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A LABORATORY EVALUATION OF NH₃ INJECTION TECHNOLOGY TO REDUCE HCI EMISSIONS FROM INCINERATORS

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ABSTRACT

A laboratory-scale experimental study is conducted to investigate the fundamental properties of NH_3 -HCl reaction in a simulated incinerator duct system. The NH_3 injection technology is a dry HCl removal process which produces solid products. The test results showed that under typical incineration operation conditions, the outlet concentration of HCl can meet the emission standards for incinerators in Taiwan. An excessive injection of NH_3 above stoichiometric requirement may not lead to the NH_3 slip problem. This is because that the excessive amount of NH_3 would probably diffuse onto the product particles. However, the enhancement of NH_3 utilization is economically not feasible. The outlet concentrations of HCl and NH_3 are almost independent of their inlet concentrations for residence time longer than 1 second. Moisture content and the NH_3 /HCl injection ratio are two of the most important factors which affect the HCl removal and NH_3 utilization.

Key words: NH₃ injection technology, NH₄Cl particles, ammonia slip, incineration

INTRODUCTION

The treatment and disposal of solid wastes is a worldwide concern problem. In order to manage the increasing quantity of solid wastes, incineration becomes one of the best recognized waste treatment technologies. Although the waste disposal problem can be minimized by the incineration process, the secondary air pollution problem of acid gas emissions is another major concern in the successful operation of an incinerator system. Among the acid gases, hydrogen chloride (HCl) is the most prevalent one. If not controlled, HCl gas may have an adverse effect on public health or welfare. It also creates corrosion problem in the incineration system.

Traditional HCl removal technologies include wet processes (e.g. scrubbers), semi-dry processes (e.g. spray dryer absorbers) and dry injection technologies (e.g. calcium hydroxide or sodium bicarbonate duct injection). These three categories of HCl removal processes have their distinct advantages and disadvantages. For example, the removal efficiency of a wet scrubber is the highest as compared to semi-dry or dry processes. However, its installation and operation costs are also the highest. It also produces water pollution problem. On the other hand, the dry injection technology has the lowest installation and operation costs. It requires less space but its HCl removal efficiency is the lowest.

Ammonia (NH₃) injection technology is a dry injection process for removing acid gases. It is commonly used in

reducing NOx emissions (for example, Selective Catalytic Reduction, Selective Noncatalytic Reduction, and Thermal DeNOx). The excellent reactive characteristic of ammonia also attracts researchers for tests on removing other major gaseous pollutants such as SO2 [1, 2] and HCl. Takacs and Moilanen [3] are the first to demonstrate the feasibility of HCl removal by ammonia injection technology. The product of NH3-HCl reaction was assumed to be NH4Cl solid particles. In their bench scale and pilot scale studies, the removal efficiency of HCl gas could be above 90% at inlet NH3 /HCl molar ratios of above 1. If this is truly attainable, the disadvantage of low HCl removal of traditional dry processes does not exist for the NH3 injection technology. In addition, since NH3 is commonly used to remove NOx from the incinerator, the cost for removing one additional acid gas becomes much less

It seems that ammonia injection technology is a promising route for the simultaneous removal of several gaseous pollutants from incinerators. However, the operating conditions were not specified clearly in the study of Tackas and Moilanen [3] and Tackas *et al.* [4]. Besides, the mechanisms which lead to a high HCl removal have not been studied. In this paper, results of a laboratory scale experimental study are presented to investigate the fundamental properties of NH₃-HCl reaction in a simulated incinerator duct system. The effects of reactor residence time, inlet HCl concentration, NH₃ /HCl injection ratio, and moisture content on the HCl removal efficiency as well as the

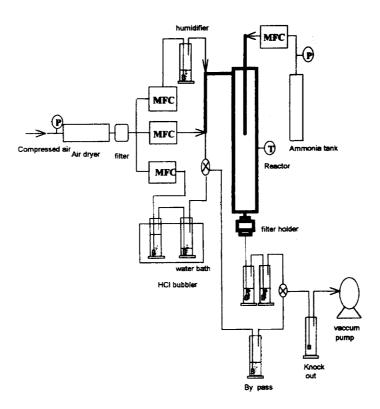


Figure 1. Experimental setup of the NH₃-HCl reaction system. The bold line indicates the part of experimental system that is temperature controlled.

NH₃ utilization are evaluated. A thermodynamic equilibrium equation without the presence of any moisture is also derived to assist in a better understanding of the effect of moisture content on the NH₃-HCl reaction.

EXPERIMENTAL SYSTEM

The experimental setup is shown schematically in Figure 1. The reaction system was a laminar flow tube reactor made of stainless steel. The inner diameter of the reactor was 22 mm, with length adjustable to obtain different reactor residence times. The inlet NH₃ gas (1% NH₃ in N₂) was introduced from a gas cylinder and then entered the top of the reactor. Tests have also been done with reactors of different diameters (11, 22 and 50 mm) to ensure the well-mixing of NH₃-HCl reaction. The test results indicated that the performance results for 11 and 22 mm diameters were almost identical. But diffusional limitation was observed using 50 mm diameter tube and the NH₃-HCl reaction rate was slower than that obtained from smaller inlet tubes.

The inlet HCl concentration was obtained by passing clean, compressed air through two bubblers in series which contain 8.5M HCl solution. The HCl bubblers were kept in a constant temperature water bath. Before entering the reactor, the HCl vapor stream was preheated to further vaporize water vapors and HCl molecules. Besides, before start-up of each experimental test, a HCl bypass impinger was used so that the concentration of HCl vapors was as stable as

possible. The HCl concentration introduced from the bubblers before dilution was 3400 ± 100 ppm.

Another two streams of clean, compressed air were served as the diluting and humidifying gases. The clean air was provided by passing the air stream through a silica gel air dryer and a Gelman-TDC filtration system. The flow rates of all gaseous streams were controlled by mass flow controllers (MKS, Andover, MA). The total flow rate in the reactor was 3.2 lpm. Temperatures of the whole reactor and preheat system were controlled by three temperature controllers and heating tapes. The temperature profile along the inner reactor was recorded before each experimental test. The maximum temperature variations along the inner reactor were controlled to within ±2.5°C. The NH₄Cl product particles were collected by a Whatman 47-mm 0.1 µm cellulose nitrate membrane filter connected to the reactor outlet. The filter holder was also maintained at the same temperature as that of the reactor. The absorption of HCl by H₂SO₄ solution was according to the recommended method of US EPA [5]. Since H₂SO₄ also absorbs NH₃ gas, the waste gas stream was passed through two impingers containing 0.1N H₂SO₄ solution for absorption of un-reacted gases. The length of each run was 15 minutes for all tests.

It was found that a significant amount of reacted particles was deposited on the reactor wall. When the reactor was insulated with ceramic fibers, there were about 50% by mass of particles deposited on the reactor surface instead of on the collected filter. This may be due to the electrostatic

force exerted by the freshly formed particles. Therefore after each experimental test, the filter was weighted and then the collected particles were extracted by washing the filter and the reactor wall separately with double-distilled water. The measurement of the solution concentrations from the extracted particles represents the reacted amount of HCl and NH₃. The HCl and NH₃ concentrations were determined by Chromatography (Dionex-4500i) and Spectrophotometer (Hitachi U-3210), respectively, as Cl and NH4+ ions. An overall mass balance was checked after each run. This was done by summing up the Cl and NH4+ ion concentrations from the extracted particle solutions and impinger solution, and compared with the inlet gas concentrations. It was found that an overall mass balance can be achieved within 5% deviations.

Equilibrium Theory Without Moisture Content

Although moisture is present in a significant amount in an incineration system, the chemical reaction kinetics as well as the thermodynamics of the $\rm NH_3$ -HCl-H₂O reaction are not available in the literature. Therefore in the theoretical analysis, the $\rm NH_3$ -HCl reaction under the absence of water vapors is presented. As the ammonia gas is injected and reacted with the HCl gas, a solid $\rm NH_4Cl$ product is formed and the reaction can be written as,

$$NH_{3(g)} + HCl_{(g)} \leftrightarrow NH_4Cl_{(s)}$$
 (i)

This reaction is widely used in the literature for both flue gas system and atmospheric studies [3, 6] If the NH_3 - HCl reaction proceeds very fast, then the maximum achievable HCl removal may be estimated by the equilibrium theory between NH_3 , HCl gases and the NH_4Cl solid particles. This maximum achievable HCl removal efficiency, η , is expressed as,

$$\eta = \left(\frac{P_{HCl}^{i} \times P_{HCl}^{e}}{P_{HCl}^{i}}\right) \times 100\%$$
 (ii)

Where P_{HCI}^{i} and P_{HCI}^{e} represent the inlet and equilibrium HCl vapor pressures, respectively. And the equilibrium vapor pressures can be obtained from the system equilibrium constant, $K_{p'}$

$$K_{p} = \left(P_{HC1}^{e} \times P_{NH_{3}}^{e}\right)^{-1}$$
 (iii)

The value of K_p is a function of temperature which can be derived from the van't Hoff's equation [7],

$$\ln\left(K_{p}\right) = \frac{\Delta H^{0}}{RT} - \frac{\Delta G^{0} - \Delta H^{0}}{298R} - \frac{1}{RT} \int_{298}^{T} \Delta C_{p}^{0} dT + \int_{298}^{T} \frac{\Delta C_{p}^{0} dT}{T}$$
(iv)

Table 1. Thermodynamic properties of HCl, NH₃ gases and NH₄Cl solid particles under standard conditions.

	ΔH _f °	ΔG _f °	C,°
NH ₃	-10.98	-3.929	6.7+0.00630T (T=300-800°K
			(1-300-000 K
HCl	-22.063	-22.777	6.7+0.00084T
			(T=273-
			2000°K)
NH₄Cì	-75.332	-48.73	9.8+0.0368T
			$(T=273-457^{\circ}K)$

 $\Delta H^0 = 42.289 \text{ Kcal/mole}, \Delta G^0 = 22.025 \text{ Kcal/mole}, \Delta C_p^0 = 3.6-0.297T, cal/mole 0K$

reference: Parker et al. [8] Perry and Green [9]

Where ΔH^0 and ΔG^0 are the standard enthalpy and standard free energy changes for the reaction at 298°K, and $\Delta C_p^{~0}$ is the difference between the specific heats of the products and the specific heat of the reactant at temperature T and standard conditions. The thermodynamic properties of the reactants and the product are listed in Table 1 [8, 9]. Using the data of Table 1, the temperature dependence of K_p is,

$$\ln K_p = -1.8116 \ln T + 21404.5 T^{-1} - 54.1731 + 0.007473 T$$
 (v)

where the unit for K_p is ppm⁻². Equation (v) is applicable in the temperature range from 300 to 457°K. The values of K_p at low temperatures are in excellent agreement with those derived by Pio and Harrison [6] which are applicable for atmospheric environment.

RESULTS AND DISCUSSION

Effect of Reactor Residence Time

Although the purpose of this study is not to investigate the chemical kinetic of NH3 -HCl reaction, it may be of interest to understand that whether the NH3-HCl reaction reaches a state of quasi-equilibrium during possible residence time in the incinerator system. The value of natural logarithm of the product of HCl and NH3 concentrations (i.e. $ln(C_{NH3}*C_{HC})$) as a function of residence time in the reactor is plotted in Figure 2. Experiments were conducted for inlet HCl concentrations of 270±5 and 990±20 ppm. The inlet ratios of NH₃/HCl were from 0.95 to 1.05. Reaction temperature was 127.5±2.5°C. This temperature was chosen since the potential application of NH3 injection to remove HCl gas is economically feasible at higher duct temperatures. And the highest operation temperature conducted by Takacs and Moilanen [3] was 127°C. The test range of reactor residence time was from 0.45 to 4.28 seconds. The test data for an inlet HCl concentration of 270±5 ppm and residence times of 2.39

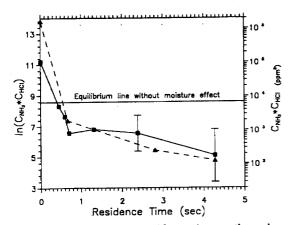


Figure 2. Effect of reactor residence time on the value of $\ln(C_{NH3}^*C_{HCl})$. The inlet NH_3/HCl molar ratio is approximately 1.0 and the reaction temperature is 127.5°C: (=-=-) HCl inlet concentration of 270 ppm; (Δ - Δ - Δ) HCl inlet concentration of 990 ppm.

and 4.28 seconds were repeated and sample standard deviations are indicated in Figure 2 with error bars. No water vapor was added into the experimental system. However, a slight amount of moisture could not be avoided which was introduced from the HCl bubblers. Moisture contents were less than 0.4% v/v.

As can be seen in Figure 2, the reaction proceeds quickly for residence times less than around 0.7 second. For reactor residence times longer than around 1 second, the effect of inlet HCl concentration becomes less significant. The values of ln(C_{NH3}*C_{HCl}) are approximately the same for inlet HCl concentrations of 270 and 990 ppm, they decrease from around 7 to 5 as reactor residence times increase from 1 to 4.28 seconds. Although increasing the residence time leads to a decrease in the value of ln(C_{NH3}*C_{HCI}), it may not be able to have such a long residence time available in the duct of an incinerator system. But the values of ln(C_{NH3}*C_{HCl}) from 5 to 7 correspond to NH3 and HCl outlet concentrations of 12 to 33 ppm for a 1:1 inlet ratio of NH3:HCl. Therefore the reaction rate becomes so slow that it can be treated as at a near steady-state from engineering point of view. Also to be noted is that the equilibrium prediction of ln(C_{NH3}*C_{HCl}) at 127.5°C is 8.58, which is larger than any of the experimental data in Figure 2. This seems not possible since thermodynamic equilibrium predictions indicate the lowest achievable outlet concentrations of NH3 and HCl, unless there are other factors which enhance the reaction potential of the NH3-HCl reaction system. Possible factors may include moisture effect and additional adsorption of NH3 and HCl gaseous molecules onto the product particles. The additional reaction of NH3 and HCl gaseous molecules could occur in the free space, or on the deposited particles. If further consumption of NH₃ and HCl molecules were on the surface of particles which deposited on the filter, then the reaction time was extended.

Effect of Moisture Content

Previous results on the HCl removal and NH₃ utilization were conducted under low moisture content conditions. The amount of water vapors in the flue gas of an incineration system may vary from a few percentages to more than 20% by volume. It depends mainly on the moisture content in the solid wastes. Therefore water vapors were introduced purposely by passing a clean stream of air into a humidifier, which were then mixed with HCl and NH₃ gases in the reactor. The outlet concentrations of HCl and NH₃ as a function of moisture content are plotted in Figures 3a and 3b.

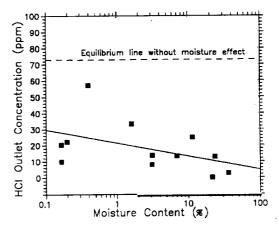


Figure 3a. Effect of moisture content on the HCl outlet concentration at temperature of 127.5°C and residence time of 2.39 seconds. The inlet ratio of NH $_3$ /HCl is approximately 1.0. Solid line is the regression curve of the experimental data.

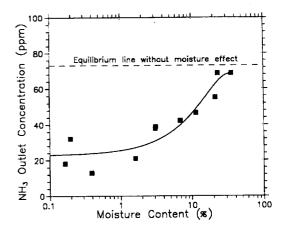


Figure 3b. Effect of moisture content on the NH₃ outlet concentration at temperature of 127.5°C and residence time of 2.39 seconds. The inlet ratio of NH₃/HCl is approximately 1.0. Solid line is the regression curve of the experimental data.

The reaction temperature was 127.5°C and reactor residence time was 2.39 seconds. It was observed from Figure 2 that HCl and NH $_3$ inlet concentrations do not significantly influence their outlet concentrations. Therefore the HCl and NH $_3$ inlet concentrations ranging from200 to 300 ppm were chosen as the data basis. The inlet molar ratios of NH $_3$ /HCl ranged from 0.90 to 1.10. The regression lines of the experimental data are also shown.

As can be seen in Figure 3a, the HCl outlet concentration decreases slightly as the moisture content increases. The outlet concentrations of HCl can meet the emission standard for incinerators in Taiwan, 40-60 ppm. Besides, all of the experimental data are lower than the thermodynamic predictions when water vapors are absent, that is, HCl equilibrium concentration of 73 ppm. There are two possible routes for the enhancement of HCl removal. The first is that water vapors act as catalysts or reactants in the NH3-HCl reaction in the free space. The other route is that the deposited NH₄Cl particles continue to be exposed to the gas stream, the product particles (whether they were in the free space or deposited on the filter) and the filter materia provided new media for heterogeneous reaction of NH2 and HCl molecules. The heterogeneous reaction is affected by moisture content as shown in Figure 3a. Both routes could occur individually or simultaneously.

However, as seen in Figure 3b the outlet concentration of NH₃ increases with an increase in the moisture content. This is opposite to the dependence of HCl outlet concentration on the moisture content, and indicates that the increasing removal of HCl gas with increasing moisture content leads to an inhibition of NH₃ utilization. But it is also seen that the NH₃ outlet concentrations are still lower than its equilibrium prediction, 73 ppm, under absence of water vapors. This indicates that NH₃ molecules were also consumed due to heterogeneous reaction.

In order to explore the possible reason for the enhancement of HCl and NH₃ reaction by diffusional deposition followed by heterogeneous reactions on the product particles, the product particles were taken for Scanning Electron Microscopic (SEM) analysis. The morphology of product particles in the SEM pictures appears to be very porous. The high porosity of the particles provides high surface areas and increases the chance of diffusion of water vapors, NH₃ and HCl molecules onto the product surfaces to undergo heterogeneous reactions. It was also observed during experimental tests that the product particles exert an electrostatic force. This will increase the deposition rate of gaseous molecules by diffusion.

Effect of Inlet Ratio of NH3/HCl

Figure 4a shows the effect of inlet molar ratio of $\mathrm{NH_3/HCl}$ on the HCl removal under typical moisture contents of an incineration system (>3.0% $\mathrm{H_2O}$ by volume). The inlet $\mathrm{NH_3}$ concentration was 260±30 ppm and HCl inlet concentration was varied to obtain the desired inlet molar

ratio. Reactor residence time was 2.39 seconds and reaction temperature was 127.5 °C. The theoretical equilibrium predictions on the HCl removal with the absence of water vapors are also shown. The solid line is plotted with an inlet $\rm NH_3$ concentration of 260 ppm and the two dashed lines are the upper and lower bonds of predicted results with inlet $\rm NH_3$ concentrations of 230 and 290 ppm, respectively. It is seen that the theoretical predictions on HCl removal increase with an increase in the $\rm NH_3/HCl$ inlet ratio for substoichiometric inlet ratios of $\rm NH_3/HCl$. The HCl removals are not enhanced as the $\rm NH_3/HCl$ injection ratios are greater than 1. But the experimental data do not show this tendency. The HCl removals are almost independent of the $\rm NH_3/HCl$ inlet ratio. The HCl removals for $\rm NH_3/HCl$ inlet ratios of less than 1.0 indicated that they are almost 30% more

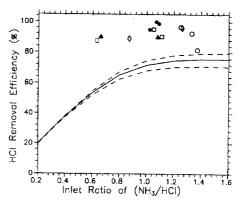


Figure 4a. Effect of NH₃/HCl inlet ratio on the HCl removal efficiency at temperature of 127.5EC and residence time of 2.39 seconds: (OOO) H₂O=3.1%; (\square \square) H₂O=6.9%; (\triangle \triangle) H₂O=11.3%; (\lozenge \lozenge) H₂O=12.6%; and (\bigcirc \bigcirc) H₂O> 20% v/v. Lines are equilibrium predictions under no moisture content.

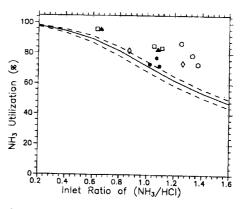


Figure 4b. Effect of NH₃/HCl inlet ratio on the NH₃ utilization at temperature of 127.5EC and residence time of 2.39 seconds: (OOO) H₂O=3.1%; (□ □ □) H₂O=6.9%; (▲▲▲) H₂O=11.3%; (⋄◊⋄) H₂O=12.6%; and (● ●) H₂O> 20% v/v. Lines are equilibrium predictions under no moisture content.

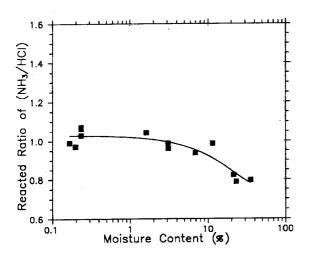


Figure 5. Effect of moisture content on the NH_3/HCl reaction ratio for inlet ratio of approximately 1.0. The reaction temperature is 127.5EC and residence time is 2.39 seconds. Solid line is the regression curve of the experimental data.

than the predicted values.

Figure 4b shows the effect of NH₃/HCl inlet ratio on the NH₃ utilization. The experimental conditions were the same as those for Figure 4a. The experimental data of NH₃ utilization are higher than equilibrium predictions. The exception is that data conducted at NH₃/HCl inlet ratios of around 1.0 and moisture content of larger than 20%. It has been demonstrated that for NH₃/HCl injection ratios between 0.8 and 1.2, the HCl removals are approximately the same. On the other hand, an increase in the NH₃/HCl injection ratio of greater than 1 leads to over-consumption of NH₃ gas.

Therefore under typical moisture content conditions in the incinerator system, the optimal injection ratio of NH_3/HCl would be slightly less than 1.

Reaction Ratio of NH₃/HCl

If diffusion of gaseous molecules onto surfaces to undergo heterogeneous $HCl-H_2O$ and NH_3-H_2O reactions is one of the mechanisms for the removal of HCl and NH_3 gases other than chemical reaction equation (i), the reaction ratio may not be stoichiometric, i.e. 1:1. This was checked by Figure 5 that shows the effect of moisture content on the NH_3/HCl molar reaction ratio. The NH_3/HCl inlet ratios were from 0.96 to 1.05. As can be seen that the regression values of

 $\mathrm{NH_3}/\mathrm{HCl}$ reaction ratio are slightly above 1.0 under low moisture contents. This may be due to that the diffusivity of $\mathrm{NH_3}$ is almost double of the diffusivity of HCl in air. Therefore $\mathrm{NH_3}$ gaseous molecules have higher tendency to diffuse to the product particles. Increasing the moisture

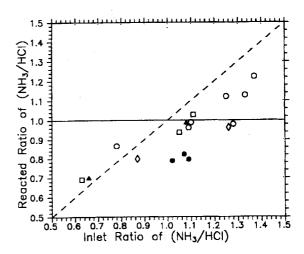


Figure 6. Effect of NH₃/HCl inlet ratio on their reaction ratio at typical moisture content of incineration system. The reaction temperature us 127.5EC and residence time is 2.39 seconds: (OOO) $H_2O=3.1\%$; ($\Box\Box\Box$) ($H_2O=6.9\%$;($\Delta\Delta\Delta$) $H_2O=11.3\%$; ($\Diamond\Diamond\Diamond$) $H_2O=12.6\%$; and ($\bullet\bullet\bullet$) $H_2O>20\%$ v/v.

content leads to a decrease in the reaction ratio of $\mathrm{NH_3/HCl}$. This indicates that the heterogeneous $\mathrm{HCl\text{-}H_2O}$ reaction is enhanced by the presence of large quantity of water vapors.

The $\mathrm{NH_3/HCl}$ molar reaction ratio as a function of $\mathrm{NH_3/HCl}$ inlet ratio is shown in Figure 6 for typical moisture content in an incineration system (>3.0% $\mathrm{H_2O}$ by volume).

The symbols are experimental data, solid line is the prediction of 1:1 reaction ratio for NH₃/HCl reaction, and dash line is where the NH₃/HCl inlet ratio is equal to the reaction ratio. If the NH₃-HCl reaction follows reaction equation (i), the experimental data should fall around the solid line. This is not observed in Figure 6. Instead, for NH₃/HCl inlet ratios of less than 1.0 the experimental data fall around the dash line. And an increase in the NH₃/HCl inlet ratio leads to an increase in its reaction ratio.

CONCLUSION

Several factors which influence the NH₃-HCl reaction system have been examined in this study. The experimental results indicated that inlet concentrations of HCl and NH₃ have a slight effect on their outlet concentrations as reaction residence time of longer than 1 second. Under typical incinerator duct systems, the outlet concentrations of HCl could meet the emission standards for incinerators in Taiwan. An excessive injection of NH₃ above the stoichiometric requirement may not lead to the NH₃ slip problem. This is because that the excessive amount of NH₃ would probably diffuse onto the product particles. However, the enhancement of NH₃ utilization is economically not feasible. Therefore the optimal NH₃/HCl injection ratio is slightly less

than 1.

The presence of water vapors lowers the values of $\rm NH_3$ and HCl concentrations as compared to their equilibrium predicted values where no water vapor presents. This may be due to that water vapors enhance the heterogeneous reaction between $\rm NH_3$ - $\rm H_2O$ and HCl - $\rm H_2O$ molecules. The ability of heterogeneous HCl- $\rm H_2O$ reaction is higher than the $\rm NH_3$ - $\rm H_2O$ reaction at very high moisture content conditions.

Because study of NH₃-HCl reaction under absolutely no water vapor condition is not possible under the experimental setup used in this study, a fully understanding of the role of water vapors on the NH₃-HCl reaction can not

be achieved at the present time. This may be an interest topic for future work since the reaction between NH₃, HCl and water vapors has wide applications not only in the removal of HCl by NH₃ injection technology. It is also a basic reaction for studying the mechanisms of gas-to-particle formation processes in the fields of aerosol science and atmospheric science.

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