A critical review on the factors influencing the design of test methods for assessing acid attack in concrete

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Abstract. Concrete being alkaline in nature is highly vulnerable to acidic environment. When concrete is exposed to industrial effluents, the acids in these effluents alter the chemical equilibrium of cement matrix resulting in microstructural deterioration. Currently, there are no standard procedures for assessing the performance of concrete subjected to acid attack. In order to develop such test methods, explicit knowledge about the factors influencing test method for assessing the acid attack is imperative. This paper outlines the need to develop such test methods and also classifies the factors influencing the selection of test methods as test condition related factors, specimen related factors and acid related factors. Test condition related factors comprises of the testing environment adopted for the study. The test should simulate the actual conditions and type of attack expected to occur at the field, which can be achieved through replenishment of acid solution, alternate wetting and drying, abrasion etc. Specimen related factors deal with the physical properties of the specimen used whereas the acid related factors include various aspects of acid chosen for the study other than its material properties. As the surface area to volume ratio of the specimen increases, more surface area is exposed to the chemical attack, which hastens the rate of deterioration. Also, the ratio of volume of acid solution to specimen enhances rate of concrete degradation. Hence, many factors together have to be considered and studied properly while designing a test method for assessing acid attack.

1. Introduction
Concrete, being the most abundantly used construction material, is subjected to various aggressive environments throughout its life. Acid attack is one of the major causes of deterioration in concrete, which includes attack by strong inorganic acids in industrial structures and attack by organic acids when exposed to agricultural and agro-food industries. The underlying mechanism behind acid attack is that concrete being alkaline in nature is highly vulnerable to acidic environment, which alters the chemical equilibrium of cement matrix in concrete resulting in microstructural deterioration. While organic acids are considered to be destructively inferior to inorganic acids, they are harmful to the cement matrix and enhance embedded reinforcement corrosion and disturb the facilities of industrial production. For example, because of the acid buffer action and the high solubility of the reaction products, organic acid such as acetic acid have a significant damaging impact on concrete when compared to strong acids such as sulphuric or hydrochloric acid [1]. In terms of basic degradation mechanisms and aggressiveness, the action by weak biological acids like acetic acid is similar to that of strong mineral acids such as hydrochloric and nitric acids [2].

Concrete is used to construct structures designated for effluent processing, collection, storage and treatment as it meets specifications such as economical, watertight, ensures good thermal inertia and
complies with health standards placed in the agro-food industry [3]. There is a wide variety of organic acids responsible for acid attacks on concrete such as liquid manure and silage juices from livestock rearing activities, also waste water from agro-food industries, such as whey and white waters released from the dairy industries, molasses as well as vinasses from the distilleries, sugar and fermentation industries. A list of different sources of organic acids that are capable of attacking concrete is given in Table 1.

| Industry            | Wastewaters | Acids         | Concentration (mmol/L) | pH  |
|---------------------|-------------|---------------|------------------------|-----|
| Dairy industries    | Whey        | Acetic        | 14.0                   |     |
|                     |             | Citric        | 9.2–9.6                |     |
|                     |             | Lactic        | 3.4–5.7                |     |
|                     |             | Propionic     | 1.0                    |     |
|                     |             | Butyric       | 0.6                    |     |
|                     |             | Total (max.)  | 30.9                   |     |
| Wine industry       | Vinasses    | Tartaric      | 3.2–8.73               | 4–6 |
|                     |             | Acetic        | 3.1–5.0                |     |
|                     |             | Lactic        | 2.2–5.6                |     |
|                     |             | Succinic      | 0.3–6.8                |     |
|                     |             | Malic         | 0.5–0.8                |     |
|                     |             | Total (max.)  | 26.9                   |     |
| Sugar industries    | Sugar cane vinasses | Lactic | 18–80.4             |     |
|                     |             | Glycolic      | 12–25                  |     |
|                     |             | Citric        | 1.7–10.4               |     |
|                     |             | Trans-Aconitic| 0.2–4.8               | 4-7 |
|                     |             | Cis-Aconitic  | 0.1–2.8                |     |
|                     |             | Oxalic        | 0.08–0.8               |     |
|                     |             | Fumaric       | 0.1–0.4                |     |
|                     |             | Total (max.)  | 124.2                  |     |
| Animal rearing      | Silage juice| Lactic        | 55.3–63.7              | 4–5 |
|                     |             | Acetic        | 25.3–28.3              |     |
|                     |             | Butyric       | 43.4                   |     |
|                     |             | Propionic     | 3.1                    |     |
|                     |             | Total (max.)  | 99.1                   |     |
|                     | Liquid manure| Acetic,       | 31.7–213               | 5-8 |
|                     |             | Butyric       | 1.1–28.8               |     |
|                     |             | Propionic     | 5.4–37.8               |     |
|                     |             | Total (max.)  | 279.6                  |     |

Table 1. Major organic acids from agro-food industries [4]

Table 1 exhibits the immense exposure of concrete to organic acids, which highlights the significance of understanding the degradation mechanism of acid attack and also to develop suitable material mixtures that perform well in these aggressive aqueous environments. In order to recognise the performance of these mixtures exposed to acid attack, proper test methods should be adopted. However, there is no standard procedure for analysing concrete exposed to acid attack. Due to the lack of a standardised test procedure, various test methods have been used and various criteria have been modified to determine the resistance of the materials. These tests can be broadly divided into three groups: chemical tests using mineral and organic acids, microbial simulation tests and in situ tests, where the first two are conducted in a laboratory and in situ tests on actual concrete structures exposed to acids.
Chemical tests are used to analyse the degradation kinetics and mechanism of concrete due to chemically synthesized organic and inorganic acids. These tests predominantly examine chemical effects, but physical and mechanical effects can also be studied. For the study, parameters to analyse alteration kinetics such as mass loss, altered depth, change in thickness etc. and characterization techniques such as X-ray diffraction (XRD), scanning electron microscopy (SEM), X-Ray Fluorescence (XRF) etc. can be used.

Microbial simulation tests are mainly conducted for the study of corrosion due to sulphuric acid, nitric acid etc. The significance of this test is that biogenic sulphuric acid (BSA) is the primary cause of corrosion of concrete in sewer systems and waste water treatment plants. In this test, microbes are cultured in a laboratory to simulate the actual condition. Biofilms are developed in the concrete specimen during these tests and molecular techniques can be adopted for studying the same.

In situ tests are conducted by immersing specimens on various real sites where there is active acid attack. Parameters like hydrogen ion consumption rate, mass changes, microstructural tests etc. can be utilised for studying the impact of acid attack and the results can be analysed to understand the severity of attack.

To design standardised test methods, a clear understanding on various aspects like exposure conditions, physical and mechanical effects, specimen requirements, properties of acid to which the materials are exposed etc. are required, which can be generalised as the factors influencing the selection of tests for acid attack.

2. Factors influencing tests for acid attack
According to Ramaswamy et al., the factors influencing acid attack are broadly categorised into material related factors and test related factors [2]. Material related factors include both acid related factors and material (binder and aggregate) related factors. Acid related factors deal with the properties of acid specifically the aggressiveness of acid depending on the type of acid used, its concentration, acid dissociation constant (pKₐ) of acid, poly-acidity of acid and the formation of organometallic complexes. Material related factors comprise of type and proportion of binders, water to binder ratio, binder to aggregate ratio and the mineralogical nature of the aggregates [2].

Table 2. The solubility of major calcium salts of various organic acids [2, 5, 6, 7]

| Acid   | Acid dissociation constant (25°C) | Aggressiveness | Major calcium salts | Solubility (g/L) |
|--------|----------------------------------|----------------|---------------------|------------------|
| Acetic | 4.76                             | Moderate       | Calcium acetate monohydrate $\{\text{Ca(CH}_3\text{COO)}_2\cdot\text{H}_2\text{O}\}$ | 347 (20°C) / Very soluble |
| Citric | 3.14/4.77/6.39                    | Very high      | Calcium citrate tetrahydrate $\{\text{Ca}_3(\text{C}_6\text{H}_5\text{O}_7)_2\cdot\text{4H}_2\text{O}\}$ | 0.95 (25°C) / Slightly soluble |
| Lactic | 3.86                             | Moderate       | Calcium lactate pentahydrate | 79 (30°C) / Soluble |
| Tartaric | 3.04/4.37                       | Moderately low | Calcium tartarate tetrahydrate $\{\text{Ca}_4\text{C}_4\text{H}_6\text{O}_6\cdot\text{4H}_2\text{O}\}$ | Insoluble |
| Succinic | 4.16/5.61                      | High           | Calcium succinate trihydrate | 5.17 (20°C) / Slightly soluble |
| Oxalic | 1.23/4.19                        | Very Low       | Calcium oxalate monohydrate $\{\text{CaC}_2\text{O}_3/\text{CaC}_2\cdot\text{H}_2\text{O}\}$ | Insoluble |

In addition to this, there are factors related to the complex salts formed during the attack. These salt related factors mainly include the solubility of salts formed, molar volume, mesoscopic shape and the
affinity of salt to the cement matrix [4, 5]. The solubility data of major calcium salts that may be formed due to organic acid exposure is provided in Table 2.

The factors influencing the test methods adopted for determining acid attack can be broadly classified into test condition related factors, specimen related factors and acid related factors. Test condition related factors incorporate the settings and arrangements adopted for the study, whereas specimen related factors deal with the physical properties of the specimen used. Acid related factors include various aspects of acid chosen for the study other than its material properties.

2.1 Test condition related factors
The testing environment, in which analysis of degradation due to acid attack is carried out, has an influence on the selection of the test method for evaluating the same. The conditions may represent either real condition which resembles field situation or an accelerated condition in the laboratory. In real condition, the degradation is a prolonged process and may take years to observe minute changes, but accelerated tests are carried out in a more ideal and controlled condition to obtain accelerated results. But the results obtained through accelerated tests may not coincide with that of realistic conditions. The main benefit of performing the experiments in real scenarios is the assurance that there is no alteration in the degradation process.

According to De Belie, in realistic conditions, the lower concentration of aggressive acids can be used along with a sensitive deterioration detection method and an extrapolation method can be used to predict the degradation which can occur in the future [8]. Due to the slow pace of realistic testing, accelerated tests are preferred by researchers, mainly because long term effects due to acid attack on concrete can be analysed in less time and more parameters can be assessed simultaneously to obtain significant and reliable results in less time. For accelerated conditions, rate of degradation can be improved by different approaches such as higher acid concentrations, greater contact surfaces etc.

2.1.1 Test simulation conditions. According to realistic situations simulated in the study, test methods are generally categorised into static test methods and dynamic test methods. In static method, the test conditions are kept unchanged. For example, in static immersion tests, the acid solution initially prepared for the study has not been altered, and the specimens are immersed continuously in this solution till the end of the test. Dynamic methods are tests in which the test conditions are frequently changed.

2.1.2 Replenishment of acid solution. The acid solution in which the specimens are placed may or may not need renewal depending on the real condition to be simulated. To represent acid attack in storage silos, the characteristics of the solutions are kept unchanged from the initial conditions [7] or periodically maintained, either through the renewal of the solution or addition of concentrated acid to keep the pH constant [9] or within a range [10]. To simulate the conditions in a wastewater collection unit, the acid solution is constantly renewed either by constant replenishment [11] or by automatic titration [12].

2.1.3 Alternate drying and wetting. In order to analyse the physical effects occurring on the specimen exposed to organic acid, alternate drying and wetting is implemented. The specimens are immersed in the acid for a fixed exposure period and then allow it to dry for another period which may or may not be same as the wetting period, this cycle is repeated. Here, the degradation process is accelerated by capillary suction. During wetting, the capillary suction forces act on the acid solution, which is in contact with the specimen. Hence the acid penetrates into the matrix, where the depth of penetration depends on the capillary forces and the permeability of the material. This penetrated acid reacts with the hydrated products forming complex salts which may dissolve and migrate through the concrete. During drying, the dissolved substances get deposited at or near the surfaces, leading to crystallisation and micro-cracking due to shrinkage, resulting in higher penetrability of the material [13, 14, 15]. Alternate wetting and drying cycle method is adopted to simulate the dynamic exposure mode.

2.1.4 Effect of abrasion. Abrasion is the process of removing loosely packed particles, either the degraded layer or the precipitated salts attached to the surface of the specimen. Abrasion indicates the mechanical action of organic acid exposure on concrete. During the mechanical attack the degraded
layer gets removed, and it exposes new layer for chemical action. This will significantly enhance the rate of degradation [8]. Dynamic exposure mode is obtained by introducing the effect of abrasion using wire brushes, pressurised water jets etc. [16].

2.1.5 Type of attack expected. The type of attack expected to occur on concrete and the corresponding real life situation has a great influence on the type of test selected for degradation analysis. The type of attack is categorised into three [3]:

- Purely chemical attacks - these generally simulate the submerged zone of effluents like the storage silos, some treatment plants and collection systems, which are carried out by immersion tests where the specimens are submerged in the acidic solution for the study. It can be either static or dynamic condition [7, 9, 10]. The test methods adopted for this type may include immersion tests, batch extraction tests etc.
- Combined chemical and physical attacks – these types of attack mainly occurs on concrete floors or storage silos [3]. Alternate wetting and drying simulates this condition and behaves similar to this type of attack
- Combination of physical, chemical and mechanical attacks – these attacks take place particularly in animal houses or production areas [3], which can be simulated by immersion test in the acid solution for identifying chemical attack, alternate drying and wetting cycles for evaluating physical attack and washing with pressurised water jets or wire brushing for analysing mechanical effect [16].

In addition to the above, since organic acids can be produced by microorganisms, the need to explore the biogenic effects of organic acid attack becomes necessary. For example, the degradation due to biologically generated lactic acid and chemically synthesised lactic acid may be different, and the difference in degradation mechanism should be studied thoroughly. This area of research needs further explorations.

2.1.6 Degradation measurement technique. The test methods should be selected based on the technique used to measure degradation. For assessing the changes in properties of concrete with respect to time, i.e., the degradation kinetics, parameters like visual observations, altered depth, mass loss, pH changes, compressive strength changes, changes in dynamic modulus of elasticity etc. can be adopted. Likewise, for studying the microstructural characterisation, i.e., degradation mechanism, techniques like X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), X-Ray Fluorescence (XRF), Fourier Transform Infrared Spectroscopy (FTIR), Thermo-gravimetric Analysis (TGA), X-ray Computed Micro-tomography (X-µCT), Mercury Intrusive Porosimetry (MIP) etc. can be adopted.

If the study focuses on identifying the suitability of a particular material in an aggressive acidic environment, material properties, compatibility of the materials, degradation kinetics and mechanism should be studied.

2.1.7 Initial curing in saturated lime water solution. Initial curing in lime water solution increases the compressive strength of concrete, also it helps in preventing the leaching of calcium hydroxide from the concrete, thereby reducing the permeability of concrete. So, before exposing to the acids, concrete should be adequately cured in saturated lime water solution for better permeability and thereby improving the durability of concrete. Givi et al. examined the effect of curing in lime water solution on the strength and water absorption of concrete, and observed that water absorption and permeability can be reduced by providing initial curing in saturated lime water solution. The optimum duration for initial curing is found to be 28 days because thereafter, only slight changes are observed [17].

2.1.8 Temperature of the medium. An increase in temperature enhances the chemical kinetics of the acid solution, thereby accelerates the rate of degradation. In addition to this, as the temperature rises, the water in the pores of the concrete gets evaporated, which increases the porosity and thereby permeability of concrete that again hastens the rate of degradation [8].

2.2 Specimen related factors
The physical properties of the specimens cast have a significant influence on the results obtained for various test methods. The main properties include surface area to volume ratio, type, shape size and
nature of specimen. These physical characteristics should be further studied to identify their effect on the results.

2.2.1 Surface area to volume ratio. The surface area of the specimen to its volume has a great impact on the rate of deterioration of concrete. As the contact surface area of the specimen increases, more surface area is exposed to the chemical attack, which enhances the rate of concrete deterioration [8]. For a given volume of the specimen, the surface area can be increased either by selecting a suitable shape for the specimen or by changing the nature of the specimen. For example, for a given volume, the cylindrical specimen has a high surface area to volume ratio, enhancing the rate of degradation. Also powdering a lump specimen causes no change in volume, but the number of particles increases which results in greater surface area owing to an accelerated rate of reaction.

2.2.2 Type of specimen. Paste specimens can be preferred for the analysis of deterioration due to acid attack, as the aggressive acids primarily attack the paste phase of the concrete. Hence, it is convenient to carry out both degradation kinetics and mechanism study in cement paste rather than in concrete specimens. The paste analysis also makes it possible to quantify uniform deterioration without aggregate disruption and therefore the quantification of kinetic factors may be more important particularly when comparing the performance of different binders in resisting acid attack [18].

| Point in Time | Before | After 1 day | After 3 days | After 7 days |
|---------------|--------|-------------|--------------|--------------|
| Acid types (pH = 2.5) | Samples | Reconstructed 2D-cuts based on multiple measurements with 3D-μXCT |
| Sulphuric acid | Hardened cement paste |
| Acetic acid | Concrete |
| Sulphuric acid |
| Acetic acid |

**Figure 1.** Hardened cement pastes and concrete specimens (cubes 30 x 30 x 30 mm³) subjected to acid attack [12]

Not only for the analysis of degradation kinetics, but chemical and mineralogical analyses also prefer paste specimens. This is mainly because, for chemical analysis, the presence of aggregates complicates the analysis and hence need to be extracted before the tests. For mineralogical analyses like X-ray diffraction (XRD) and X-ray fluorescence (XRF), the peaks obtained for cementitious matrix are of very low intensity compared to peaks from siliceous aggregates, which leads to misinterpretation of results [3].

The primary concern with the use of paste sample is that whether it is representative of the real condition. The effect of aggregates cannot be identified when paste samples are studied. Firstly, the mineralogical nature of aggregates are different, there can be reactive aggregates which may alter the
degradation kinetics and mechanism. Also, the presence of large aggregates in cement matrix results in interfacial transition zone (ITZ) [3]. ITZ has comparatively more pores in the vicinity of aggregates and supposed to be the weakest plane in concrete. So, the presence of ITZ also affects the degradation kinetics and mechanism. This makes the use of concrete specimen vital. Figure 1 shows various types of specimens used for assessing degradation characteristics. Mortar specimens can be used for experiments to qualitatively analyse the suitability and performance of materials. Mortar specimen is an intermediate between paste and concrete specimens.

2.2.3 Shape of the specimen. Cylinders, cubes, and prisms are the usual shapes cast for concrete testing. Generally, cylindrical specimens are preferred to prismatic for strength tests, mainly because of the presence of edge zones in prismatic specimens. In order to prevent axial specimen heterogeneity, the loading zones of the specimen should also be properly protected against acid exposure or abraded if it is exposed before testing [3]. Cylindrical specimen exhibits a greater rate of reaction as the surface area to volume ratio is higher for cylinders. The major advantage of the prismatic specimen is that it can be cut into smaller specimens for microstructural characterisations. Various shapes adopted for casting the specimens are depicted in Figure 2.

![Figure 2](image)

**Figure 2.** Specimens of various shapes (a) Small specimen cut from a prism for X-ray CT imaging (b) Cube specimen (c) Cylindrical specimen [18, 19, 4]

2.2.4 Size of the specimen. All sample sizes can be used for degradation analysis, provided that a significant homogeneous attack zone can be identified in the sample. [3]. Smaller specimens exhibit a higher rate of deterioration [8]. Smaller specimens have more contact surface area exposed for the chemical attack which increases the rate of reaction, also smaller specimen requires only smaller quantity of aggressive acid solution for experimental work but exhibits better results than the use of larger specimens.

2.2.5 Nature of the specimen. The specimen chosen for the study can be either a solid specimen or powdered sample. The solid specimens can be cylinders, cubes or prisms. These can be used for the analysis of mechanical properties and degradation kinetics. The powdered samples are generally obtained by crushing these solid specimens and mainly used for microstructural analysis [20]. The main advantage of having solid specimens for assessing acid attack is that zones of degradation can be easily distinguished. Different zones of degradation can be separately chipped off from the solid specimen and powdered so that microstructural characterisation of each zone can be obtained. Also, the contact surface area increases as the sample is more powdered, which enhances the rate of degradation when exposed to acids.

2.3 Acid related factors

Organic acids are designated as weak acids and are partially dissociated in water according to their dissociation constant, which can be one or more depending on their poly-acidity. Organic acids require more concentration for making the pH same as that of strong acids, thereby increasing the aggressiveness towards the cementitious matrix [3]. This makes the concentration of organic acid more significant than pH.
2.3.1 Concentration of acid chosen for study. Low concentrations are usually observed in real life conditions, where higher concentrations are adopted for accelerated tests. Higher the concentration of acid chosen, greater is the rate of degradation [8]. This is mainly because higher the concentration, more anions of the acids are freely available to react with the hydration products, thereby increasing the degradation.

It is also observed that the same binder may behave differently when exposed to both lower and higher concentrations of the same acid. Dyer compared the efficiency of three types of binder namely, Ordinary Portland Cement, Portland cement blended with fly ash and Calcium sulphonylate cement (PC, PC/FA and CSA) using laboratory experiments when exposed to acetic and butyric acids [21]. Figure 3 depicts the mass loss from cement pastes exposed to acetic acid at low and high concentrations. From the figure, it can be seen that at lower concentrations (0.01M) CSA shows higher mass, i.e., least mass loss whereas at higher concentrations (0.1M) CSA shows the lowest mass or highest mass loss. This indicates that the same material can behave differently when exposed to different concentration of acids [21].

![Figure 3. Difference in performance of cement pastes on exposure to same acid at high and low concentrations [21]](image)

2.3.2 Interchangeability of acids. Certain organic acids cannot be used for experimental studies. It is possible to substitute organic acids that are malodorous, such as butyric, iso-butyric, valeric, iso-valeric, etc., with other acids with similar effects on the matrix. Bertron et al. have shown that in terms of aggressiveness and degradation mechanisms, butyric, iso-butyric and valeric acids are identical to acetic acid in accordance with their comparable pH and solubility of calcium salts [20]. Preliminary checks should be done on all other cases before using one acid instead of another. In addition, it should be noted that some organic acids are corrosive in nature and are irritating (eye and skin contact and ingestion should be avoided) [3]. In addition to this, some organic acids like oxalic acids are toxic in nature, and so should be handled carefully or replaced with another acid exhibiting equivalent effect on the cementitious matrix.

2.3.3 Volume of acid solution to the specimen. Volume of acid solution with respect to that of the specimen indicates the rate of change in pH and hence the rate of attack [2]. As the ratio of volume of acid solution to specimen increases, the pH of the solution does not elevate rapidly, which results in increasing the aggressiveness of the environment and hence increasing the rate of deterioration.

2.3.4 Acid exposure period. In systems where acid renewal is not carried out, as the acid exposure period increases, degradation increases till it attains a steady state, i.e., all the free anions are used up for forming salts and complexes. In systems where acid renewal is done, as the acid exposure period
increases degradation continuously. In such cases, to analyse the long term effects on concrete, tests should be periodically conducted for a prolonged duration.

3. Conclusion

The immense exposure of concrete to acidic environment highlights the significance of understanding the degradation mechanism of acid attack and also to develop suitable materials that perform well in these environments. However, currently there are no standard procedures for assessing the performance of concrete exposed to acids. For developing such a procedure, as a first step, the factors influencing the selection of a test method should be clearly identified.

The factors are broadly categorised into test condition related factors, specimen related factors and acid related factors. Test condition related factors incorporate the settings and arrangements adopted for the study whereas specimen related factors deal with the physical properties of the specimen used. Acid related factors include various aspects of acids chosen for the investigation other than its material properties.

However, there are no actual studies regarding the interaction between these factors. Based on the factors chosen, the ranking of acid resistance of various binder systems may vary. Further research is therefore needed to determine a correlation between these factors in the analysis of concrete degradation characteristics.

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