An Experimental Investigation on Durability Properties of Reactive Powder Concrete

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ABSTRACT

The durability of concrete subjected to an aggressive environment is a major issue faced by constructional engineers. Many researchers have tried to evaluate the durability characteristics of concrete against aggressive fluids. Amongst the fluids, compounds of sulphuric acid and chloride ions caused massive deterioration in concrete. Reactive powder concrete (RPC) is a type of ultra-high-strength cement composite. In the present study, an effort is made to assess durability properties of RPC. The RPC with compressive strengths 110, 120 and 130MPa have been produced. Acid immersion test, salt crystallization test and chloride ion penetration tests have been carried out to assess the degradation of concrete. Samples were exposed to sulphuric acid solutions over a period of 60 days with concentrations varying from 0.5-2%. The salt crystallization tests were carried out by immersing samples in 14% Na₂SO₄ solutions. Visual observations and deterioration in terms of mass and compressive strength reductions are recorded. The RPC showed high resistance towards the crystallization of salts. A significant amount of weight loss and strength loss was observed for the samples exposed to high concentrations of sulphuric acid. A negligible amount of chloride ion penetration was observed.

doi: 10.5829/ije.2022.35.02b.08

1. INTRODUCTION

1.1 General

The durability of concrete may be defined as the ability of a material to retain its physical, chemical and visual performance characteristics in the intended environment, within a reasonable tolerance for an economic life expectancy. Early age deterioration is observed in concrete subjected to an aggressive environment. The deterioration is caused by chemical attacks and acid rain precipitating in the form of dissolved acids. The degree of decay depends mainly upon the physical and chemical characteristics of concrete.

1.2 Durability Studies on Concrete

The concrete used in the construction of chemical industries and sewer lines is subjected to the acidic environment. A massive deterioration of concrete is observed due to the action of compounds of various acids such as sulphuric acid, nitric acid, hydrochloric acid etc. An investigation was carried out to study the durability properties of concrete [1]. The concrete was exposed to 0.1%, 1% and 5% concentrations of sulphuric acid. The factors for strength and weight reduction were provided depending on the depth of sulphuric acid penetration. Due to the high alkalinity of portland cement and chemical reactions

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of such acids, corrosion is induced in the concrete, which leads to the deterioration of structure [2]. The effect of sulphuric acid attack on normal strength concrete (NSC) was studied by immersing cylindrical specimens in 1% sulphuric acid solutions. The degree of deterioration was expressed in terms of changes in weight and thickness of specimen. The measurement was obtained for 10, 20, 30 and 70 days immersion periods. The results indicated that the volumetric expansion in specimens was in the range of 5-10%. The weight loss was observed to be 15-20%. Silica fume and fly ash were added as supplementary cementitious materials in reinforced cement concrete (RCC) [3]. It was observed that, the addition of supplementary cementitious material as a replacement for aggregates and cement content reduced the carbonation depth. Significant resistance towards chloride ion content was also observed. The degradation due to chemically and microbiologically induced corrosion was studied on high sulphate resistant portland cement concrete and polymer modified concrete [4]. Addition of polymers increased expansion in concrete along with increased loss of material. Acid resistance of self-compacting concrete (SCC) was compared with conventional concrete for 1% of sulphuric and hydrochloric acid solutions [5]. The SCC has high resistance to sulphuric acid attack than compared to conventional concrete. A laboratory investigation on portland cement systems exposed to aggressive environments was carried out to predict the durability characteristics [6]. The results indicated that the type of cement and water-binder (w/b) ratio have significant effect on the extent of degradation. It was suggested to lower the w/b ratio to increase durability.

High volume fly ash concrete (HVFC) was produced by replacing cement with 10-70% of ACTM class C - fly ash [7]. The loss in weight, compressive strength and split tensile strength were obtained for 5% H₂SO₄ solutions over the period of 90 days. A higher rate of deterioration was observed for steam cured samples than compared to normal cured samples. The durability properties of geopolymer concrete (GPC) with compressive strengths 53MPa and 62MPa were studied to understand the effect of sulphuric acid attack [8]. The mass reduction was observed to be 40% after 4th week. The compressive strength loss was in the range of 32-37%. An air entrained metakaolin concrete with a lower w/b ratio and autoclave curing showed better durability properties towards sulphate attack [9]. The anaerobic bacteria present in sewer pipes produce hydrogen sulphide (H₂S) under low pH values. In the presence of oxygen and moisture, this dissolved H₂S is then oxidized to form sulphuric acid, which leads to corrosion in concrete [10]. ASTM class - F fly ash 20% by weight and silica fume 8% by weight of cement were added to produce ternary blended concrete [11]. M20, M30 and M40 grade concretes were produced and durability studies were carried out for 5% H₂SO₄ and 5% HCL solutions. A maximum of 30-40% mass loss was observed with the strength values reducing up to 60-70%. The performance of GPC under 10% H₂SO₄ and 10% MgSO₄ solutions was evaluated [12]. The results for 15-45 days exposure period, indicated that heat cured GPC samples show better durability characteristics than conventional concrete. Change in color from dark grey to lime grey with deposits of whitish compounds of burite on concrete exposed to seawater was observed [13]. The solutions of inorganic and organic chemicals were used to evaluate flexural behaviour of polyester polymer concrete [14]. After two months of exposure, an average loss of 35% in flexural strength was observed for inorganic acids. The durability of concrete was improved by addition of glass fibres. The fibres reduced the shrinkage cracks by 40% and hence permeability of chloride ions was observed to decrease [15]. An environmentally friendly green concrete (GC) was produced by replacing cement with 20% alccofine [16]. The pore structure was improved due to addition of alccofine which in turn decreased the water absorption. Investigations were carried out to evaluate durability performance of SCC [17 -19]. Different compositions of SCC were produced by incorporating fly ash, alccofine, nanoparticles of aluminium oxide, super absorbent polymers and glass fibres. The results showed that partial replacement of pozzolanic material increased the durability properties to a greater extent along with an improvement in mechanical properties.

1.3. Reactive Powder Concrete Reactive powder concrete (RPC) is a type of ultra-high strength concrete, with a strength greater than 80MPa. It was first developed in France [20]. The optimum granular mixture was achieved by addition of superfine silica fume as a pozzolanic material and by eliminating coarse aggregates. Steel fibre, 1.5-3% by volume, were added to enhance the ductility. The application of heat and pressure during curing resulted in producing RPC with strengths 200-800MPa. The durability studies for RPC with strengths 200MPa was studied by performing various tests [21]. The durability properties were assigned by measuring porosity, water absorption, chloride ion penetration, carbonation and corrosion of reinforcement. The influence of coarse aggregates on mechanical properties of RPC was studied by replacing fine sand with natural, well graded coarse aggregates of maximum size 8mm [22]. The results indicated the compressive strength did not change due to partial replacement of fine quartz sand with coarse aggregates. When the fine quartz sand was fully replaced with the coarse aggregates, the homogeneity and effective bond strength between cement matrix and aggregates was reduced. As a result, the compressive strength and flexural strength was observed to decrease. The effect of water, steam and autoclave curing on production of RPC
with various proportions of steel fibre content was investigated [23]. The results concluded that ultra-high strength concrete (200-300MPa) can be produced with commonly available materials and traditional curing methods. The durability properties of steel fibre reinforced RPC with strength 180MPa were studied by performing the acid tests, accelerated corrosion tests and rapid chloride penetration tests [24]. The mass was reduced by 20% and strength was reduced by 60% for 60days exposure period. The mechanical properties, stress-strain under uniaxial compression and flexural loads, along with frost resisting durability properties were investigated for RPC used for construction of highway bridge [25]. An extensive investigation on the production of RPC was carried to study the effect of mixing method, mixing duration and speed, dosage of polypropylene fibres, and effect of different curing regimes [26-30]. The effect of the dosage of superplasticizer and quartz powder on mechanical properties was studied [31]. An optimum of 15% of quartz powder with cement content 900 kg/m³ and 1000kg/m³ resulted in RPC with strengths 160MPa and 170MPa, respectively. A study was carried out to understand the effect of different curing temperatures on properties of RPC. An optimum temperature of 90°C was obtained and RPC with compressive strength 111.43MPa was produced [32].

1.4. Research Significance In recent years, the applications of reactive powder concrete are increasing in the construction of bridges, harbours, seashore structures, nuclear powerplants, etc. These structures are subjected to severe environmental changes which affect the durability properties of materials used for construction. Many investigations have been carried out to study the durability properties by considering the effect of various acids. A vast amount of literature is available for normal strength, high strength, self-compacting, geopolymer, green concrete etc. However, there are limited researches on durability studies of RPC. The present research focuses on producing an ultra-high strength reactive powder concrete with compressive strength 110MPa, 120MPa and 130MPa. The durability studies are carried out by performing acid tests for various concentrations of sulphuric acid, salt crystallization tests and rapid chloride ion penetration tests. The results obtained will help the construction engineers to predict and assess the degree of deterioration based on visual and physical inspection.

2. EXPERIMENTAL PROGRAM

Three grades of reactive powder concrete mixtures are produced with locally available materials. The constituents for mix proportions are 53grade ordinary portland cement (OPC), river sand, quartz powder (QP) and water. Silica fume (SF) is added as a pozzolanic material. To get a workable mix, superplasticizer (SP) Glanium-8233 is used. The mix proportions have compressive strengths in the range of 110MPa (M1), 120MPa (M2) and 130MPa (M3). The details of mix proportions have been provided in Table 1. The durability aspects are studied by conducting various laboratory tests. The tests conducted are:

- Acid immersion tests
- Salt crystallization tests and
- Rapid Chloride Penetration Test (RCPT).

2.1. Acid Immersion Test For immersion of RPC samples, special acid resistant trays of size 750mmx650mmx50mm were used. The arrangement of specimens is shown in Figure 1. Separate specimens are used for the different observation periods which varies from 3, 7, 28 and 60 days. For each concentration of sulphuric acid, there were four trays which includes three grades of concrete. After every observation period, the samples were removed from the respective solutions and thoroughly washed with pure water, oven-dried at 60°C and then cooled for taking further observations to measure the degradation. Due to the decay of samples and change in environmental conditions, the concentration of the solution may vary; therefore, care is to be taken to maintain the pH value constant throughout the period of exposure with the pH meter. After removal, weight loss due to the acid effect was evaluated, hence the extent or rate of deterioration is

| Mix | Sand | W/B ratio | SF Kg/m³ | QP Kg/m³ | SP Kg/m³ | % |
|-----|------|-----------|----------|----------|----------|---|
| M1  | 954.68 | 0.21 | 45 | 5 | 90 | 13.5 | 1.5 |
| M2  | 910.26 | 0.20 | 90 | 10 | 90 | 18 | 2.0 |
| M3  | 875.87 | 0.19 | 135 | 15 | 90 | 22.5 | 2.5 |

Figure 1. Specimens immersed in H₂SO₄ solution.
known. The compressive strength after decay was obtained and compared with initial strength. The percentage of reduction in strength is calculated. The percentage of losses in strength and weight were calculated from equations 1 and 2.

\[
\text{Reduction in compressive strength (\%)} = \left( \frac{f'_{c28} - f''_{c-AO}}{f_{c28}} \right) \times 100
\]

\[\text{Percentage mass loss} = \left( \frac{M_2 - M_1}{M_1} \right) \times 100\]  

(1)

(2)

2. 2. Salt Crystallization Test  
The test was conducted on untreated reactive powder concrete specimens. The samples were cleaned and oven dried, which were then immersed in 14% Na$_2$SO$_4$ solution for 18 hours at room temperature as shown in Figure 2. After the required immersion period, samples were removed and allowed to drain for half an hour. Later they were dried in oven for 4 hours at a temperature of 105 ± 5°C and then cooled for one hour at room temperature. That forms one cycle of 24 hours and the same was repeated for the required number of cycles.

For the evaluation of the progressive change in the physical and durability characteristics a sample of three specimens were taken out at every five cycles. All such specimens withdrawn were thoroughly washed to remove the salt deposited and then cooled and weighed. Weight losses and compressive strength values were calculated.

2. 3. Rapid Chloride Penetration Test  
The specimens of required thickness were obtained by slicing cylinders of size 100 mm diameter and 200 mm length. A water-cooled diamon saw cutter was used to cut the samples. The diameter of each specimen is 100mm with a thickness of 50mm. These samples were subjected to 60V DC voltage across their thickness. It was applied between two cells containing sodium chloride (3% NaCl) and sodium hydroxide (0.3M NaOH) solutions. Tests were carried out for a duration of 6 hours. To avoid any leakage of solution from the cell, the surface was cut at a right angle to the length of cylinder and was perfectly plane. The laboratory setup is shown in Figure 3. RCPT setup consists of mainly three functional units; which are specimen conditioning unit, sample holding cell and measuring unit. The microprocessor based electronic unit provides a constant 60 ± 0.1 V DC voltage across the two circular surfaces of the test specimen. The measurement of total electrical charge in terms of Coulombs is also done by the same unit. It also displays the set time, test time duration and remaining time for completion of the test. The total charges passed through the specimen are obtained after 360 minutes.

3. RESULTS AND DISCUSSION

3. 1. Acid Test Results  
The 28 days cured samples of RPC were immersed in dilute sulphuric acid solutions of different concentrations, thereby creating the actual media which happens in the environment. The concentration of solution is varied in the range of 0.5, 1, 1.5 and 2%. The samples were removed at various periods of exposure and tested. The compressive strength, weight loss and strength variation with exposure intervals of 3, 7, 28 and 60 days, were obtained.

3. 1. 1. Visual Observations  
For visual observations, the shape of specimens is taken into account. It was observed that samples immersed in different concentrations exhibit different types of decay. After 3 days of exposure period, there was no change in shape, also the samples exhibit a significant resistance to any scaling and spalling of concrete (Figure 4).

At 7 days of exposure, a negligible amount of surface erosion in the form of white deposits is seen to occur at 0.5 and 1% solutions. It is shown in Figure 5. At 1.5 and 2% concentration discolouration of specimens takes place from grey to white. For samples immersed in 1.0, 1.5 and 2% solutions we can see scaling or peeling of concrete where top surface layer of samples get eroded which goes on increasing with the increase in concentration. Some specimens show reddish-brown colour on the top surface with damage to edges. The

![Figure 2. Specimens immersed in Na$_2$SO$_4$ solution](image)

![Figure 3. Rapid Chloride ion Penetration Test](image)
scaling indirectly leads to change in shape, thereby reducing strength carrying capacity.

The samples for normal strength concrete (NSC) immersed in 1% $\text{H}_2\text{SO}_4$ solution over the period of 28 days show disintegration and peeling of the surface layer [5]. The appearance of white pulpy mass over external surface was seen. For SCC samples, expansion and peeling is observed. After 28 days of exposure period, the RPC specimens get degraded severely than 7 days. The samples exposed to 0.5% solutions show scaling of concrete from all four sides with peeling of edges. The samples also exhibit discolouration from grey to light greenish but no spalling of concrete is observed. The spalling of concrete is seen in 1.0, 1.5 and 2% solutions in 28 days and specimens are eroded from all four sides by forming white deposits surrounding the cubes as shown in Figure 6.

Samples placed in for 60 shows that even at 0.5% solution of acid there is a considerable scaling of concrete which is absent in 7 and 28-days immersed specimens. From the clean observations, we can judge that scaling in the samples of M1 is predominant when compared to M2 and M3, shown in Figure 7.

In the remaining solutions, samples have lost a significant amount of material from surface, edges and original shape is completely disrupted. Spalling is predominant with discolouration of reddish-brown on the top surface. It is shown in Figure 8.

3. 1. 2. Physical Observation

For 1% $\text{H}_2\text{SO}_4$ solution and 28 days exposure period the weight loss observed for NSC is 11% [1], SCC is 5% [5], HVFC is 5% [7] and GPC is 3% [13]. It is observed that the rate of weight loss decreases with an increase in grade of reactive powder concrete. The maximum weight loss at 28 days was observed to be less than 1% for all the grades of RPC considered. But a progressive increment is obtained in weight loss with an increase in exposure time and concentration of solutions. The samples for M1 show significant weight loss compared to M2 and M3. The maximum amount of weight loss obtained are, M1-13.98%, M2-10.09% and M3-8.38%, respectively, at 60 days exposure and 2% solutions. The weight loss obtained, for considered exposure time and different percentages of solution, are shown in Figures 9, 10 and 11 for M1, M2 and M3, respectively.

3. 1. 3. Compressive Strength

From the experiments conducted with different conditions, it is observed that samples lose their visual and physical properties for longer duration of exposure and higher concentrations of solutions, which in turn reduce the compressive strengths of RPC. For 1% $\text{H}_2\text{SO}_4$ solution and 28 days exposure period the strength loss observed
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Figure 9. Variation of reduced weight for M1

Figure 10. Variation of reduced weight for M2

Figure 11. Variation of reduced weight for M3

Figure 12. Variation of Compressive strength for M1

Figure 13. Variation of Compressive strength for M2

Figure 14. Variation of Compressive strength for M3

for NSC is 70% [1], SCC is 62% [5], HVFC is 58.5% [7] and GPC is 40% [13]. All the specimens of RPC show that there is a significant reduction in compressive strength. For samples immersed for 3 days show small amount of strength reduction i.e. in the range of 9-14%, whereas 7 days and 28 days samples show a considerable amount of strength reduction. It is in the range of 25-

35%. This shows that RPC has better resistance to sulphuric acid attack compared to other types of concrete. The maximum amount of strength loss is observed at 60 days samples in the range of 45-70%. The reduction of compressive strengths are shown in Figures 12-14.

3.2. Salt Crystallization Test (SCT)

3.2.1. Visual Observations Sodium sulphate crystallization is initiated for samples kept in Na$_2$SO$_4$ solution for 5 days cycle. It shows a progressive
development till 20 days cycle, as shown in Figures 15-18. There is not much change in shape or structure of specimens. However, at 20-30 days cycles a clear salt layer of greenish white color is formed on all edges and surfaces of the specimens. These are shown in Figures 19 and 20. Surface erosion and change in shapes have been observed. The effective surface area is reduced. Cracks are initiated at edges of surface and continue to propagate throughout the body of the samples. From 35 days samples, it is observed that the samples have grown a significant amount of salt crystalline layer. Due to the cracks initiated in early cycles, the samples show a significant amount of scaling over the surface. The edges are distorted and discoloration of samples was observed. The effects are shown in Figure 21.

3.2.2. Physical Observations

The results of durability tests show that the rate of decay is not uniform. It is slow up to 20 cycles and increases rapidly after 20 cycles. At five days of durability cycles, the initial reduction in weight is 0.06-0.08%, where very slight appearance of salt crystalline layer is observed. At 15 days cycle weight loss observed is 0.16-0.19% which is 60% greater than five days cycle. Similarly, the percentage of weight loss at 20 days cycle obtained is 0.25-0.30%, which is almost 75% greater than five-day cycle. From Figure 22, it is clear that the samples exhibit similar behaviour of weight reduction till 25 days cycle and after that, the curve shows a steep increase. The maximum weight reduction for M1, M2 and M3 are 1.52%, 1.29% and 1.11%, respectively. The reduction in weight decreases as the grade of concrete is increased.

3.2.3. Compressive Strength

At the initial stages of cycles, the reduction in compressive strength shows a linear behavior as shown in Figure 23. At 15 days cycle, a clear salt layer appears with increased intensity of salt at all sides of the sample with a 10% decrease in strength for all the grades of concrete. The strength reduction is up to 13-16 MPa at 20 days samples. The decrease in magnitudes of water absorption and weight takes place progressively. At the end of 35 cycles the strengths are reduced up to 28-34 MPa, which is nearly one fourth of the original strength with huge amount of formation of crystalline salt layer as shown in Figure 21. The variation of strength with durability cycle is shown in Table 2.
3. 3. Rapid Chloride Ion Penetration Test (RCPT)

For this experiment, 3 samples were cast for each grade of concrete. The M1 is cast with a 0.21, M2 with 2.0 and M3 with 0.19 water cement ratio, respectively. This test is carried out to know the effect of water-cement ratio on chloride permeation. The average passed charges (in coulombs) through all the mixes is noted and presented in Table 3.

### Table 3. RCPT test results for 3 grades of RPC

| Samples          | W/C ratio | Charges passed | Average passed charges | Chloride ion permeability |
|------------------|-----------|----------------|------------------------|---------------------------|
| M1 (110MPa)      | 0.21      | 141            | 144                    | Very Low                 |
| M2 (120MPa)      | 2.0       | 109            | 112                    | Very Low                 |
| M3 (130MPa)      | 0.19      | 79             | 82                     | Negligible               |

From the results, it is clear that the RPC samples with higher water-cement ratios show higher charges passed when compared low water-cement ratio. The RPC with grade 130 MPa (M3) exhibits negligible chloride ion penetration showing homogeneous and dense microstructure. RPC with grades 120MPa (M2) and 110MPa (M1) shows very low chloride ion penetration.

4. CONCLUSIONS

Based on the experimental investigation on RPC specimens for durability studies, the following conclusions are drawn.

The durability aspects have been assigned by carrying out visual observations and reduction in weight and compressive strengths of RPC samples.

1. The acid immersion tests with different concentrations of H$_2$SO$_4$ and Na$_2$SO$_4$ solutions have resulted in the determination of decay caused by acid attacks and crystallization of salts.

2. The lower concentration of sulphuric acid (0.5%, 1.0%) have less effect on the durability of RPC. Whereas higher concentration (1.5%, 2%) leads to discoloration, erosion, scaling, appreciable degradation in weight and significant reduction in strength of concrete.

3. At Higher concentration of acid, the RPC specimens show that the compressive strengths are reduced up to 75% of their original strengths.

4. RPC samples exhibit resistance towards salt crystallization up to 20 days cycles. However, at 35 days cycles, disruption of surfaces and edges is observed leading to a huge amount of weight loss and reduction in compressive strengths up to 30MPa.

5. The RCPT indicated negligible to very low charges passing through all the samples. It signifies the high resistance of RPC towards the effects of chloride ion penetration.
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**Persian Abstract**

چکیده

دوام بتن در معرض یک محیط تهاجمی یکی از مسائل مهمی است که مهندسان ساختمان با آن مواجه هستند. سیالات تهاجمی باعث تخریب شدید بتن می‌شوند. بتن پودری راکتیو (RPC) نوعی کمپوزیت سیمانی با مقاومت فوق‌العاده بالا است. در مطالعه حاضر، تلاشی برای ارزیابی خواص دوام RPC انجام شده است. کار تولید سه نوع RPC با مقاومت فشاری 110، 120 و 130 مگا پاسکال انجام شده است. آزمایش‌های غوطه ور در اسید سولفوریک و آزمایش کریستالیزاسیون نمک و آزمایش نفوذ یون کلرید برای ارزیابی خواص دوام RPC انجام شدند. نمونه‌ها در یک دوره 60 روز در محیط سولفوریک با غلظت‌های مختلف از 0.5% تا 2% قرار گرفتند. این آزمایشات نمک و کلرید نفوذ نشان دادند. مقدار ناچیز از نفوذ یون کلرید مشاهده شد.