An Overview of Ammonium Chloride (NH₄Cl) Corrosion in the Refining Unit

Akpanyung K.V¹, Loto R.T¹, Fajobi M.A.¹

¹Department of Mechanical Engineering, Covenant University, P.M.B 1023, Ota, Ogun State, Nigeria.
kakpanyung@gmail.com

Abstract:
Ammonium chloride is a destructive agent of localized corrosion which poses devastating threat to refining structure integrity and the safety of the refinery processes. Ammonium chloride is an underdeposit corrosion commonly found in overhead equipment and piping for crude and hydroprocessing units. This form of insidious form of corrosion had caused severe fouling that posed negative impact on the operating reliability of various processing units. This review addresses the corrosion mechanism of ammonium chloride, affected materials and equipment, Environmental factors for its impact, thermodynamic behavior of a wet ammonium chloride system, some case studies of ammonium chloride and the preventive measures to mitigate its effect.

Keywords: ammonium chloride, dew point, salt formation, ionic equilibria, hydroprocessing, distillation overhead systems.

1. Introduction
Corrosion is the spontaneous process of converting metals from their unstable or higher energy state (excited state) to their stable or lower energy state. Corrosion (oxidation) is therefore considered as a reverse process of metallurgy (reduction) process, a process of extracting metals from their combined or raw states [1], [2]. Ammonium Chloride (NH₄Cl) corrosion is a destructive form of localized corrosion. Corrosion propagation by wet ammonium chloride salt is a great issue in refining units due to its outrageous effect as a result of the increasing cost of materials, labor, maintenance and repair. Ammonium chloride corrosion poses devastating threat to refining structure integrity and the safety of the refinery processes. The forms of corrosion by ammonium chloride are majorly general or localized corrosion. Pitting corrosion in absence or limited supply of free water phase is always encountered where there is deposit of ammonium chloride salt. The destructive effect of ammonium chloride salt deposits only sets in at temperatures above the dewpoint. In the refineries, condensed water washes the deposit away at temperatures which is less than the dewpoint. Ammonium chloride is a salt gotten from strong acid, HCl and weak base, NH₃; it is considered an acid salt. Ammonium chloride is a complex metallic ion which contains the corrosive chloride ion. The chloride ion is major catalyzing agent to its deteriorating effect. Ammonium chloride either as gas, solid, or in solution is corrosive [10]. NH₄Cl at near aqueous dew point or at high concentration level is highly corrosive, and is generally less corrosive when diluted. Material selection and corrosion protection is of high priority in an environment where ammonium chloride is found or generated such as the refining industries and the fertilizer industries. NH₄Cl also lead to extensive fouling and plugging, thereby causing pressure drops and loss of throughput. Due to its hydroscopic nature, NH₄Cl salt readily absorbed water which enhances its deteriorating strength. The determining factors to the extent of corrosion of ammonium chloride are concentration, temperature, moisture, and pH. As a result of these mentioned factors and fluid variation process fluid, chemical composition, there are several
percentages of corrosive components experienced in the processing system of the refining [4]. Wet ammonium chloride corrosion at high concentrated conditions are much rapid at rates which approach hydrochloric acid, greater than 1,000 mpy (25 mm/y), on carbon steel [3]. Ammonium bisulfide has similar destructive effect like ammonium chloride, but ammonium chloride corrosion is not much easy to be predicted due to higher limit of temperature and unpredictability of deposition location as a result of temperature and HCl/ammonia partial pressures [21].

2. **Affected Materials and Equipment**

The susceptibility of materials to ammonium chloride corrosion with their increasing order of resistance is give thus: carbon steel, low alloy steels, 300 series SS, Alloys 400, duplex SS, 800, and 825, Alloys 625 and C276 and titanium. Chloride stress corrosion cracking can set in when 300 series stainless steels is in contact with the deposits of ammonium chloride [21]. Ammonium chloride corrosion causes higher degree of destruction to equipment and piping in the following refining sections: hydroprocessing effluent systems, overhead systems of crude distillation and fractionation columns of thermal and catalytic conversion, Fluidized Catalytic Cracking Unit (FCCU), and product stabilizer towers and recycle gas systems in catalytic reforming units, HDS effluent exchangers and a hydrotreater desulfurizer prefractionator feed/bottoms exchanger, [3]. Reactor effluent streams at about ~300F of temperature are susceptible to ammonium chloride salt fouling and corrosion.

3. **Environmental Factors**

Availability and concentration of (HCl, NH₃ or amine salts), availability of water and temperature are the striking parameters to ammonium chloride formation and corrosion. The concentration of NH₃ and HCl, is the determining factor to the precipitation of ammonium chloride salt from high stream of temperature as it get cooled, and thus corroding equipment and piping at temperatures that is much beyond water dewpoint [~300oF (149°C)]. Due to the hygroscopic nature of ammonium chloride and its readability to absorb water, little quantity of water can result into much aggravating corrosion ~360 mpy, based on the temperature [21].

4. **Ammonium Chloride Corrosion in Crude Distillation Units (CDU)**

The table below shows the corrosive compounds located at different components of refining system.

| Location/equipment         | Corrosion Compounds                          |
|----------------------------|-----------------------------------------------|
| Tank                       | HCl, H₂S, dissolve O₂ and water              |
| Preheat exchange           | HCl, H₂S                                     |
| Furnace                    | H₂S and S-compound                           |
| Flash zone                 | H₂S, S-compound, and organic acid            |
| Middle zone                | H₂S                                          |
| Top zone                   | HCl and water                                |
| Tower overhead system      | HCl, H₂S, and water                          |
| Bottom exchanger           | H₂S and S-compound                           |
The major corrosive compounds encountered at crude oil refining system are HCl, H2S, inorganic and organic chloride (such as ammonium chloride, amine chloride, etc) and S-compounds. Hence diverse forms of corrosion are experienced based on the different corrosive compounds listed in the above table in the crude oil refining process plant. This thus calls for appropriate preventive means to combat their effects by applying right chemical dosing at the right region on the process system.

Figure 1. Dosing process of chemicals in crude oil refining process

The following are some of the corrosion challenges, which are mainly caused by high chloride content in crude desalter or improper controls to ensure fractionation conditions in the refining unit above aqueous dew point:

- underdeposit corrosion as a result of the sublimation of species like ammonium chloride.
- aqueous corrosion as a result of hydrochloric acid (HCl)
- fouling challenges related to the buildup of sublimating species [6].

Figure 2. shows the regions where ammonium chloride salt corrosion is detected. These include HDS effluent exchangers, hydroprocessing reactor effluent air cooler tubes and associated piping, a hydrotreater desulfurizer prefractionator feed/bottoms exchanger, or in fractionater overhead systems.
The major cause of crude overhead corrosion at the crude distillation unit is the availability of the vapor hydrogen chloride from salt hydrolysis at the distillation unit atmosphere. HCl is gotten during chloride salt hydrolysis such as sodium chloride, magnesium chloride, calcium chloride and salts at temperatures above 250°F (121°C).

\[
\text{NaCl} + H_2O \rightarrow NaOH + 2HCl \\
\text{CaCl}_2 + 2H_2O \rightarrow Ca(OH)_2 + 2HCl \\
\text{MgCl}_2 + 2H_2O \rightarrow Mg(OH)_2 + 2HCl
\]

HCl can also be formed from organic chloride decomposition. HCl entered into the crude condensing system of the overhead unit and is absorbed in condensing water. Different preventive measures always carried to minimize this acidic attack of the condensed HCl are the neutralizing compounds such as ammonia and organic amines, wash water systems, filmforming inhibitors, and close temperature control at the overhead circuit. These preventive measures if not well applied and control results into fouling, under-deposit corrosion, among other challenges [6]. Most times, the ammonia and organic amines respectively combine with HCl to form corrosive compound of ammonium chloride and amine chloride respectively, both having similar corrosive effect on structural components. The main preventive means in the CDU overhead circuit from excessive attacks of chloride is to ensure proper desalination to remove soluble chloride salts. The CDU overhead circuit temperatures could be control by adequate understanding of the dew point and salt formation temperatures in the system. This enable places that are prone to any form of underdeposit corrosion, severe acid attack, fouling, etc to be identified in the circuit.

Neutralizers such as ammonia and amines (an organic soluble compound) used in the refinery unit such as CDU to enhance the mitigation of HCl attack, being the major cause of corrosion in the unit, also contribute to further challenges such as under-deposit corrosion and fouling issues as a result of the deposition of HCl salts and the neutralizers. There is an expected minimum amount of neutralizer at a specific dew point pH. This minimum quantity of neutralizer for a permissible pH dew point is considered as the essential neutralization variable. During neutralization process, ammonia gas is injected straight into the overhead circuit and the amines dissolved in water then injected into the circuit. Alkyl, alkanol, alkoxy, and cyclic ether types 4 of the organic amine compounds are some of the used organic amines. Amines are preferred to ammonia as neutralizer.
due to the following shortcoming: less neutralization effect at the early stage of water condensation, formation of salt beyond the dew point of water, and challenge in the control of injection rate. At the point of water condensation, ammonia does not serve to neutralize acid because HCl dissolves easily in water than ammonia. Temperatures of salt formation beyond the dew-point may lead to corrosion, fouling and underdepositing.

5. THERMODYNAMIC BEHAVIOR OF A WET AMMONIUM CHLORIDE SYSTEM

The corrosion rate of wet ammonia and hydrochloric acid are comparable under certain conditions since they both exhibit similar corrosion action. The thermodynamic behavior of a NH₃-HCl-H₂O tertiary system and HCl-H₂O binary system is rather not the same. This is because in the HCl-H₂O binary system, the presence of small amount of HCl in the vapor does not significantly change the dew point of water. It only increased by few degrees even when HCl concentration is as high as several hundred ppmv. On the other hand, a NH₃-HCl-H₂O tertiary system can substantially elevate the dew point of water and form a high concentration of NH₄Cl solution at the initial dew point, even in the presence of little amount of HCl and NH₃. Hence, the elevated dew point and aqueous solution salt concentration can only be estimated by ionic equilibrium simulation and not by simple correlations [6].

5.1 Chemical Equilibria in a Wet Ammonium Chloride System

Below give an example of wet ammonium chloride system with the following chemical species:
- Vapor phase: NH₃ (v), HCl (v), and H₂O (v)
- Aqueous phase: NH₃ (aq), HCl (aq), NH₄⁺, Cl⁻, H⁺, OH⁻
- Solid phase: NH₄Cl (s) (possible)

The following chemical equilibria exist amongst these species:
- Vapor/Liquid Equilibrium
  \[ \text{HCl (v)} \leftrightarrow \text{HCl (aq)} \]
  \[ \text{NH₃ (v)} \leftrightarrow \text{NH₃ (aq)} \]
- Sublimation/Salt Formation Equilibrium
  \[ \text{HCl (v) + NH₃ (v)} \leftrightarrow \text{NH₄Cl (s)} \]
- Dissolution/Precipitation Equilibrium
  \[ \text{NH₄Cl(s)} \leftrightarrow \text{NH₄⁺ + Cl⁻} \]
- Acid/Base Equilibrium
  \[ \text{NH₃ (aq) + HCl(q)} \leftrightarrow \text{NH₄⁺ + Cl⁻} \]
  \[ \text{H⁺ + OH⁻} \leftrightarrow \text{H₂O} \]

Where HCl(aq) and NH₃(aq) (or NH₃ and H₂O) are physically dissolved HCl and NH₃ molecules in the aqueous phase not yet dissociated (i.e ionized). The equilibria hold for systems with no liquid hydrocarbon phase or where the effect of liquid hydrocarbon phase is considered negligible. This is because the solubility of the above species, especially the ionic species, in hydrocarbon liquids are very small and may be neglected [3].

6. Case study

D. R. Clarida investigation on the reoccurring issues with HDS effluent exchanger corrosion came to a conclusion of ammonium chloride corrosion existence. He stated that it was as a result of the inadequacy to eliminating chlorides on the crude units due to lower quality of changed crudes, or extra ammonia is being formed on the HDS units [21]. The exchangers which are made of 304 stainless steel and carbon steel are found midway to the effluent train. Investigation explained that
change in the neutralizer chemistry in the refinery is much of important, [8]. Examination was made on a refinery that used much neutralizer injection to mitigate extra HCl available in the overhead system after turning the crude feed to opportunity crude. Extra added neutralizer injection resulted in a salt point temperature of 20°F (11°C) above the temperature of the dew point, resulting in underdeposit corrosion and system fouling. Reduction of the neutralizer's base strength resulted in a reduction in the salt point temperature, stopping fouling and underdeposit corrosion. Another study made was similar in the examination of corrosion and fouling in overhead-heat-exchangers. Also, a change to a lesser amine neutralizer base strength mitigated heat exchanger corrosion and fouling action. Gutzeit [14] investigated the sudden carbon steel tubes breakdown and stress corrosion cracking of 300 series stainless tubes. The crude feed contamination by chloride was discovered as the main source of the upset condition. Oil pipelines were periodically infected with organic/inorganic chlorides due to leakage of fed and hydrocarbon waste into the refinery. The organic chloride infection degree of the refinery was estimated to be 50ppm. This resulted in a 50-fold increase in the amount of HCl in the CDU overhead from the usual inorganic chlorides of about 4ppm. The greater degree of HCl in the overhead circuit gave much stronger acid attack and substantial extra salt formation. The dew point pH with the increased level of HCl was evaluated to be as small as pH 0. It was found that the temperature of salt formation increased to 270°F (132°C), resulting in salt deposits all through the overhead circuit. The formed deposits resulted into underdeposit via chloride fouling and corrosion in the steel tubes. Giesbrecht [24] noticed inadequate and insufficient water wash systems as the cause of overhead fouling and corrosion in two different investigations. The first study pinpointed the reason for exorbitant fouling to be inappropriate water wash installation of the nozzle. It was also revealed that changes in neutralizer chemistry as well as change in water wash are the main requirement to solving overhead circuit fouling challenges. Changes in neutralizing amine lead to lower formation of salt temperature while higher water wash flow enhanced the system to flush off formed salts. Jon Dobis [21], working on a Hydrocracker and HDS unit made of carbon steel found the second stage of the reactor effluent air cooler failing. The lower rows of the tubes were noticed to corrode. In the second stage, ammonium chloride was identified to exist but was sufficiently dry to give any challenge. Trays in the recycle splitter tower were lost, causing the first stage stripper to operate at temperatures between 10 and 25°F lower. Hence, little amount of water was carried into second stage. The water transfer enhanced the emergence of corrosion. The corrosion rate was identified to be between 75 to 100 mpy at the upset of the operation. The outlet header and piping also contained deposits of ammonium chloride. A pH <1 was estimated when the deposits were dissolved in water. This shows that deposition of dry ammonium chloride create little or no effect on the system but causes aggravated effect when dissolved in water. Joerg Gutzeit making investigation on hydrodesulfurization unit suggested that amine hydrochloride which has the same corrosive effect as ammonium chloride does not required water to cause corrosion due to its hydroscopic effect hence can draw water from the environment [14]. He stated that an ammonium chloride corrosion rate is functional to humidity. Corrosion rates ranged from 50 mpy to 60 mpy, with a corresponding humidity range of 20 to 40 %. Jim Edmondson (Betz) drew a distinction between ammonia and amine chlorides at the hydrodesulfurization unit. He stated that most of the amine chlorides are molten and in the absence of water, are far more aggressive than solid salts such as ammonium chloride. Toba used carbon steel, 304 stainless steel, duplex stainless steel, Grade 2 titanium, Alloy 400, Aluminum brass, and Aluminized carbon steel to examine the relative humidity effect of on ammonium chloride corrosion in refineries to ascertain the main cause of
corrosion caused by NH₄Cl salt deposition in reactor-effluent streams in hydroprocessing units, which are the major materials commonly used in refineries [17-18]. The humidity and temperature control chamber was used to simulate the corrosion rate of the solid NH₄Cl salt at a temperature, pressure and duration of 80°C, 1atm and 100h respectively between the ranges of 20 percent to 80 percent RH. Powdery NH₄Cl reagent was used to totally bury the samples in cylindrical polytetrafluoroethylene (PTFE) crucibles and placed in the control chamber for temperature and humidity. The coupon weight losses were used to calculate the corrosion rates. While the control chamber for temperature and humidity was used to estimate water absorption test. The tests showed the corrosion rate to be highly influenced by relative humidity, RH [7]. Hence, the corrosion rate and water absorption of the salt is a direct function of the critical RH, which showed that critical RH is a determining factor to corrosion rate in NH₄Cl environment. Carbon steel gave the highest level of degradation of 7.15 mm/y above 50% RH and decreases to about 1.35mm/y at 80% RH. The corrosion rate of aluminized carbon steel was less than that of the carbon steel with 1.54 mm/y corrosion rate at its peak at about 60%RH. Aluminum-brass had a similar corrosion rate as aluminized carbon steel at about 60% RH. Grade 2 titanium and Alloy 400 showed moderate rate of corrosion to be 0.24mm/y and 0.25mm/y at 50%RH and 60%RH respectively. 304 stainless steel and duplex stainless steel weight loss was negligible, but at 50% and 60% RH pitting was observed. Alloy C-276 showed neither pitting nor general corrosion effect, but at 50% RH it tarnished.

Table 2. Summarized the type of corrosion encountered [18]

| Alloy type         | Form of corrosion          |
|-------------------|----------------------------|
| Carbon steel      | aggressive general corrosion|
| Aluminized carbon steel | aggressive general corrosion |
| Aluminum brass    | aggressive general corrosion|
| Grade 2 titanium  | general corrosion           |
| Alloy 400         | general corrosion           |
| Type 304 stainless steel | Pitting corrosion         |
| Duplex stainless steel | Pitting corrosion        |

The appearance of each specimen gotten from the corrosion test was given thus: Aggressive general corrosion was noticed on both aluminized carbon steel, aluminum brass, and carbon steel. Removing from the salt crucible, the carbon steel surface was covered with thick brownish corrosion products. The 60%RH specimen gave the most abundant corrosion products. Thick corrosion product was also noticed on aluminum brass at the end of the test. The Al and Al/Al-Fe layers in the aluminized carbon steel corroded exposing the base metal at 60% RH and above. The case of general and pitting corrosion were not seen on the surface between 20% and 40% RH. The grade 2 titanium and Alloy-400 at 50% and 60% RH produced general corrosion respectively. Grade 2 titanium and Alloy 400 were found to tarnished at 50% RH and at all RH, respectively. 304 stainless steel, duplex stainless steel, and Alloy C-276 did not show effect of general corrosion. 304 stainless steel and duplex stainless steel respectively produced pitting at 50%RH and 60%RH. A condenser system was identified by Chambers [7] that the pH measurement between 2-3 pH units less than the predicted modeling and simulation pH values. Chemical analysis of condenser system samples was discovered that extreme high non-HCl acidic species concentrations were available. Sulfate, bisulfates, thiosulfates, carbonates, and carboxylic acids were the available.
acidic species. The investigation showed that desalter water wash oxygen content has significant difference between 10 ppm to 3.5ppm. Higher oxygen content in the distillation unit resulted into sulfur-oxide formation by the reaction of H2S with O2. The pH values obtained were found to be much close to the ones predicted by modeling after the correction of the desalter water wash oxygen content issue. Investigation on the neutralizer effect [25-26] showed that outrageous utilization of ammonia as a neutralizer can result in great fouling of NH4Cl. By using modeling to study a lower dose of ammonia in the system, the overhead system temperature was found to remain above the temperature of the salt point, hindering fouling and salt formation. Study showed the major fouling issues were discovered using modeling in an overhead system disclosed that the challenged area of the overhead system operated at 42°F (23°C) below the temperature of the salt point. Increasing the temperature to 25°F (14°C) above the salt point temperature caused the overhead circuit to stop fouling. Giesbrecht et al [24] discovered inadequate and insufficient water wash system along the heat exchanger system. It was revealed that installing water wash nozzle wrongly on the immediate upstream of 90° elbow was the cause of excessive fouling that made no water to reach the back end of a bundle of heat exchangers. He concluded that changing both the neutralizer chemistry and the water wash are much demanded in order to eliminate the overhead circuit fouling challenges. The neutralizing ammonium chloride and/or amine change leads to lowered formation of salt temperature while higher water wash flow permitted the flushing of salts formed from the system. Clarida et al [8] also pinpointed some heat exchangers which utilized standard neutralizing amine resulting to corrosion with rates surpassing 30mpy (0.8 mm/yr). This was equipped with water wash system to reduce the corrosion effect to less than 5mpy (0.1 mm/yr). Although the system of the water wash may be able to reduce the problems of fouling and under-depositing corrosion, water wash erosion became a major problem.

7. CONCLUSION
Due to the thermodynamic behavior of ammonia in the refinery system adequate eliminating system for chloride and proper ammonia control into the system is demanded to keep the system at check against the corrosion action of ammonium chloride. The understanding of the neutralizers mechanism such as ammonia will strongly curb refinery system degradation.

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