A review on the deterioration of cement-based materials in ammonium salt solutions

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Abstract. It is essential to understand the durability aspects as the cementitious systems exposed to agricultural zones, fertilizer plants, waste waters, mining and industrial applications can be subjected to chemical attack by ammonium based compounds. This paper reviews the different mechanisms of deterioration of cement composites on exposure to different ammonium compounds namely ammonium sulphate, nitrate and chloride. The magnitude and mechanism of degradation follows different trends depending on the type of anion associated with the ammonium salts. The principle process involved in the deterioration of cementitious systems in the presence of ammonium compounds is the chemical reaction of portlandite with the anion of the ammonium salts. Generally the interaction of ammonium salts with cement based materials results in decalcification, leaching and/or expansion accompanied by strength loss. The presence of Supplementary Cementitious Materials (SCMs) along with cement reduces the amount of portlandite which may be attributed to the dilution of cement component or increased pozzolanic activity, resulting in the production of additional or secondary C-S-H gel. It is observed that the incorporation of SCMs is found to have low penetrability, thus capable of controlling the degradation process upon exposure to ammonium based salt solutions.

1. Introduction
Cement composites exposed to agricultural zones, fertilizer plants, waste water, mining, nuclear and chemical industries can be subjected to chemical attack by ammonium based compounds. The reaction between certain ammonium salts and cementitious systems has long been recognized as potentially aggressive. Salts such as ammonium nitrate, chloride, phosphate, sulphate, sulphide, sulphite and bicarbonate are being considered as most harmful whereas ammonium carbonate, oxalate and fluoride being harmless. The magnitude and mechanism of degradation follows different trends depending on the type of anion associated with ammonium salts, as it determines the solubility of the calcium salt produced. The principle process involved in the deterioration of cementitious systems in the presence of ammonium compounds is the chemical reaction of portlandite and CSH gel with the ammonium salts. Limited researches are reported on the resistance of cementitious systems in such ammonium environments. Hence, this paper reviews the different mechanisms and the effect of deterioration of
cement composites exposed to different ammonium compounds namely ammonium sulphate, nitrate and chloride. Ammonium sulphate attack is considered as the most aggressive type of chemical attack characterised by an acid sulphate damage resulting in softening, expansion, cracking and spalling [1]. The ammonium nitrate and chloride attack is characterised by leaching, decalcification and expansion resulting in cracking and swelling of cement composites accompanied by strength loss.

2. Ammonium sulphate attack

2.1 Mechanism of deterioration
Deterioration by ammonium sulphate is the one of the most aggressive corrosion, characterised by intensive dissolution of cement hydrates and expansion due to the formation of gypsum and ettringite. Ammonium sulphate attack is found to be very superficial and gypsum is not found on the core of specimens [2]. The deterioration is characterised by rapid cracking due to surface shrinkage and release of ammonia gas from cement matrix when OPC mortar specimens are washed after immersion in ammonium sulphate solution. The surface deposits on specimens are found to be needle like gypsum crystals having low solubility [3]. Deterioration in ammonium sulphate is caused by acid, sulphate and ammonium corrosion [4]. In addition to the attack by sulphate ions, there is a deleterious action caused by ammonium ($\text{NH}_4^+$) ions. Portlandite is highly soluble in ammonium solutions, and there is a neutralization action of ammonium cations due to acid hydrolysis with the cementitious matrix [5]. The reaction of cementitious matrix with ammonium ions thus results in the release of ammonium gas.

$$\text{NH}_4^+ + \text{OH}^- \rightarrow \text{NH}_3 + \text{H}_2\text{O}$$

Corrosion of concrete due to ammonium sulphate has a combined acid–sulphate attack nature and can be generally characterized by the following reactions:

$$(\text{NH}_4)_2\text{SO}_4 + \text{Ca(OH)}_2 + 2\text{H}_2\text{O} \rightarrow \text{CaSO}_4 \cdot 2\text{H}_2\text{O} + 2\text{NH}_4\text{OH}$$

$$4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O} + 3\text{CaSO}_4 \cdot 2\text{H}_2\text{O} \rightarrow 3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaSO}_4 \cdot x\text{H}_2\text{O} + \text{Ca(OH)}_2$$

$x\text{Ca}_3\text{SiO}_3 \cdot \text{aq} + x(\text{NH}_4)\text{SO}_4 \cdot x\text{H}_2\text{O} \rightarrow x\text{SiO}_2 \cdot \text{aq} + x\text{CaSO}_4 \cdot 2\text{H}_2\text{O} + 2x\text{NH}_3$

The chemical reaction of ammonium sulphate with portlandite forms gypsum which leads to a reduction in pH and an increase in porosity of the concrete surface in contact with the solution. After the complete depletion of portlandite, decalcification of calcium silicate hydrate (C–S–H) occurs resulting in the further reduction of pH of the pore solution. This reaction starts with a reduction of the calcium-to-silicate ratio of the C–S–H structure with the progressive loss of cohesion and eventually its disintegration. The formation of gypsum and ettringite along with gaseous NH$_3$ in the pore structure can lead to volume swelling [6]. The reaction of ammonium sulphate with calcium aluminate results in the formation of calcium sulpho-aluminate resulting in expansion which may be attributed to its low solubility [7].

The damage mechanism of cementitious materials in ammonium sulphate solution is characterised by decomposition, softening, expansion, cracking and spalling, the extent of damage depends on the type of binder and its microstructural and mineralogical characteristics. The resistance of cementitious materials to deterioration depends on physical penetrability, amount of calcium aluminate in cement, amount of portlandite available, volume of C-S-H vulnerable to decomposition or decalcification by ammonium ions [1].

2.2 Role of SCMs
The addition of Supplementary Cementitious Materials (SCMs) is found to have significant effect on the durability against ammonium salt solutions. Among different SCMs such as flyash, slag, silica fume and metakaolin, silica fume yielded best results in improving the durability when exposed to ammonium sulphate solution [2]. The presence of SCMs along with cement reduces the amount of portlandite which
may be attributed to the dilution of cement component or increased pozzolanic activity, resulting in the production of additional or secondary C-S-H gel. It is observed that the concrete specimens incorporating SCMs is found to have low permeability, thus capable of controlling the degradation process upon exposure to ammonium based salt solutions [1].

The porosity and mass change of different mixtures of high strength OPC concrete densified with ultrafine particles of various types of binder materials (10% of the cementitious material content) such as Silica fume (SF), Metakaolin (MK) and Ground granulated slag (GGS) exposed to ammonium sulphate solution is shown in Figures 1 and 2 respectively. The porosity is determined by Mercury Intrusion Porosimetry (MIP). In this study, cylindrical specimens having diameter 23.7 mm and height 40 mm were used and the specimens were subjected to 6 cycles of degradation in lime saturated water and ammonium sulphate solution. Each immersion cycle consists of 4 weeks of immersion in the aggressive solution followed by 1 week of drying at 20 ⁰C. It is found that after 6 cycles of degradation, the mass loss is in the range of 6-10% depending on the type of fine particles used. It is also found that mass loss is least for the mix with silica fume as the fine particles, while the mass loss is high for metakaolin (Figure 2). Also, from Figure 1, it is evident that there is considerable decrease in the porosity of concrete mixes with the addition of ultrafine particles which might have led to the improved performance [2].

Figure 1. Changes in the porosity of high strength concrete specimens with different binders [2]

Figure 2. Mass loss after six cycles of degradation [2]
Measuring the pH of the solution is also tried to investigate more about the degradation kinetics [1]. The initial and final pH of ammonium sulphate solution is measured using digital pH meter. The solutions were renewed after every 4 weeks to maintain the pH at a neutral range of 6.0-8.0. The pH of the original solution is found to be about 6.0, and the change of pH over the exposure period is as shown in Figure 3 [1].

![Figure 3. Average change of pH in solution over 52 weeks [1]](image)

Increase in the pH of the solution in the weeks following the renewal of solution may be due to the dissolution of hydrates in concrete. As the pH of the solution is lower as compared to the pH of the pore solution of concrete or mortar specimens, there will be diffusion of hydroxyl ions from the specimens to the surrounding solution, thus increasing the pH [8].

The use of three component binders (Portland cement, pulverized slag and silica fume) in cement composites results in the development of a compact gel Calcium Silicate Hydrate (C-S-H) phase with reduced ratio of CaO to SiO₂. This lowers the quantity of reactive and the most unstable phase (i.e. calcium hydroxide caused by the lower alite concentration (dilution effect) and pozzolanic reaction with silica fume) and the sealing of microstructure with the micro filler, significantly reduce porosity and improve the resistance of cement composites and thus improve the performance in chemically aggressive environments [4]. Depending on the type, nature and dosage of SCMs, the interaction of binders with ammonium sulphate solution represents different modes and levels of deterioration. Thus the physico-mechanical trends of cement composites is characterised by softening with and without significant expansion. The cement composites incorporating silica fume/nano-silica and flyash showed good performance in ammonium sulphate solution due to mutual balance between physical and chemical resistances [1].

On the other hand, Bassouni and Nehdi [5] inferred that the presence of some portlandite in the system is rather useful in offering some resistance. The binary binders with a low replacement level of OPC (92% OPC and 8% silica fume) led to inferior performance of Self Compacting Concrete (SCC) specimens in the ammonium sulphate solution due to the lack of portlandite, abundance of Calcium Silicate Hydrate (C-S-H) and the absence of a protective zone. While the ternary and quaternary binders at a high OPC replacement level (50%) typically led to matrices rich in C–S–H and poor in portlandite, thus performed well in the ammonium sulphate solution. The diffusion of ammonium sulphate slowed down in these systems due to the formation of a thick passivating layer of hydrous silica (amorphous silica gel) encapsulating the gypsum in the outer layers.
3. Ammonium nitrate attack

3.1 Mechanism of deterioration
Ammonium nitrate solution is considered to be an aggressive medium causing leaching of cement composites resulting in strength loss. The mechanism of deterioration is found to be different from ammonium sulphate. The leaching process leads to decalcification and the dissolution of hydration products so that the distribution of calcium ion distribution and pH value in leaching area reduces. The extent of leaching due to ammonium nitrate depends on the concentration of the solution, strength of cement composites and duration of immersion [9]. Ammonium nitrate attack is characterised by rapid decalcification of calcium rich hydrates, accompanied by strength loss due to high solubility of calcium nitrate in solution. The surface deposits on specimens exposed to ammonium nitrate solution is found to be rhombic calcite, which is formed due to the reaction between calcium and carbon dioxide (CO$_2$) dissolved in water [3].

The chemical attack by ammonium nitrate leads to the formation of soluble calcium nitrate, calcium nitro aluminate salt and ammonia emanation. These reactions results in the total leaching of portlandite and progressive decalcification of calcium silicate hydrate [10]. As per the literature by Arafa et al. (2015) [11], the chemical reactions are as follows:

$$2\text{NH}_4\text{NO}_3 + \text{Ca(OH)}_2 + 4\text{H}_2\text{O} \rightarrow \text{Ca(NO}_3)_2 \cdot 4\text{H}_2\text{O} + \text{NH}_4\text{OH}$$

$$4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O} + \text{Ca(NO}_3)_2 \cdot x\text{H}_2\text{O} + \text{Ca(OH)}_2 \rightarrow 3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{Ca(NO}_3)_2 \cdot x\text{H}_2\text{O} + \text{Ca(OH)}_2$$

$$(\text{CaO})_3 (\text{SiO}_2) (\text{H}_2\text{O})_8 + 4\text{NH}_4\text{NO}_3 \rightarrow 2\text{Ca(NO}_3)_2 + 4\text{NH}_3 + (\text{CaO}) (\text{SiO}_2) (\text{H}_2\text{O})_6 + 4\text{H}_2\text{O}$$

The deterioration of cement based materials in contact with ammonium nitrate solution can be of two types, depending on the environment – it can be a very large increase in the porosity with the weakening of mechanical properties or a notable swelling along with cracking which may be due to the formation of expansive crystals [10].

The bulk density of cement mortar is moderately affected by different concentrations of ammonium nitrate solution. For 5% concentration of ammonium nitrate, the bulk density of the mortar decreased from 2.18 g/cm$^3$ to 2.12 g/cm$^3$ while the bulk density decreased from 2.18 g/cm$^3$ to 2.04 g/cm$^3$ for 20% concentration at 60 days of immersion. The loss in bulk density of cement mortar immersed in 50% ammonium nitrate solution for 60 days is about 9%. The reduction in bulk density may be attributed to the mechanism of calcium hydroxide removal and decalcification of calcium hydrate compounds. This process results in moderate loss of cement mortar constituents, and increase in the matrix porosity, which may result in reduction in bulk density of mortar specimens [11].

3.2 Role of SCMs
The use of cement, fly ash and silica fume as binders makes concrete less susceptible to damage in ammonium nitrate solutions. The reduction in pH value and rate of degraded depth is found to be less for concrete incorporated with cement, fly ash and silica fume as binders [8]. With the increase in the concentration of ammonium nitrate solution and exposure duration, there is significant reduction in the physico-mechanical properties of cement composites. It is observed that CaO to SiO$_2$ of C-S-H decreased with the increase in the concentration of ammonium nitrate solution implying the decalcification of C-S-H gel. There is significant increase in the porosity of hardened cement paste with the increase in the concentration and exposure duration; the deteriorated specimens have a coarser structure [11].

Wong et al. (2013) carried out study on two concrete mixes G50 and G60 exposed to ammonium nitrate solution [8]. G50 is a concrete mix with Ordinary Portland Cement (OPC) and silica fume as binder material, and river sand and granite as fine and coarse aggregates. G60 is another concrete mix incorporating OPC, Fly ash and silica fume as binder and a mixture of river sand, crushed sand stone and granite as aggregates. These specimens were immersed in 20% concentration of ammonium nitrate
solution for duration of 56 and 90 days respectively after 28 days of initial curing. It is found that water absorption of concrete specimens immersed in ammonium nitrate solution is higher as compared to specimens cured in water. But the rate of water absorption decreased with concrete age for both concrete type G50 and G60. It is also found that concrete type G50 absorbs more water as compared to G60. The difference between the water absorption of concrete exposed in ammonium nitrate solution and water at 56 days is 0.75% for concrete type G50 and 0.69% for G60. The difference increased to 1.92% for G50 and 1.27% for G60 at 90 days. The increase in the rate of water absorption is due to the dissolution of calcium hydroxide caused by ammonium nitrate solution. Removal of calcium hydroxide introduces more permeable voids within the concrete and increases the porosity of concrete. As porosity increases, the diffusion coefficient of calcium is also increased leading to higher rate of hydrates being dissolved. This results in the modification of microstructure of concrete, causing an increase in permeability and a decrease in the bulk density.

It is also found that the water sorptivity coefficients of concrete immersed in ammonium nitrate solution increased with the increase in the exposure duration [8]. It is found that the sorptivity of G50 specimens immersed in ammonium nitrate solution for 56 days is 27.4% higher than that of specimens immersed in water for the same duration. After 90 days of exposure, the difference in sorptivity coefficient of G50 specimens increased to 34.8%. For concrete type G60, the difference is only 9.3% for ammonium nitrate treated concrete at the age of 56 days compared to those immersed in water at the same age. Subsequently, the sorptivity increased 22.0% at 90 days of exposure. The compressive strength of these concrete specimens exposed to ammonium nitrate solution was also measured. For concrete type G50, the strength decreased to 42.71 N/mm² at the age of 56 days and continued to decrease until 30.88 N/mm² at the age of 90 days. For concrete type G60 exposed to ammonium nitrate solution, the strength decreased from 51.11 N/mm² at the age of 56 days to 38.28 N/mm² at the age of 90 days. The percentage of difference in strength between concrete treated with ammonium nitrate solution and water at the age of 56 days was about 22.48% while for concrete at the age of 90 days, the difference in strength was 42.06%. The rapid loss in strength may be attributed to the disintegration of hydrates and subsequent increase in the matrix porosity. The loss of strength for concrete type G60 was less than concrete type G50 after leaching by ammonium nitrate solution, indicating that concrete type G50 was more susceptible to damages done by ammonium nitrate solution compared to concrete type G60. This suggest the fact that properly designed concrete with ternary binder systems may offer increased resistance owing to a denser microstructure and lowered permeability [8].

Another recent study done by Rehman et al. [12] concluded that the addition of nano-silica in photocatalytic cementitious materials showed better resistance against leaching on exposure to ammonium nitrate solution. The presence of nano-silica resists the microstructural changes and degradation of hardened properties of cementitious materials due to leaching on exposure to ammonium nitrate solution.

4. Ammonium chloride attack

4.1 Mechanism of deterioration

Ammonium chloride (NH₄Cl) is an inorganic white crystalline salt that is highly soluble in water. Solutions of ammonium chloride are slightly acidic which aggravates the deterioration process further. The ammonium chloride can be considered as another important ammonium based salt solution which can induce degradation of cement composites. The chemical reaction proposed is as follows:

\[
\text{Ca(OH)}_2 + 2\text{NH}_4\text{Cl} + 2 \text{H}_2\text{O} \rightarrow \text{CaCl}_2 \cdot 2\text{H}_2\text{O} + 2\text{NH}_4\text{OH}
\]

\[
4\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot x\text{H}_2\text{O} + \text{CaCl}_2 \cdot 2\text{H}_2\text{O} + \text{H}_2\text{O} \rightarrow 3\text{CaO} \cdot \text{Al}_2\text{O}_3 \cdot \text{CaCl}_2 \cdot x\text{H}_2\text{O} + \text{Ca(OH)}_2
\]

\[
\text{Ca(OH)}_2 + \text{CaCl}_2 + \text{H}_2\text{O} \rightarrow \text{Ca(OH)}_2 \cdot \text{CaCl}_2 \cdot \text{H}_2\text{O}
\]
When cement based materials are exposed to ammonium chloride solution, the Ca(OH)$_2$ formed reacts with NH$_4$Cl forming hydrated calcium chloride (CaCl$_2$.2H$_2$O). This hydrated calcium chloride thus formed further reacts with calcium silicate hydrate formed during cement hydration and forms calcium chloro aluminate hydrate. The calcium chloride also reacts with calcium hydroxide forming calcium hydroxychloride hydrate. The interaction of ammonium chloride with cement composites results in the decalcification and expansion. Formation of double salt chloroalumino hydrate results in considerable increase in volume leading to expansion and disruption of hardened concrete [13].

Ammonium chloride attack is characterised by dissolution of portlandite resulting in an increase in porosity. The crystallization of thaumasite occurs followed by dissolution of ettringite. Another intermediate phase is characterized by the formation of Friedel’s salt. The final products formed due to the chemical reaction of ammonium chloride with cementitious materials was found to be gypsum, calcite and vaterite [14]. However, the mechanism is not well understood and more micro-analytical characterization studies are to be performed to understand the mechanism in a better way.

4.2 Role of SCMs
According to Slupik [14], the experimental investigations carried out by for measuring the effect of saturated ammonium chloride on the pastes produced from CEM I 42.5N-HSR/NA and CEM III/A 32.5N-LH/HSR/NA slag cement shows that there are many different fronts of corrosion which clearly indicates the progress of destruction. From the microstructural studies, it is found that the dissolution of Friedel’s salt released chloride into the pore solution. The crystallisation of Friedel’s salt is found to be less for CEM I 42.5N-HSR/NA, thus having better durability as compared to slag cement. The crystallisation of large amount of gypsum and calcium carbonate in the pores of surface layers caused a reduction in the total volume of pores. The hardened cement paste of CEM I undergone a large increase in the porosity in the initial stages but slowed down in later stage as compared to CEM III. But in the case of CEM III cement, the increase in porosity is found to be about 50% [14]. There are only limited studies available on the durability in ammonium chloride environment and there is scope for more research to understand the behaviour of SCMs in such environment.

5. Summary of deterioration
The deterioration of cement-based materials due to the various ammonium salts in solution primarily depends upon the salts formed and the characteristics of the salts. Table 1 summarizes the various aspects related to the deterioration.

| Ammonium compounds | Reaction products | Solubility of salt | Deterioration process |
|---------------------|-------------------|-------------------|----------------------|
| Ammonium sulphate   | Gypsum, silica gel, ettringite, ammonia | Very low | Expansion, softening of the matrix |
| Ammonium nitrate    | Calcium nitrate, silica gel, calcite, ammonia | High   | Leaching, increase in porosity |
| Ammonium chloride   | Calcium chloride, Friedel’s salt, calcite, vaterite etc. | High   | Leaching, increase in porosity |

Table 1. Summary of deterioration products
6. Conclusions
Cement concrete exposed to agricultural land, fertilizer plants, waste water treatment plants, mining and industrial applications may be vulnerable to chemical attack by ammonium based solutions. Ammonium based salts are widely used as fertilizers in agricultural zones. Fertilizers are mostly nitrogen based and consist of ammonium salts. Concrete undergoes progressive deterioration due to the reaction of ammonium salts with cement hydrates such as calcium hydroxide (Ca(OH)₂), tricalcium aluminate (C₃A) and calcium silicate hydrate (C-S-H) gel. Thus, it is necessary to understand the durability aspects of cementitious compounds exposed to ammonium based solutions. Based on the review of literature, the most common and deleterious ammonium based salts are found to be ammonium nitrate, ammonium chloride, ammonium sulphate, ammonium phosphate, ammonium superphosphate etc.

The aim of the study was to investigate the durability aspects in terms of microstructural features and physico-mechanical properties of cement mortar with and without Supplementary Cementitious Materials (SCMs) exposed to different ammonium based salt solution prepared using chemical grade ammonium nitrate, ammonium chloride, and ammonium sulphate. The SCMs considered for the study include Class F fly ash, Ground granulated blast furnace slag, and silica fume. Some of the available literature in this area was critically reviewed in order to understand the mechanism of deterioration in various ammonium environments.

Nowadays, the demand for green and sustainable concrete has increased due to the greenhouse gas emissions and uncontrolled pollution from the cement industry (due to the production of OPC). Also, concrete made by OPC is reported to be less resistant in aggressive chemical media. It is observed that there are significant changes in the microstructure and reduction in the mechanical properties of cement composites when exposed to different ammonium based solutions at different concentrations. A better solution for this situation is to explore the role of alternate binding materials such as SCMs in offering resistance against harsh aggressive environments. In general, the incorporation of Supplementary Cementitious Materials (SCMs) along with cement is found to be effective in improving the durability of cementitious systems when exposed to ammonium based solutions and the durability performance of various SCMs (either as binary or ternary inclusions) has to be investigated further. Only limited studies are reported on the deterioration of cement-based materials due to ammonium compounds present in agricultural fertilizers. Hence, it is imperative to understand the nature of deterioration of cementitious materials due to these chemicals in order to formulate the mixes that are durable in these aggressive exposure conditions.

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