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Sulfate and Acid Attack Resistance of Iron Slag and Recycled Coarse Aggregate Concrete

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Abstract **-** This study specifically explores the impact of mutual inclusion of 'iron slag (IS) and recycled concrete aggregates (RCA)' in non-traditional concrete (NTC) aiming to estimate the resistance against sulfate and acid attacks. The newness of this experimental study is in exploring of role of IS along with RCA in NTC for abovementioned durability properties, which have been comparatively underexplored. Natural sand (NS) and natural coarse aggregates (NA) were replaced with IS and RCA at constant (30%) and varying (25%-100%) amount respectively. In all, six (6) numbers of NTC were tested for sulfate and acid attack resistance while compressive and tensile tests were performed in general support till 90 days of standard curing. The resistance against sulfate and acid attack was measured in relation to variation of mass and corresponding strength performance of designed NTC. The highest increase in mass (by 6%) was noted for NTC with 30% of IS and 100% of RCA. Likewise, for the same NTC the reduction in mass due to acid resistance was limited to 17%. While NTC with IS (IS30RCA0) only and 25% of RCA (IS30RCA25) emerged as equivalent performers in terms of resistance and strength outcomes. These findings not only demonstrate a positive potential of mutual inclusion of IS and RCA in NTC but also present a promising start towards utilization of sustainable construction materials.

Keywords**:** *Concrete, Iron slag, Recycled concrete aggregates, Sulfate attack, Acid attack*

1. Introduction

Concrete permeability performs a critical role in countering several environmetal attacks [1]. Sulfate and acid attacks may result in decline of performance of concrete due to reduction in mass, spalling, softening and unnecessary expansion [2]. The resistance to such attacks is usually determined by quantifying the compressive strength (CS) and the corresponding reduction/loss of mass of cubic specimens in laboratory [3]. The shortage of natural aggregates in developing of traditional concrete has pushed the concrete manufacturers to opt for the alternative ingredients while considering the concept of sustainability. For past decade the traditional concrete has now been transformed into non-traditional concrete (NTC) while introducing recycled coarse concrete aggregates (RCA) and iron slag (IS) as alternative of natural sand (NS) and natural coarse aggregates (NCA). Both IS and RCA are nowadays considered to be a futuristic solution in manufacturing of sustainable NTC.

As per literature, the addition of RCA for upper replacements usually result in decrement of various properties while introduction of IS for lower levels aids in achieving the equivalent performance as compared to conventional concrete. B. M. V. Kumar, H. Ananthan, and K. V. A. Balaji [4] and M. Limbachiya, M. S. Meddah, and Y. Ouchagour [5] inspected the impact of RCA towards resistance to sulfate ingression in RCA concrete. The findings direct that 30% of RCA (coarse) has no negative impression on overall durability however, RCA beyond 30% drastically reduced resistance towards sulfate attack [6]. Similar observations have been noticed for acid attack resistance for RCA based NTC [7], [8]. Such behavior was attributed due to crack construction and disorder of hardened cement paste and expansion of gypsum and ettringite. However, resistance of RCA-based NTC was found to be enriched with inclusion of cement admixtures [3], [5], [9], [10].

Iron slag is an industrial by-product originated from iron/steel industries. Such slag falls under the category of the materials which does not have high scrap value and are mostly considered as waste [11]. For the past decade, IS has been incorporated in concrete as alternative to NS. The addition of IS in concrete fulfils the physical requirements allowing the blended cement to hydrate slowly because of its shape and low concentration of calcium silicate. Hence, IS somewhere assist in improving the functional qualities of ordinary concrete. The only negative aspect of IS, is its expansive nature and possibility for unfavorable reactions between other concrete components [12]. In general, very few studies are present in the literature wherein abovementioned behavior has been investigated in detail.

Likewise, the existing literature is underexplored in regard to acid resistance for concrete containing IS as aggregates. In context to impact of IS on the sulfate resistance of the concrete it has been explored that IS generally decreased the overall resistance against the sulfate attack [3], [13]. A. Maharishi, S. P. Singh, L. K. Gupta, and Shehnazdeep investigated the property of acid attack in concrete containing copper slag as NS with varying amount (0, 20%, 40%, 60%, 80% and 100%). The findings revealed that 40% replacement level of slag resulted in least change of mass (5.63%) compared to other NTC mixes. However, most of the specimens were deteriorated with higher concentration of slag for direct contact of acid solution [14].

2. Research significance and objectives

This experimental study is an attempt to deal with two major concerns of the current time i.e., safe dumping of industrial-by-products (such as IS) and to reduce ecological imbalance by reusing the construction industry wastes. Before suggesting such NTC in the concrete industry a through information regarding durability performance is mandatory. Therefore, this research concentrates on synchronized use of IS and RCA for manufacturing of durable NTC (structural grade) specifically in terms of sulfate and acid attack resistance. Both the above said are necessary parameters for evaluating the long term durability of NTC. The past studies direct that considerable research has been focused on RCA and IS but most of them involved either one at single go, rather than mutual inclusion as replacement of NS and NCA respectively. Herein, the aspect of novelty is related to the durability estimation of IS and RCA based NTC in aggressive environment. Hence, the prime objective is to estimate the sulfate and acid attack resistance of NTC based on IS and RCA with constant and different substitutions respectively. Both the tests were proposed to be conducted till curing of 90 days. In common support, the proposed NTC were also evaluated for basic strength parameters i.e. compressive and tensile strength (CS and TS).

3. Experimental program

3.1 Materials

Indian origin cement of 43 grade and river based NS and NCA conforming mandatory standards such as 'IS: 8112-2013 [15] and IS: 2836- Part I, 1963 [16], IS 2386- Part III, 1963 [17] , IS 383-2016' along with IS and RCA was used to develop NTC combinations [18], [19], [20], [21]. Iron slag is a waste product of iron industry and is a blend of metal and silicon oxide. It was procured from the famous manufacturing plant of tractors 'Sonalika International Tractors Limited in Hoshiarpur, Punjab'. The details of microstructure, manufacturing process and physio-chemical composition of IS, is presented in Figure-1 [22]. The RCA were produced from discarded specimens of concrete using jaw crusher and maximum size was kept as 12.5 mm. The gradation charts (curves) are presented in Figure-2. Normal tap water (pH 6.5-8.5) was used for cast of specimens of NTC.

Composition	% by mass
Fe_2O_3	86.12
SiO_2	5.41
Mn_2O_3	5.11
Al_2O_3	1.19
Na_2O	0.52
MgO	0.35
CaO	0.56
Fineness modulus	1.36
Water absorption	18.33 (%)
Specific gravity	2.77

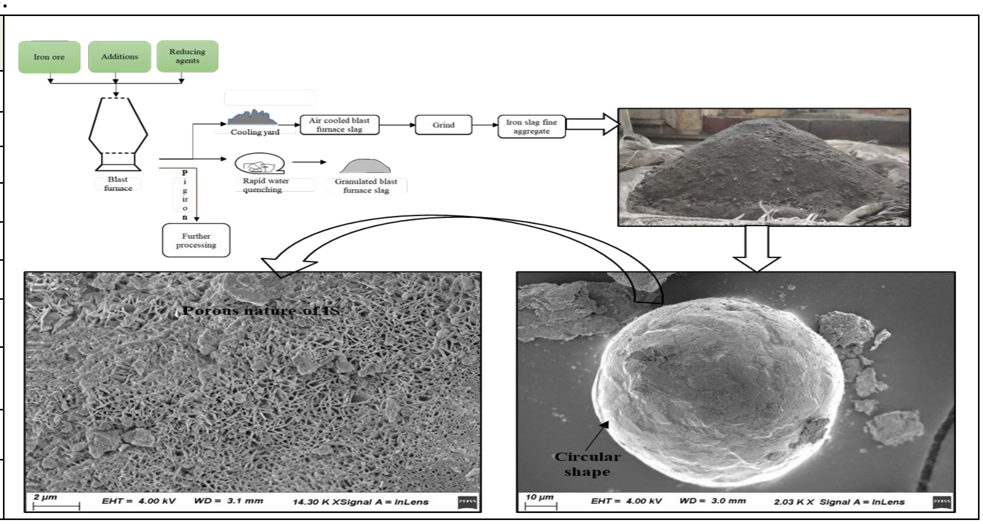
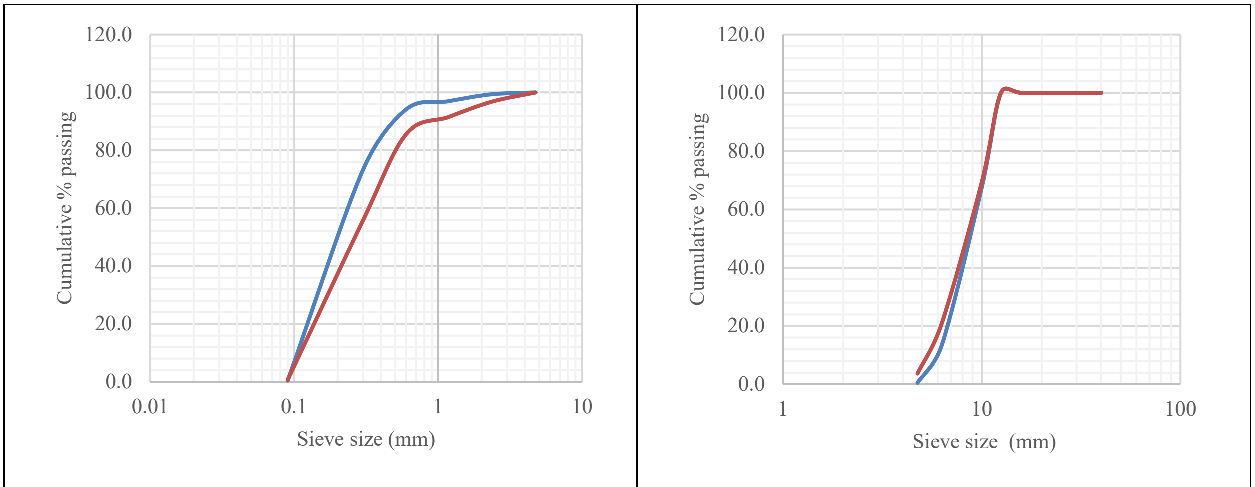


Fig.1: Structure of IS [22]



Gradation of IS (blue) and NS (orange)	Gradation of RCA (blue) and NCA (orange)

Fig. 2: Gradation of materials

3.2 Concrete mix details

In the present investigation, structural grade M25 was designed for a w/c of 0.48 for medium workability complying with 'IS 10262-2019 [23]'. The aggregate used in this investigation were used at 'saturated surface dry' state. In all, six (6) numbers of mixes were prepared; out of which the base mix comprised of 100% NS and NCA (IS0RCA0). The remaining NTC were prepared with fixed amount of IS (30%) and varying content of RCA. The constant level of IS (30%) was decided on the basis of literature and outcomes of the volume approach trials conducted before actual cast of specimens of NTC. The detailed description of base and NTC mixes is presented in Table-1 while the proportioning is presented in Table-2.

Table 1: Description of Mixes

Mix code	**Mix code description**
IS0RCA0	100%NCA+0% RCA + 100%Cement+100%NFA+0%IS
IS30RCA0	100%NCA+0% RCA + 100%Cement+70%NFA+30%IS
IS30RCA25	75%NCA+25% RCA + 100%Cement+70%NFA+30%IS
IS30RCA50	50%NCA+50% RCA + 100%Cement+70%NFA+30%IS
IS30RCA75	25%NCA+75% RCA + 100%Cement+70%NFA+30%IS
IS30RCA100	0%NCA+100% RCA + 100%Cement+100%NFA+30%IS

Table 2: Proportioning of Mixes

Cement (kg/m^3)	**w/c ratio**	**NS (kg/m^3)**	**IS (%)**	**IS (kg/m^3)**	**NCA (kg/m^3)**	**RCA (%)**	**RCA (kg/m^3)**
421	0.48	701	0	0	1058	0	0
		490.7	30	198.7	1058	0	0
					793.5	25	223.97
					529	50	447.93
					264.5	75	671.90
					0	100	895.87

3.3 Cast, curing and testing

Cube specimens (100*100*100 mm) were cast for CS, sulfate and acid attack test while cylindrical specimens (100 mm in diameter and 200 mm in length) were cast for TS. The specimens were water cured till 90 days in a 'temperature-controlled curing tank' to avoid any disruption in hydration process due to temperature variation. The slump test was conducted in accordance with IS 1199 Part 2 for determination of workability (Standard 1199, 2018). The CS was calculated using 'Indian Standard-IS 'Methods of testing for strength of concrete – IS 516-2018'. The CS tests were performed till 90 days of curing with applied rate of 14 N/mm2 per minute. Similarly, the TS was calculated using 'IS [IS-516:2018 Part 1]' on cylindrical specimens [24]. The sulfate attack tests were performed using ASTM C1012-04. After desired water curing (28/90 days), specimens were removed from curing tank and the mass of each specimen was measured before the test.

After 28 days of watering, the cubical specimens (100x100x100mm) were subjected to a sulfate attack test by submerging in '5% sodium sulfate (Na_2SO_4)' solution. After desired immersion days, the variation in mass and corresponding CS and TS was assessed. By comparing the change in observed CS/TS and mass of specimens before and after exposure, sulfate resistance was evaluated. Likewise, acid attack test was performed using 'ASTM C1898-20' wherein the deterioration of specimens was measured after immersing the specimens in 'sulphuric acid (H_2SO_4)'. After 28 days of water curing, the specimens were removed from the curing tank and permitted to dry for 6-8 hours. The mass of the specimens was measured before immersion in acid. After 28/90 days curing, specimens were subjected to dipped in 5% H_2SO_4 solution. Till 90 days, the water was changed in every two weeks in order to keep the pH constant. After desired period, the loss in CS/TS and change in mass was assessed.

4. Results and discussions

4.1 Workability/slump flow tests

The workability of designed NTC was determined with aid of slump cone test as per guidelines of '[25]'. The targeted workability criterion was duly complied in terms of slump values as the same were in range of 42- 68 mm (Figure-3). Mix containing 100% RCA (IS30RCA100) shows minimum slump value (highest decrease) while NTC mixes IS30RCA50 and IS30RCA75 resulted in decrease of 10.9% and 21.8% compared to base concrete (IS0RCA0). The drop was ascribed to rougher texture, presence of porous and higher water absorption of clinging mortar. Higher absorption and rough texture ultimately result in lower workability. However, NTC with 30% of IS and 25% of RCA resulted in marginal higher but comparable slump value to that of base mix (IS0RCA0). Further, NTC mix IS30RCA0 resulted in peak slump value (almost 6.25%) greater than the base mix. Such enhancement in workability was accredited to more-finer and spherical nature of IS. This behavior implied the positive effect of IS (due to its physical character) despite the presence of RCA (25%).

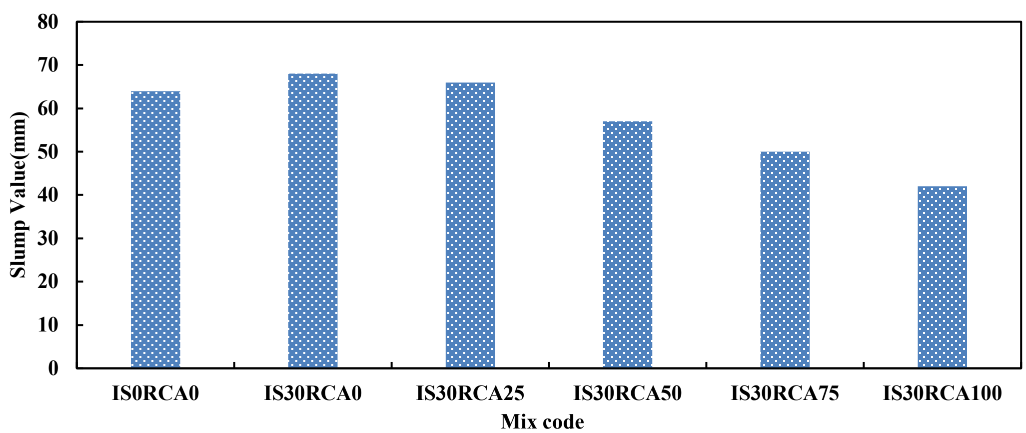


Fig. 3: Workability/slump tests results for base and NTC mixes

4.2 Compressive and tensile tests

The CS for the designed mixes with varied RCA levels (0%, 25%, 50%, 75%, and 100%) along with a consistent mass of IS was evaluated after 7, 28, and 90 days watering (Figure-4). The 28-days CS in NTC mixes 'IS30RCA25, IS30RCA50, IS30RCA75 and IS30RCA100' was decreased by 5.6%, 20%, 23% and 31.8% in comparison to base concrete (IS30RCA0). Similarly, the CS at 90 days was also on lower side. Though, IS as NFA replacement (IS30RCA0) in concrete lead to in increase for CS of 28 (24.2%) and 90 (12.9%) days on comparing with base mix (IS0RCA0). This indicated that 30% of IS with NCA yielded superior outcomes than NTC mixes with RCA. The strong natured, much finer and spherical profile of IS particles was an apt reason such variation in CS values.

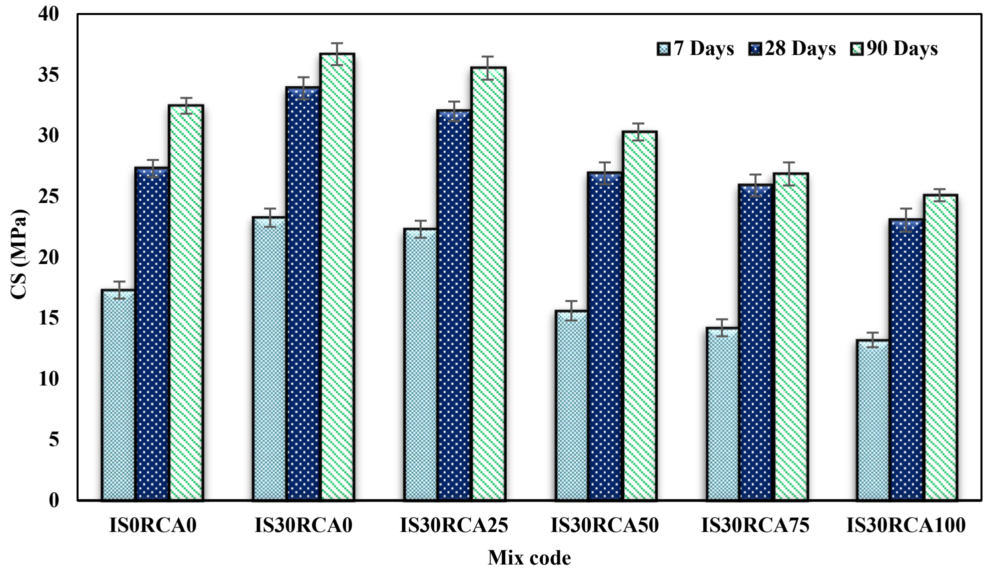


Fig. 4: CS result of IS-RCA based concrete

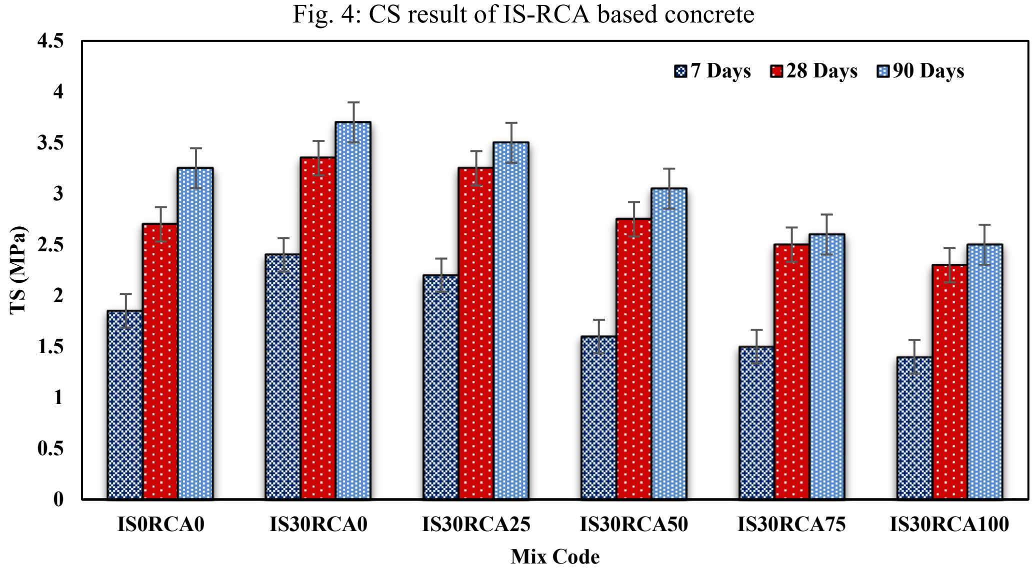


Fig. 5: TS results of IS-RCA based concrete

Similar curing ages were adopted for TS tests and corresponding results were compiled (Figure-5). The results of TS tests also indicated the regular decline identical to CS results. The highest fall in TS values reached up to 34% for NTC mix IS30RCA100 while the least difference was noted for around IS30RCA25 (5.4%) till 90 days water curing as usual comparison with base concrete (IS30RCA0). However, inclusion of IS alone enhanced the TS of concrete mix, as 30% of IS and 100% of NCA resulted up to 11% higher TS values after 90 days. The faulted ITZ (at higher levels) of RCA and stronger IS particles (for lower levels) created dense microstructure yielded in ample variation of TS in NTC mixes.

4.3 Sulfate and acid attacks tests

Cubic specimens (100mmx100mmx100mm) were cast to estimate the effect of sulphate attack in mass variation. For accuracy, at least three specimens of each NTC mix at each curing age were tested. After finishing of defined (28/90 days) curing process, the specimens were allowed to submerge in 5% sodium sulphate (Na_2SO_4) solution. During the entire testing, it was assured that the pH of the solution remains constant (2.0), hence at regular intervals the pH was checked. For determination of mass variation and degradation of each specimen, the 'initial mass and the final mass' [specimen after the immersion/submergence duration (28/90 days)] was noted. From the Table-3 and Table-4, it has been observed that for all designed NTC mixes a gain in mass and corresponding increase in CS with immersion period of Na_2SO_4 solution was noticed. For both curing ages (28 and 90 days), NTC mix IS30RCA100 exhibits the highest gain in mass compared to other NTC and base mix(s) respectively. Correspondingly, the CS of designed NTC mixes was increased with increasing amount of RCA but the rate of increase kept on decreasing. This behaviour may be explained from the fact of development of additional ettringite crystals due to interaction of hydrates [$Ca(OH)_2$ and Na_2SO_4]. In fact, the admittance of salts into pore filled specimens in the presence of pore solution, combine with $Ca(OH)_2$ and sulfate precipitates in the microstructure, ultimately leading to increase in mass and subsequent CS of the NTC mixes [26].

Table 3: Variation of mass due to sulphate attack

28 days curing				**90 days curing**		
Mix Description	**Mass(g)**		**% increase**	**Mass(g)**		**% increase**
	Before immersion	**After immersion**		**Before immersion**	**After immersion**	
IS0RCA0	2575	2591	0.62	2662	2701	1.46
IS30RCA0	2515	2553	1.51	2572	2635	2.44
IS30RCA25	2325	2381	2.40	2418	2496	3.22
IS30RCA50	2200	2264	2.90	2368	2470	4.30
IS30RCA75	2181	2256	3.43	2266	2386	5.29
IS30RCA100	2151	2241	4.18	2228	2359	5.87

Table 4: Variation of CS due sulphate attack

28 days curing				**90 days curing**		
Mix Description	**CS (MPa)**		**% increase**	**CS (MPa)**		**% increase**
	Before immersion	**After immersion**		**Before immersion**	**After immersion**	
IS0RCA0	24.1	24.94	3.48	25.1	26.31	4.82
IS30RCA0	25.93	26.74	3.12	26.63	27.8	4.39
IS30RCA25	26.23	27.03	3.04	27.10	28.14	3.83
IS30RCA50	24.22	24.91	2.84	25.17	25.81	2.54
IS30RCA75	21.89	22.31	1.91	22.96	23.38	1.82
IS30RCA100	20.88	21.12	1.14	22.14	22.48	1.53

On similar pattern as that of abovementioned for sulphate tests, the mass and variation in CS caused by acid attack was estimated for the defined periods. This time, the cured specimens were submerged into 5% potential of sulphuric acid (H_2SO_4) 90 days, with maintained pH of the solution (2.0). The initial and the final mass was measured and the parallel degradation was noted. The effect of sulphuric acid on NTC mixes in terms of variations in mass is shown in Tables-5 and Table-6.

In 5% H_2SO_4 solution, the NTC mix IS30RCA0 exhibits the strongest resistance against mass loss and parallel reduction in CS at 28 days, while NTC mix IS30RCA100 exhibits lowest resistance against the attack. A similar pattern was noted for after 90 days of cured specimens of all designed NTC mixes. Undoubtedly, the attack of H_2SO_4 turned the concrete matrix weak, consequently reduce the size of specimens due to forfeiture of cement paste. The weaker aggregates (RCA) become further weaker during direct contact of acid in spite of the presence of stronger material (IS). Moreover, the rate of chemical reaction due to acid become much faster due to higher quantity of chemically active old clinging mortar present on RCA. Such action resulted into drastic change in CS values of designed NTC mixes compared to base concrete (Tables-5 and Table-6).

Table 5: Variation of mass due to acid attack

28 days curing				90 days curing		
Mix description	Mass (g)		% reduction	Mass (g)		% reduction
	Before immersion	After immersion		Before immersion	After immersion	
IS0RCA0	2411	2294	4.85	2418	2209	8.64
IS30RCA0	2524	2389	5.34	2527	2261	10.52
IS30RCA25	2571	2421	5.83	2534	2261	10.77
IS30RCA50	2558	2357	7.85	2587	2245	13.21
IS30RCA75	2496	2245	10.05	2463	2086	15.34
IS30RCA100	2422	2143	12.24	2540	2106	17.08

Table 6: Variation of CS due to acid attack

28 days curing				90 days curing		
Mix description	CS (MPa)		% reduction	CS (MPa)		% reduction
	Before immersion	After immersion		Before immersion	After immersion	
IS0RCA0	23.81	22.2	6.76	25.66	23.21	8.94
IS30RCA0	26.79	24.61	8.13	29.8	26.61	10.70
IS30RCA25	26.33	23.89	9.26	27.5	24.35	11.45
IS30RCA50	24.11	21.56	10.57	24.7	21.13	14.45
IS30RCA75	21.63	19.23	11.09	22.30	18.63	16.45
IS30RCA100	20.36	17.73	12.91	23	18.73	18.56

5. Apposite NTC Mix For Optimum Performance

Based on discussions in previous sections, some of the NTC mixes fall in the category of ‘inferior quality’ in contrast to base concrete while few of them performed satisfactory. For few cases equivalent the levels as that of base concrete were achieved. Herein, it may be understood that the aim is to develop sustainable NTC mix while incorporating/utilizing the maximum amount of left-overs of industry. Experimental results directed that NTC mix containing IS only has best values for CS, TS, sulphate and acid resistance while NTC containing IS and limited amount of RCA (25%) exhibited alike to base concrete. Hence, NTC mixes follow the order of performance as: IS30RCA0 > IS30RCA25 > IS0RCA0 > IS30RCA50 > IS30RCA50 > IS30RCA75> IS30RCA100 for CS and TS. Similarly, for sulphate and acid attack resistance the opposite order is of: IS0RCA0 > IS30RCA0 > IS30RCA25 > IS30RCA50 > IS30RCA50 > IS30RCA75> IS30RCA100. Out of above, NTC mix IS30RCA25 has been selected as apposite designed concrete compared to base concrete.

6. Conclusions

i) The workability of all designed NTC mixes was decreased (up to 22%) due to inclusion of IS and RCA. The adhered mortar absorbs the available water making the NTC mixes drier.

ii) The 28 days CS was decreased up to 8% for NTC mix IS30RCA100 while minimum reduction of 5.5% was noted for NTC mix IS30RCA25. Parallel, the TS results followed the identical patterns for the designed NTC mixes.

iii) Sulphate attack results indicated the variation of mass and CS of the designed NTC mixes. The highest increase in mass was noted for NTC mix IS30RCA100 (around 6%) while the least increase was noticed for mix IS0RCA0. The corresponding variations were noticed CS of the specimens for all the NTC mixes. Likewise, acid attack tests results indicate the deterioration of NTC mixes in terms of mass, as maximum change was noted for the abovementioned concrete (17%) while the least reduction was seen for base mix IS0RCA0 (8.5%).

iv) Due to inherent conduct the stronger nature of IS and weaker performance of RCA significantly affect the resistance values against sulphate and acid attacks. Considering extreme exposures, the study encourages the inclusion of IS and RCA in limited content in order to conserve resources and for promotion of sustainability.

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