was attributed to the porous RA with adhered mortar. However, the inclusion of IS along with RA lead to a slight improvement in the water absorption resistance. The concrete containing 30% IS and 25% RA resulted in slight decrease in IRA ($0.0191\text{mm}/\sqrt{\text{s}}$) in comparison with the CM ($0.0199\text{mm}/\sqrt{\text{s}}$). Alike, Singh and Siddique, 2016¹⁴ also reported beneficial impact of IS up to 40% by weight as less absorption was noticed compared to CM.

The IS being finer than NFA resulted in enhanced pore refinement leading towards denser microstructure and lower water absorption¹⁴. It can be concluded that the porous RA governs over the filling effect of IS at 50% replacement, leading to an increase in water absorption. Figure 5(a) and Figure 5(b) shows the noted values of IRA and plot of absorption I (mm) vs $\text{time}^{1/2}$ respectively. In the current investigation, the method of least squares and linear regression analysis was performed for the experimental outcomes.

A commonly used mathematical technique of 'least

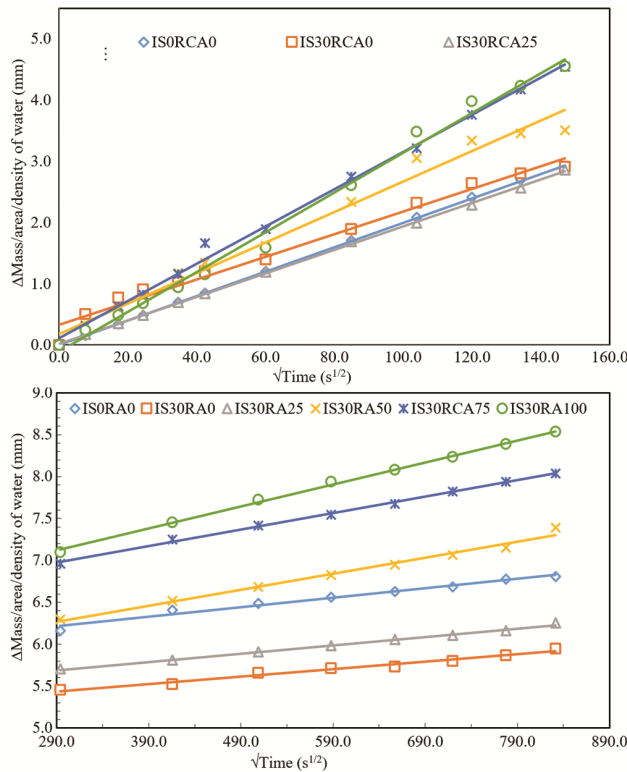


Fig. 5 — Determination of (a) IRA, and (b) SRA using least squares and linear regression plots

squares method’ permits the analyst to evaluate ‘the best way of fitting a curve’ on top of charts with certain experimental data. In general, the method is globally used to prepare ‘scatter plots’ with easier interpretation and can be easily related with ‘regression analysis’. Herein, for the experimental outcomes, linear regression analysis was performed correspond to least square method for reference. Herein, Fig. 5(a) and Fig. 5(b) presents IRA and SRA rates were determined using least square method and corresponding plots were drawn using liner regression analysis for different concrete mixes at different curing periods.

3.2 Initial surface absorption test

The test evaluates the rate of water flow inside the concrete surface through a specified surface area. The initial rate of surface absorption of all mixes at 28 days of curing is shown in Fig. 6(a). These figures indicate that mix containing 30% IS and 100% NCA have lower initial surface absorption than nominal mix (IS0RA0). The figure indicates that concrete with 30% IS and 100% NCA has lower initial surface absorption than nominal mix (IS0RA0). Similarly, concrete IS30RA25 observed lower but comparable

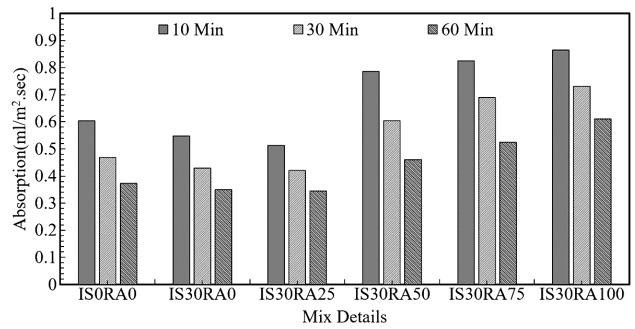


Fig. 6 — Initial surface absorption for all the mixes at (a) 28 days, and (b) 90 days curing age.

water absorption in relative to CM (IS30RA0). However, increasing RA content beyond 25% results in significant increase in ISA as mixes IS30RA50, IS30RA75, and IS30RA100 demonstrated 44.45%, 51.85%, and 59.25% higher ISA-10 values than CM (IS30RA0) (0.547 ml/m².sec) at 28 days curing period. Similar pattern was also observed at 90 days, as concrete IS30RA50, IS30RA75, and IS30RA100 shows higher ISA-10 values of 0.698, 0.746, and 0.761 ml/m².sec than CM (0.445 ml/m².sec) as shown in Fig. 6(b).

Similar findings were noted by ²⁰ wherein an 35% of increase in ISA values was observed on entire replacement by RA ³⁵. However, it can be seen that the addition of IS was effective in reducing surface absorption values as mix containing 25% RA and 30% IS has shown a minimum value (0.513 ml/m².s) of ISA-10. Therefore, it can be concluded that the use of RA as a partial replacement of NCA resulted in a significant increase in the ISA, whereas the addition of IS helps in improving the water absorption resistance of concrete. Due to the presence of old and new ITZs, RA has a much more complicated character than NCA. The ITZ phase is the weakest due to the presence of old adhering mortar or cement paste. The NCA has dense hydrates, whereas RA include loose and porous hydrates. When compared to NCA, RA has high absorption of water, low bulk density, lower specific gravity, and higher abrasion loss. The increase in water absorption of RA generally reaches 2-3 times more than that of conventional aggregate.

3.3 Sulphate and acid attack test

The resistance to sulphate attack is generally measured by evaluating CS and loss in mass / weight of specimens. Usually, RA with higher content results mostly in decrement of resistance to sulphate

Table 4 — Variation of weight due to sulphate attack

Mix Description	28 days curing			90 days curing		
	Weight(gm)		% increase	Weight(gm)		% increase
	Before immersion	After immersion		Before immersion	After immersion	
IS0RA0	2575	2591	0.62	2662	2701	1.46
IS30RA0	2515	2553	1.51	2572	2635	2.44
IS30RA25	2325	2381	2.40	2418	2496	3.22
IS30RA50	2200	2264	2.90	2368	2470	4.30
IS30RA75	2181	2256	3.43	2266	2386	5.29
IS30RA100	2151	2241	4.18	2228	2359	5.87

Table 5 — Variation of CS due sulphate attack

Mix Description	28 days curing			90 days curing		
	CS (MPa)		% increase	CS (MPa)		% increase
	Before immersion	After immersion		Before immersion	After immersion	
IS0RA0	24.1	24.94	3.48	25.1	26.31	4.82
IS30RA0	25.93	26.74	3.12	26.63	27.8	4.39
IS30RA25	26.23	27.03	3.04	27.10	28.14	3.83
IS30RA50	24.22	24.91	2.84	25.17	25.81	2.54
IS30RA75	21.89	22.31	1.91	22.96	23.38	1.82
IS30RA100	20.88	21.12	1.14	22.14	22.48	1.53

Table 6 — Variation of weight due to acid attack

Mix Description	28 days curing			90 days curing		
	Weight (gm)		% reduction	Weight(gm)		% reduction
	Before immersion	After immersion		Before immersion	After immersion	
IS0RA0	2411	2294	4.85	2418	2209	8.64
IS30RA0	2524	2389	5.34	2527	2261	10.52
IS30RA25	2571	2421	5.83	2534	2261	10.77
IS30RA50	2558	2357	7.85	2587	2245	13.21
IS30RA75	2496	2245	10.05	2463	2086	15.34
IS30RA100	2422	2143	12.24	2540	2106	17.08

ingression but concrete with IS performed slightly better. Concrete specimens measuring 100 mm x 100 mm were cast to determine the effect of sulphate attack on weight and CS. The specimens endured 28-day water curing before being submerged into 5% sodium sulphate solution. Throughout the whole testing of 90-days duration the pH of the solution was kept constant. For the purpose of determining the mass variation resulting from the degradation of concrete specimens, the initial mass and the mass after immersion of 28 and 90 days were measured.

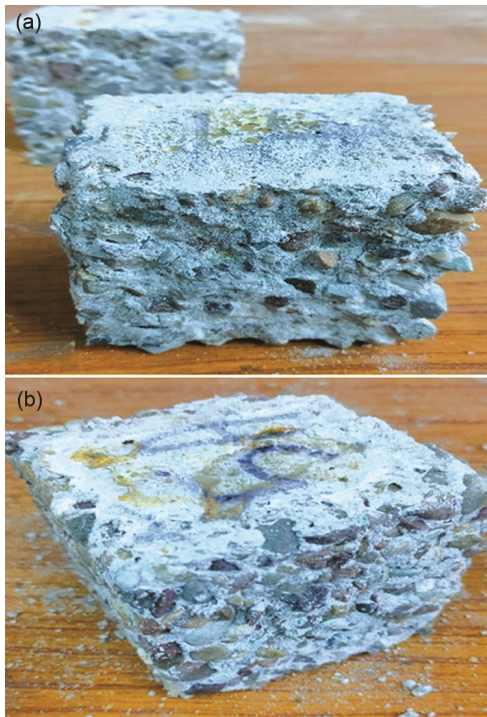
From the Table 4 and Table 5, it has been observed that all concrete mixes resulted in gain of mass and increase in CS with progressive immersion period in Na_2SO_4 solution. After both curing durations (28 and 90 days), the concrete IS30RA100 exhibits the largest

gain in mass compared to other concrete mixes. CS of the same mixes was also observed to increase with increasing of RA. This may be explained by the development of needle-shaped ettringite crystals following the interaction of the hydrates $\text{Ca}(\text{OH})_2$ and Na_2SO_4 with one another. As the additional salt enters into concrete microstructure through pore solutions, it combines with $\text{Ca}(\text{OH})_2$ along with additional sulphate precipitates thereby increasing mass and CS of the specimens.

For the purpose of determining the variation in mass caused by the degradation of concrete specimens, the initial mass and the mass of concrete specimens after the immersion time of 28 and 90 days were measured. The effect of acid attack on variation in the mass and CS of concrete is shown in Tables 6

Table 7 — Variation of CS due to acid attack

Mix Description	28 days curing			90 days curing		
	CS(MPa)		% reduction	CS (MPa)		% reduction
	Before immersion	After immersion		Before immersion	After immersion	
IS0RA0	23.81	22.2	6.76	25.66	23.21	8.94
IS30RA0	26.79	24.61	8.13	29.8	26.61	10.70
IS30RA25	26.33	23.89	9.26	27.5	24.35	11.45
IS30RA50	24.11	21.56	10.57	24.7	21.13	14.45
IS30RA75	21.63	19.23	11.09	22.30	18.63	16.45
IS30RA100	20.36	17.73	12.91	23	18.73	18.56

Fig. 7 — Concrete specimens after exposure of 5% diluted H₂SO₄ for (a) 28 days, and (b) 90 days.

and Table 7. Due to acid attack of H₂SO₄, the concrete matrix becomes fragile, and the size of the specimen is shrinks because of loss in cement paste as a result of which the CS and mass of the concrete was reduced. Similar to abovementioned acid attack resistance was evaluated by relating the change in mass / weight and CS development of concrete before and after the exposure (Fig. 7).

On contrary, all the mixes observed a reduction in weight after 28 days' exposure to 5% H₂SO₄. The RA mix IS30RA100 observed the highest loss in weight of 12.24% whereas the CM (IS30RA0) reported a weight loss of 5.34% after 28 days of exposure. The weight loss of mix IS30RA75, IS30RA50 and

IS30RA25 was 10.05%, 7.85% and 5.83% lower than the CM that further got reduced to 15.34%, 13.21%, 10.7% after 90 days of exposure. The concrete IS30RA25 observed a higher but comparable change in weight with respect to CM after 28 days' exposure to acid attack revealing enhancement in acid attack resistance in RA concrete prepared with the inclusion of 30% IS. The change in compressive strength also depicts the percentage reduction in the CS of the concrete mixes after 28 days' exposure to 5% H₂SO₄. The percentage decrement in CS was alike to variation in weight as the highest reduction of 12.91% was observed in concrete IS30RA100 while the least reduction of 8.13% was noted for concrete IS30RA25 respectively compared to CM. The altering of NFA with IS in RA concrete resulted in an improvement in resistance to acid attack. The concrete IS30RA25 observed a higher but comparable percentage reduction in CS with respect to CM after 28 days' of exposure. Table 8 represents the comparison between the results and trends reported in the present investigation with the trends observed in the existing literature.

3.4 Accelerated carbonation test

The accelerated carbonation tests accelerate the carbonation reaction and it becomes easy to determine the carbonation depth of concrete specimens in short period of time. The values of the accelerated carbonation tests of all concrete mixes prepared with various levels of RA and a constant proportion of IS are shown in Fig. 8(a) and 8(b). Both figures present the tests results attained at 28 and 90 days of curing with exposure of CO₂ up to 16 weeks. The effect of RA can be evidently perceived with the variance in carbonation depths for each IS-RA concrete mixes. In overall, the carbonation resistance of all IS and RA mixes decreased with the increase of RA content. At 28 days curing, an increase in carbonation depth of 21.3 %, 15.7 %, 19.8 %, and 13.7 % was noted for

Table 8 — Comparison between the trends observed in present study with that of existing studies

References	Materials	Replacement Levels (%)	w/c Ratio	Compressive Strength (MPa)	Water Absorption			Initial Surface Absorption ISA10 (ml/m ² s)	Acid Resistance	
					By Capillary (mm/sec ^{1/2})		By Immersion (%)		Change in Strength (%)	Change in Weight (%)
					IRA	SRA				
18	RA	0%, 100%	0.55	25.84 - 32.58	-	-	-	7.21%	-	
19	RA	0%, 25%, 100%	0.55	54.1 - 59.7	3.43-7.53*10 ⁻³ g/mm ² (72 hr)	-	13.45 - 18.60%	-	10.72%	
40	SCC, RA	0%, 50%	-	37.5 - 40	0.02 - 0.0183	-	-	0.565 - 0.631	-	
20	RA	0%, 100%	0.5	18 - 26.5	0.0325 - 0.0418	-	-	0.283 - 0.380	-	
41	SCC, IS	0%, 10%, 25%, 40%	-	68.70 - 78.33	-	-	-	-	-	
42	IS	0%, 10%, 20%, 30%	0.5	26.08-55.73	-	-	-	-	-	
43	RA	0%, 100%	0.43	66.9 - 72.6	0.032 - 0.051 mg/mm ² /s ^{0.5}	-	11.94 - 16.32%	-	-	
44	IS, RA	IS-0%, 20%, 40%, 60%, 80%, 100% RA- 25%,50%, 75%,100%	-	26.93 - 28.57	-	-	-	-	-	
14	SCC, IS	0%, 10%, 25%, 40%	-	31.5 - 41.8	-	-	4.88 - 3.81%	-	-	
6	RA	0%, 25%, 100%	0.52	48 - 50.3	5.51-7.81*10 ⁻³ g/mm ² (72hr)	-	14.2 - 18%	-	-	
Present Study	IS + RA	IS 0%, 30% RA 0%, 25%, 50%	0.48	22.3 - 32.5	0.0191 - 0.0273	0.001 - 0.002	-	0.513 - 0.825	19.34 - 27.47	6.142 - 8.97

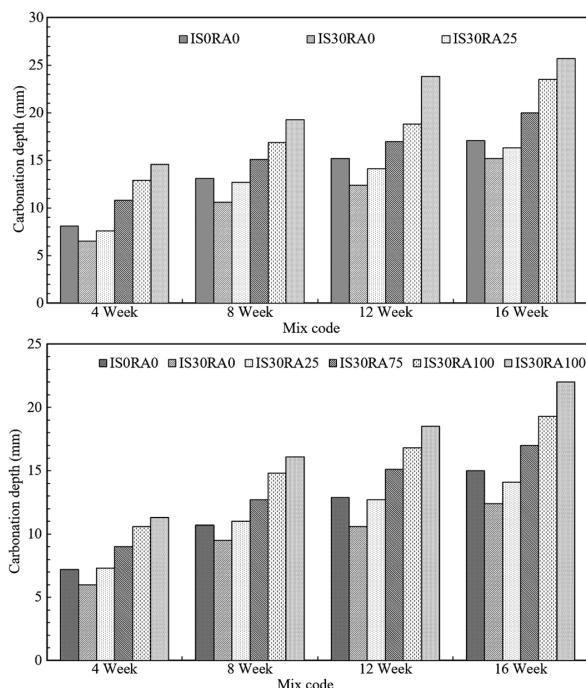


Fig. 8 — Carbonation depth of concrete at (a) 28 days, and (b) 90 days of curing period.

25% of RA (IS30R25) in comparison to CM (IS30R0)

after 4, 8, 12, and 16 weeks exposure respectively. Similarly, for same exposures, the depths were increased up to 50%, 33.1 %, 42.4 %, and 37.1 % for concrete IS30R50. Likewise, for the aforementioned

exposure, the carbonation depths were increased by 76.6 %, 55.7 %, 58.4 %, and 55.6 %, and 88.3 %, 68.4 %, 74.5 %, and 77.4 % for concrete IS30R75 and IS30R100 respectively for same curing.

As expected, with an increase in curing period carbonation depths were decreased at all exposures. For 90 days curing, an increase in the carbonation depths with respect to CM (IS30R0) for 25%, 50%, 75% and 100% of RA, have been reached up to 21.3%, 50% 76.6% and 88.3% for 4 weeks exposure to CO₂. Likewise, for exposure of 16 weeks, the corresponding rise was limited to 13.7%, 37.1%, 55.6% and 77.4% for the above said concrete respectively. The pattern of CS was similar to that of trends of decreasing carbonation resistance for the concrete mixes with more than 25% RA replacement. Similar to CS, concrete (IS30R0) with 30% IS and

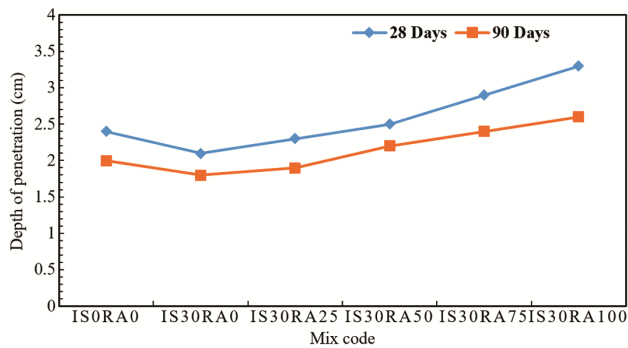


Fig. 9 — Water penetration tests results

100% NCA performed better carbonation resistance because of increased particle density of concrete mix and reduced availability of pores for ingress of CO_2 compared to CM (IS0R0). Due to enhanced dryness in the surrounding environment, the response between calcium hydroxide $[\text{Ca}(\text{OH})_2]$ and CO_2 becomes slower thereby reducing the interaction between $\text{Ca}(\text{OH})_2$ and CO_2 .³⁷

3.5. Water penetration test

As per earlier findings, utilization of RA considerably varies the performance of concrete in most of the experimental aspects^{38,39}. Out of abovementioned aspects another important aspect is the estimation of water penetration depth inside the concrete when applied at specified pre-set pressure. Such impermeable conditions are generally required for construction of water resistance structures such as dams, tunnels etc., Herein BS EN 12390-8:2000 was used in order to estimate the water penetration in IS-RA concrete, the recommended tests were performed (British Standards Institution BSI, 2003) on specimens measuring 150mm*150mm*150mm up to 90 days curing.

Figure 9 presents the results of the water penetration tests indicating clearly, an increase in water penetration depths with higher levels of RA in concrete. Up to 90 days, the water penetration in concrete IS30R100 was highest than that of the concrete IS30R0 and nominal mix IS0R0 by 57.1% and 37.5% respectively at 28 days and 44.4% and 30% respectively at the age of 90 days. The observed trends accredited to the presence of porous residual mortar of RA and coarser surface of IS. Furthermore, the results for the concrete with 30% IS and 25% of RA resulted in reduction of water penetration depth to an order of merely 4.2% and 5% in relative to the concrete IS0R0 at curing duration of 28 and 90 days respectively. Also, the concrete IS30R25, IS30R50, IS30R75 and IS30RA100 results in increase in the

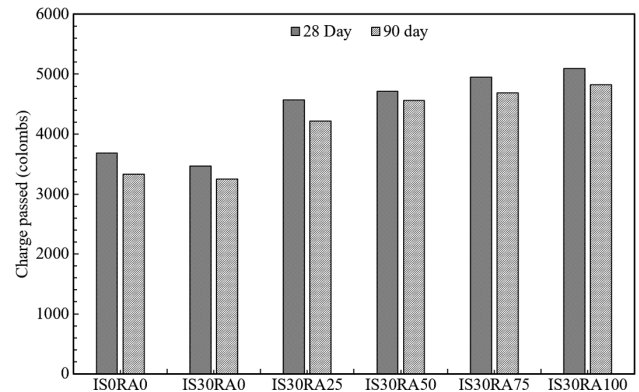


Fig. 10 — Coulombs passed through various concrete mixes containing IS and RA at 28 & 90 days curing age.

water penetration depth by 9.5%, 19.1%, 38.1%, 57.1% at age of 28 days and 5.5%, 22%, 33.3%, 44.4% at age of 90 days respectively upon linking to concrete IS30R0. It can be inferred that concrete prepared with 30% IS only resulted in drop of water absorption capacity of due to increase in denseness of concrete as fine IS particles yields better pore size refinement. Undoubtedly, the higher water penetration in RA mixes was attributed to the porous RA with adhered mortar.

3.6. Rapid chloride permeability test

The test expedites the procedure to assess the chloride diffusion of concrete in a reasonable amount of time. The ingress of chloride in concrete is one of the usual causes of durability concerns, mainly in regard to corrosion of reinforcement in concrete. The test results of various concrete mixtures at 28 and 90 days curing period are presented in Fig. 10. It can be observed that inclusion of RA as replacement of virgin aggregates badly upsets the chloride resistance of specimens of concrete as it drops with an increase in the alteration levels. The reduction in resistance towards chloride permeation is accredited to the higher porosity of RA and adherence of older mortar thereby forming an easy channel for the chloride ions to penetrate inside the concrete. On the contrary, IS as NFA improves the chloride resistance as the concrete with 30% IS reported lowest value of charge passed at all curing ages.

At 28 days curing age, concrete comprising 25%, 50%, 75% and 100% RA along with 30% IS have 31.82%, 35.86%, 42.66%, and 46.81% higher value of total charge as compared to CM. Similar pattern was observed at 90 days curing period as concrete IS30RA25, IS30RA50, IS30RA75, and IS30RA100

Table 9 — Variation of weight due to chloride attack

Mix Description	28 days curing			90 days curing		
	Weight (gm)		% increase	Weight (gm)		% increase
	Before immersion	After immersion		Before immersion	After immersion	
IS0RA0	2497	2510	0.7	2494	2537	1.72
IS30RA0	2506	2541	1.39	2540	2614	2.91
IS30RA25	2560	2597	1.44	2556	2634	3.05
IS30RA50	2501	2569	2.71	2499	2601	4.08
IS30RA75	2498	2573	3.01	2505	2654	5.9
IS30RA100	2472	2561	3.60	2413	2559	6.05

Table 10 — Variation of CS due to chloride attack

Mix Description	28 days curing			90 days curing		
	CS(MPa)		% increase	CS(MPa)		% increase
	Before immersion	After immersion		Before immersion	After immersion	
IS0RA0	24.1	24.93	3.44	24.4	25.48	4.42
IS30RA0	26.55	27.42	3.27	26.9	28.01	4.12
IS30RA25	25.44	26.22	3.06	26	27.15	4.42
IS30RA50	23.71	24.22	2.15	24.49	25.30	3.30
IS30RA75	21.71	22.01	1.38	22.99	23.53	2.34
IS30RA100	20.14	20.28	0.69	21.41	21.84	2

reported higher charge passing of 4214, 4561, 4684, and 4821 Coulombs compared with CM (IS30RA0) i.e., 3250 Coulombs. As per ASTM C1202-19, the chloride permeation of CM and nominal concrete (IS30RA0 and IS0RA0) has been found under category of 'moderate' while concrete IS30RA25, IS30RA50, IS30RA75, and IS30RA100 falls under the category of 'higher risk corrosion'.

It is well known that chloride ion penetration has a negative impact on the durability character of all sorts of concrete as permeation of chlorides leads to corrosion of steel. The prepared specimens of concrete (100 mm size cubes) were made to measure the weight and CS loss carried out due to chloride attack. The specimens were left to cure in water for 28 days before being submerged in NaCl solution at a 5% concentration. For the purpose of determining the variation in mass caused by the degradation of concrete specimens, the initial weight and the weight after the immersion time of 28 and 90 days were measured. Table 9 and Table 10 presents the effect of chloride attack bringing variation in weight and CS of different concrete specimens. It has been noted that in all concrete specimens the weight has been increased. At both curing ages, concrete IS30RA100 exhibits the largest gain in mass compared to all other concrete blends. The observed gain in mass directly leads to highest water and salt absorption through pores of RA

and IS in concrete leading to bulging of specimens. Excessive presence of salts and water resulted in slow development in strength and correspondingly weak durability performance. The above said statement has been further supported with SEM analysis wherein porous C-S-H crystals or gels are observed for the corresponding concrete. Such pattern may also be explained by the under development of needle-shaped crystals of ettringite following lesser interaction of the hydrates $\text{Ca}(\text{OH})_2$ due to higher presence of NaCl.

3.7 SEM analysis

On the fracture surfaces of specimens, SEM imaging was performed after the desired 28 and 90 days duration. The most of the common features of SEM analysis of RA concrete has been investigated earlier but the influence of RA along with IS has not been identified till date. The idea of performing SEM analysis was to co-relate the permeation behaviour associated with properties of RA with IS and to identify the flaws/faults in context to C-S-H gel / crystals and other hydration products. Figure 11 present SEM images of IS particles at different scales indicating the spherical shape and rough surface texture. The SEM images [Fig. 12 (a) and Fig. 12(b)] for the control mix IS0RA0 at 28 days and at 90 days of curing was presented for general comparison.

Herein, the SEM analysis was conducted for

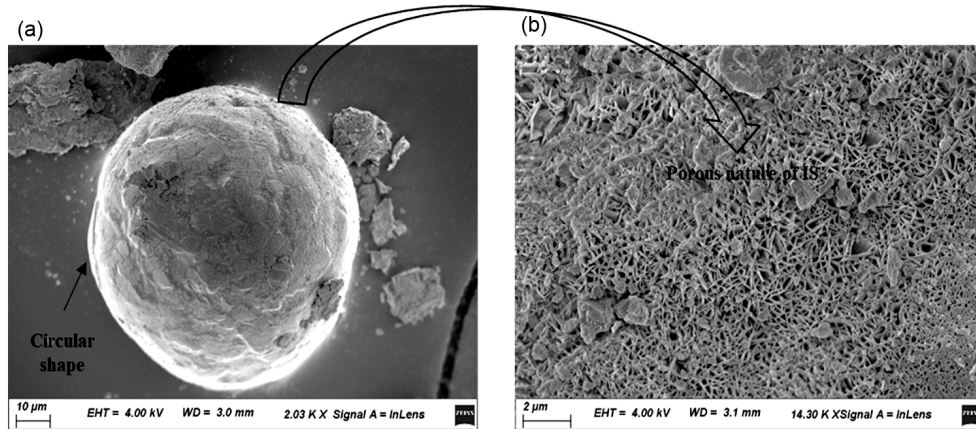


Fig. 11 — SEM image of IS showing spherical shape and rough texture of particle (a) at 10 microns and (b) at 2 microns

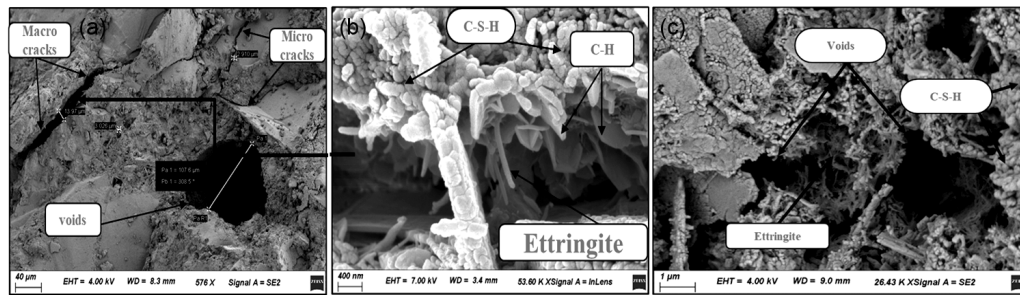


Fig. 12 — SEM picture for IS0RA0 at (a) 28 days and at (b) 90 days of curing

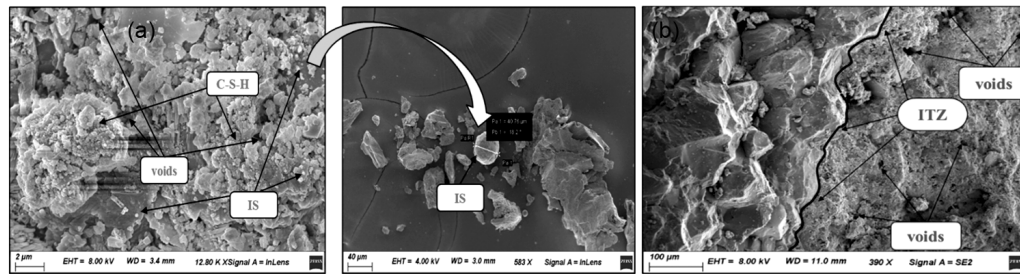


Fig. 13 — SEM image for Mix IS30RA0 at (a) 28 days and (b) 90 days of curing

the three concrete mixes IS30RA0, IS30RA25 and IS30RA100 for comparison and also to indicate clear difference of developed hydration products. It has been noted that C-S-H and hydration crystals formed in concrete IS30RA0 are considerably of dense nature at both the ages [Fig. 13 (a) and (b)] indicating condensed crystals as the curing time increases. Further, Fig. 14(a) and Fig. 14(b) describe layered development of C-S-H gels in hollow cloud and acicular forms along with needle-shaped ettringite after 28 and 90 days of curing. In contrast to CM (IS30RA0), the transformation in crystal shapes has been noticed for concrete IS30RA25 for both curing periods. The small amount of C-S-H gel and crystals

with an acicular / needle like structures demonstrated the slower rates of hydration than the CM. The ettringite formation and its presence have been further supported with the existence of micro-pores in the concrete. Generally, ettringite an important product of the hydration chemistry is frequently found in voids and fissures (ASTM-C 1723). Somewhere in the SEM images, an unreacted ettringite was also observed in the CM indicating development of hydration products at slower rate.

In contrast to CM (IS30RA0), the loose structure of the hydration products was evidently visible in concrete IS30RA100, as seen in Fig. 15(a-d). Figure 12 (a-b), show the existence of voids, broad gaps, and

C-S-H with loosely beaded shapes at 28 and 90 days of curing, respectively. Due to their brittle / porous / weak character, these varieties of flaky crystals could not be used in order to generate a condensed and viscous concrete, due to increase in the porosity.

Figures 11 and 13(a) show that IS has a positive impact as its finer and robust nature paralleled to NFA resulting as better pore confiner thereby improving the overall microstructure. Furthermore, addition of IS has also proved to be beneficial as the same has been played a major role in directly increasing mechanical strength and indirectly in enhancing the permeation behaviour compared to nominal mix (ISORA0). Furthermore, SEM analysis validates the noticed behaviour in terms of weak hydration products subsequently influencing the permeation characteristics. The presence of excessive

moisture in the pore structure of RA primarily influences development of hydration products and is root cause in affecting all other characteristics. After 28 days of curing, the development of acutely produced, extremely porous and weaker C-S-H gel supports the largest increase in water absorption and all remaining permeation properties out of all other concrete combinations compared to CM. In overall, the SEM imaging show that all above tested permeation properties match well with the trends, which are consistent with observations reported by other researchers in related literature. For effective validation of results, a brief comparison of existing study has also been made with the available related literature (Table 8).

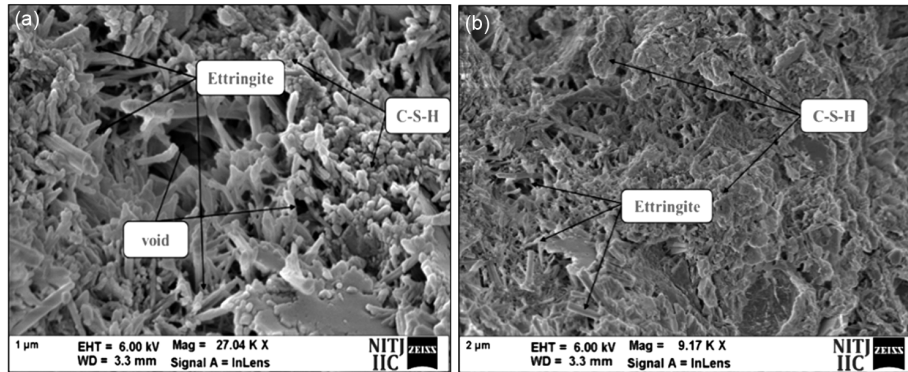


Fig. 14 — SEM image for Mix IS30RA25 at (a) 28 days and (b) 90 days of curing

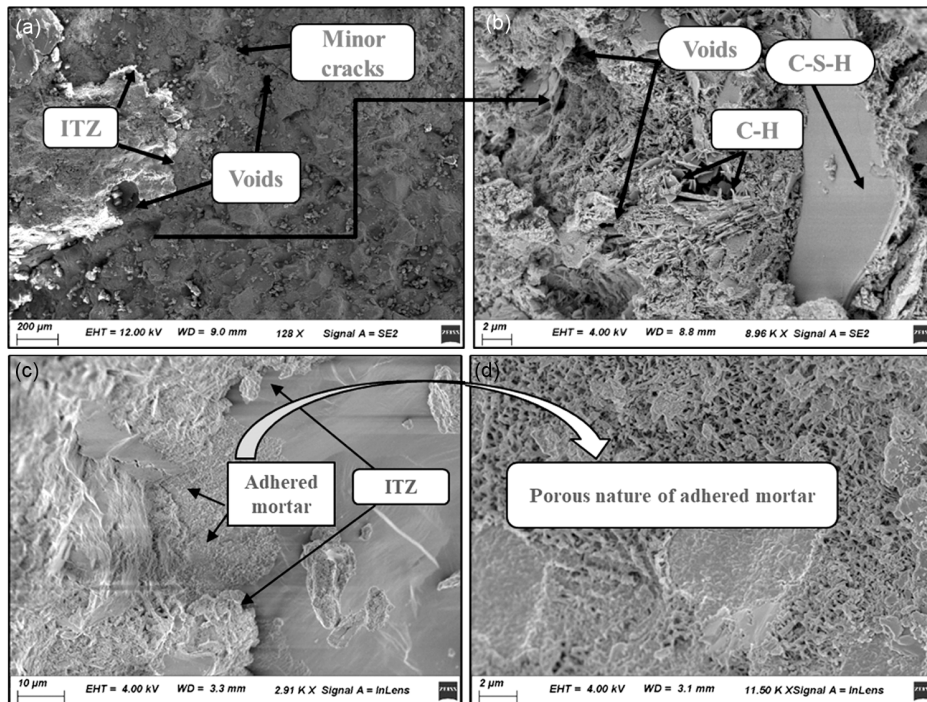


Fig. 15 — SEM image for Mix IS30RA100 at (a-b) 28 days, and (c-d) 90 days of curing

4 Conclusions

The present study investigates the influence of IS and RA specifically on permeation performance of concrete. The following conclusions are drawn from the experimental investigation:

- Use of RA end in significant increase in the IRA and SRA, whereas the addition of IS aids in improving the capillary water absorption resistance of concrete. The capillary suction absorption in IS and RA concrete is higher than the conventional concrete. Concrete containing 50%, 75% and 100% RA observed 33.51%, 64.86% and 76.75% higher IRA values than CM (IS30RA0) at 28 days curing period.
- Increasing RA content beyond 25% results in significant increase in ISA as concrete IS30RA50, IS30RA75, and IS30RA100 revealed 44.45%, 51.85%, and 59.25% higher ISA-10 values than CM thereby decreasing the resistance towards permeation significantly. The porous nature and weak adhering mortar of RA resulted in lower water absorption resistance.
- The porosity of RA influences sulphate and acid resistance of concrete as IS0RA25 (2.4%, 5.8%) and IS0RA100 (4.2%, 12.2%) reported the lowest and highest change in weight compared to CM respectively. The comparable resistance for sulphate and acid in lower RA concrete attributes

to the pore refining and robust nature of IS.

- The resistance towards chloride penetrability in RCPT results reveals that at 28 days, concrete containing RA with IS have been reduced up to 47% compared to CM. The ineffectiveness of IS and dominance of RA resulted in loss of resistance towards chloride-ion penetration. However, inclusion of IS enhances the chloride resistance of concrete as lowest charge passed was observed at all curing ages.
- The carbonation physio-chemical reactions due to presence of RA weakened the compactness inside the concrete, resulting in significant rise in carbonation depths. The maximum decrease in carbonation resistance reached up to 88% (IS30R100) with respect to CM (IS30R0) at 16 weeks of exposure after 28 days of curing. The IS acts as pore refiner yielding in better refinement of concrete (up to 25% only) as concrete IS30RA25 reported lowest carbonation depths and corresponding lower water penetration depths.
- Microstructural investigations describes intrinsic pores, voids and cracks in the ITZ of the RA, behaving as barrier in load transfer leading towards lower strength and lesser resistance towards permeation properties. Contrarily, SEM analysis of IS concrete reveals the incidence of dense microstructure and stabilized hydration products due to better pore size refinement. In overall, the appropriate replacement for effective utilization of IS and RA in concrete is 30% and 25% (IS30RA25) offering best permeation resistance.

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