

## Article

# Effects of Humidity Pretreatment Devices on the Loss of HCl Gas Emitted from Industrial Stacks

Han-Nui Gil, Trieu-Vuong Dinh <sup>\*</sup>, Jee-Hyun Lee, Byeong-Gyu Park, In-Young Choi, Sang-Woo Lee, In-Young Kim and Jo-Chun Kim 

Department of Civil and Environmental Engineering, Konkuk University, 120 Neungdong-ro, Gwangjin-Gu, Seoul 05029, Korea; hnui6427@konkuk.ac.kr (H.-N.G.); hyunlee0609@konkuk.ac.kr (J.-H.L.); smarteddy@konkuk.ac.kr (B.-G.P.); force@konkuk.ac.kr (I.-Y.C.); dk1553@konkuk.ac.kr (I.-Y.K.); lsw0511@konkuk.ac.kr (S.-W.L.); jckim@konkuk.ac.kr (J.-C.K.)

\* Correspondence: trieuvuong88@konkuk.ac.kr; Tel.: +82-2-455-2994

**Abstract:** A high humidity at a high temperature presents a common challenge in monitoring the air pollutants emitted from stationary sources. Thus, humidity removal is a pivotal issue. In this study, the effect of humidity pretreatment devices (HPDs) on hydrogen chloride (HCl) gas emitted from an incinerator stack was investigated. A conventional cooler (HPD\_CL), and poly-tube (HPD\_NP) and single-tube (HPD\_NS) Nafion™ dryers were used as HPDs in this study. HCl concentrations varied at five and 10 parts per million in volume (ppmv). Low (i.e., ~4%) and high (i.e., ~17%) humidities were generated at 180 °C. The removal efficiencies of humidity and the loss rates of HCl by the devices were determined. The removal efficiencies of humidity by HPD\_CL and the two dryers were found to be similar, at approximately 85% at a low humidity and 95% at a high humidity. In terms of HCl loss rates, HPD\_CL revealed the highest loss rates in all conditions (i.e., >95%), followed by HPD\_NP and HPD\_NS. At normal room temperature (i.e., 25 °C), the HCl loss rates of HPD\_NP were >40% at a low humidity and >70% at a high humidity, while those of HPD\_NS were >10% at a low humidity and >60% at a high humidity. The performance of the two dryers improved when they were heated to 80 °C. However, this temperature caused damage to the dryers, which reduced their lifetime.

**Keywords:** HCl; cooler; Nafion™; CEMS; incinerator



**Citation:** Gil, H.-N.; Dinh, T.-V.; Lee, J.-H.; Park, B.-G.; Choi, I.-Y.; Lee, S.-W.; Kim, I.-Y.; Kim, J.-C. Effects of Humidity Pretreatment Devices on the Loss of HCl Gas Emitted from Industrial Stacks. *Atmosphere* **2022**, *13*, 33. <https://doi.org/10.3390/atmos13010033>

Academic Editors: David Reed and Ari Preston

Received: 22 November 2021

Accepted: 24 December 2021

Published: 27 December 2021

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## 1. Introduction

Hydrogen chloride (HCl) is a colorless acid gas at room temperature and pressure because its boiling point is  $-85.05$  °C [1]. In the atmosphere, besides secondary formation from a reaction between  $\text{HNO}_3$  and sea-salt (i.e., NaCl), HCl is primarily emitted from industrial sources such as chemical production or semiconductor production and via combustion processes such as from medical waste or municipal solid waste incinerators [2–4]. The concentrations of HCl emitted from several stationary sources in Korea are shown in Table 1.

**Table 1.** Emission of HCl from various stationary sources in Korea [5].

No.	Emission Source	HCl Concentration (ppmv)
1	Municipal Solid Waste Incinerators	0.01~13.93
2	Medical Solid Waste Incinerators	0.28~2.02
3	Acid Treatment Facilities	0.01~3.18
4	Cement Factories	0.01~1.18

HCl gas causes direct and indirect adverse effects on human health [2–4,6,7]. HCl gas can cause corrosion to the eyes, skin, and respiratory system. Exposure to a low level of HCl (i.e.,  $\geq 5$  parts per million in volume (ppmv)) for a long time can damage human teeth. A person may die when exposed to a high level of HCl (i.e., >1000 ppmv). In the atmosphere, HCl gas is the precursor to ground-level ozone and fine particulate matter

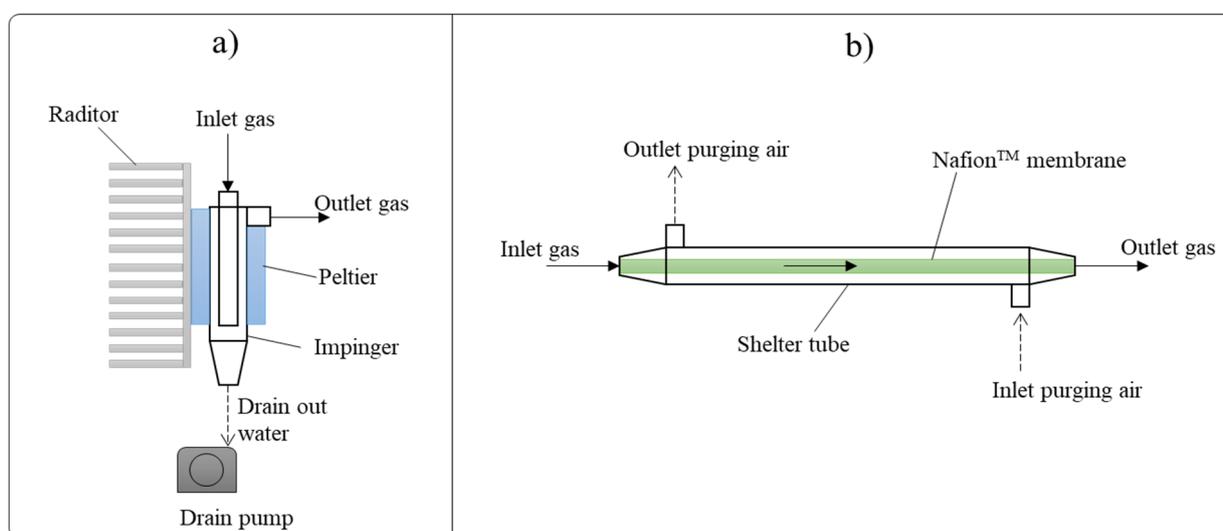
(PM) including  $PM_1$  and  $PM_{2.5}$  [2–4]. For example, HCl can react with ammonia gas in the atmosphere to product  $NH_4Cl$  particles [2,4]. Ozone and PM may have adverse effects on human health [6,7]. Therefore, anthropogenic HCl gas should be regulated. How the emission limits of HCl for municipal solid waste incinerators has been established in many countries is shown in Table 2.

**Table 2.** HCl emission limit (ppmv) for municipal solid waste incinerators [8–10].

Country	Existing		New	
	Small	Large	Small	Large
USA	250	31	25	25
Europe	171	21	17	17
Korea	15	20	~	~

USA: small incinerator < 250 tons/day; large incinerator >250 tons/day. Europe: small incinerator < 225 tons/day; large incinerator > 225 tons/day. Korea: small incinerator < 2 tons/hour; large incinerator >2 tons/hour.

The emission monitoring of HCl is a pivotal issue for the control of emissions of HCl from stationary sources. A continuous emission monitoring system (CEMS) has been widely applied to monitor HCl [11,12]. For the measurement of HCl in the CEMS, a non-dispersive infrared analyzer coupled with gas filter correlation and an ion electrode analyzer have been widely applied [11]. However, since HCl levels are only sub-ppmv (Table 1) and flue gas comprises a high humidity at high temperatures, this can cause damage to the analyzers and analytical bias [13,14]. Therefore, humidity should be removed from the flue gas before it enters the analyzers. Humidity pretreatment devices including coolers that remove humidity by cooling down the flue gas to condense water, and permeation membranes. The Nafion™ dryer has been widely used in this way, and it is usually employed to remove humidity from flue gas in a CEMS [11,15]. The basic structures of a cooler and a Nafion™ dryer are presented in Figure 1 [14]. The cooler consists of an impinger—which is cooled to about 2–4 °C by a Peltier so that the water vapor in the flue gas at a high temperature may be condensed as liquid droplets and accumulated at the bottom of the impinger—and a drain pump to drain out liquid droplets [14]. With the Nafion™ dryer, there is a tube consisting of a Nafion™ membrane which has high selectivity to water. The sample gas normally passes through the dryer, then water vapor penetrates the membrane and follows the purging air, while other gases keep moving to the outlet of the dryer [14]. Since HCl has a high solubility in water (i.e., 720 g/L at 20 °C) [1] and is strongly acidic, it might be lost in these pretreatment devices. However, there has been little investigation of this issue.



**Figure 1.** Schematic diagrams of a cooler (a) and a Nafion™ dryer (b).

Accordingly, this study was conducted to investigate the effects of the humidity pretreatment device (HPD) on the measurement of HCl under the stack conditions of a solid waste incinerator with high humidity and temperature. The humidity removal efficiency of a cooler and Nafion™ dryers of the poly-tube and single-tube types were considered. The loss rates of HCl caused by these devices were also determined with respect to various humidity levels and HCl concentrations.

## 2. Materials and Methods

### 2.1. Experimental Apparatus

A cooler (HPD\_CL) (TC-Standard 6122, Buhler technologies, Ratingen, Germany) consisting of two Teflon impingers, a three-meter Nafion™ single-tube (HPD\_NS) (SWG-A01-36/KF, AGC Engineering Co., Ltd., Tokyo, Japan), and a 30 cm Nafion™ poly-tube (HPD\_NP) (PD-50T-12MPS, Perma Pure LLC, Lakewood, NJ, USA) were used as HPDs. Ion chromatography (Dionex Aquion, Thermo Fisher Scientific Inc., Waltham, MA, USA) with an anion column (S-14, Thermo Fisher Scientific Inc.) was used to analyze HCl. Mass flow controllers (MFC) (M3030V, Linetech Inc., Korea) were used to control the flow rates of N<sub>2</sub> and HCl standard gas. A humidity generator was made based on the bubbling method to produce humid standard gas. Two humidity sensors (645, Testo SE and Co. KGaA, Lenzkirch, Germany) were applied to determine the humidity before and after HPDs. An air sampler (XC-60, Apex Instruments Inc., Fuquay-Varina, NC, USA) was used to collect a HCl sample.

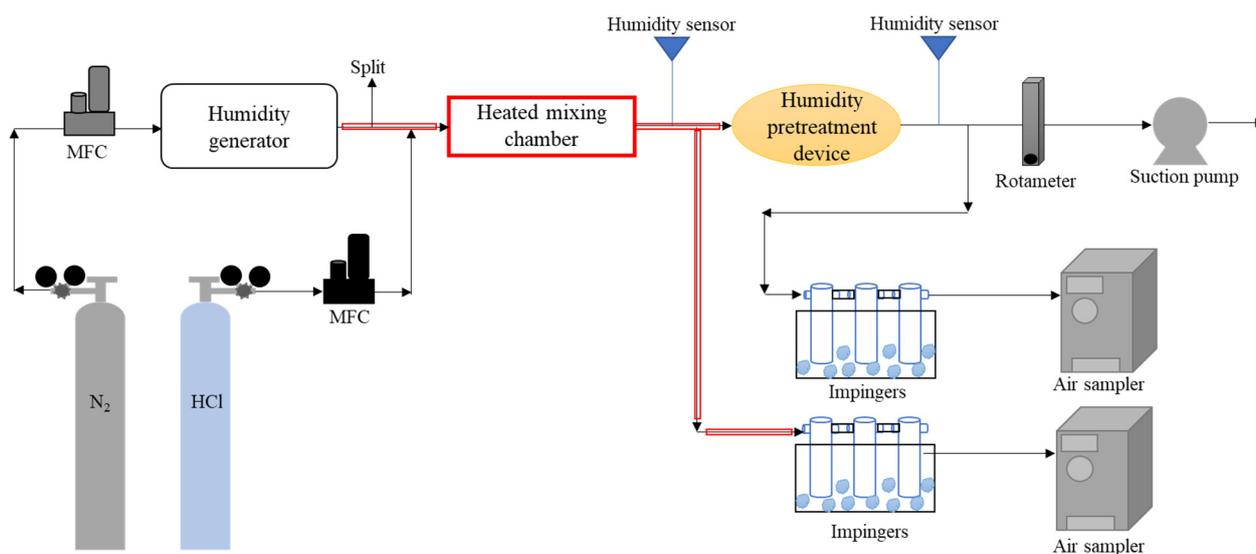
### 2.2. Experimental Materials

HCl standard gas (50 ppmv, Rigas Co., Ltd., Daejeon, Korea) and N<sub>2</sub> (99.999%, DongA Co., Ltd., Gyeonggi, Korea) were used for investigating the performance of HPDs in this study. Zero air (99.99%, DongA Co., Ltd.) was used as a purging gas for the Nafion™ dryers. These standard gases were generated based on the gravimetric method ISO 6142-1:2015, as reported by the manufacturers [16].

### 2.3. Experimental Procedure

#### 2.3.1. Investigation of the Humidity Removal Efficiency of Humidity Pretreatment Devices

The humidity removal efficiencies of HDP\_CL, HDP\_NP, and HDP\_NS were investigated. The experimental set-up is presented in Figure 2.



**Figure 2.** Experimental set-up for investigating the performance of humidity pretreatment devices.

The inlet humidity levels based on volume ratios were set as  $4 \pm 2\%$  (so call low humidity) and  $17 \pm 2\%$  (so call high humidity) at  $180\text{ }^\circ\text{C}$  based on the stack condition of a solid waste incinerator [17,18]. The mixing chamber and gaseous tubes were also heated to  $180\text{ }^\circ\text{C}$ . For HPD\_CL, its operating temperature was set as  $2\text{ }^\circ\text{C}$  and its air flow rate was  $2\text{ L/min}$  based on the recommendation of its manufacturer [19]. For HPD\_NP and HPD\_NS, their air flow rate was  $1\text{ L/min}$ . The purging flow by zero air of HPD\_NP was  $3\text{ L/min}$  and that of HPD\_NS was  $2\text{ L/min}$  based on the recommendation of its manufacturer [20,21]. Humidity before and after the HPDs were measured by humidity sensors as described in Section 2.1. These sensors were calibrated and approved by a responsible party in Korea (Korea Testing Laboratory, Gyeongsangnam-do, Korea). MFCs and rotameters were calibrated with a primary standard soap bubble method [22]. The experiment was conducted at  $25\text{ }^\circ\text{C}$  and  $1\text{ atm}$ , and repeated three times with a relative standard deviation (RSD) of  $<10\%$ . An ANOVA test was conducted to compare the humidity removal efficiency among HPDs using MATLAB (Version 9.10.0.1684407, MathWorks, Inc., Natick, MA, USA).

### 2.3.2. Investigation of the Loss Rates of HCl Caused by Humidity Pretreatment Devices

The effect of HPDs including HPD\_CL, HPD\_NP, and HPD\_NS on the loss of HCl was investigated. The experimental set-up is presented in Figure 2. The HCl concentrations were selected as 5 and 10 ppmv based on the field levels of HCl emitted from solid waste incinerators (Table 1). Other experimental conditions including the humidity of the sample gas, temperature, and the HPD set-up were also the same as the experimental conditions mentioned in Section 2.3.1. HCl samples before and after HPDs were collected by the impingers and analyzed by ion chromatography based on the standard method [23]. The method's detection limit was  $0.027\text{ ppmv}$  of HCl. For HPD\_CL, its performance was investigated at room conditions (i.e.,  $25\text{ }^\circ\text{C}$ ,  $1\text{ atm}$ ) [19]. For HPD\_NP and HPD\_NS, their performance was also investigated at room conditions as recommended by their manufacturers [20,21]. The loss rate of HCl was evaluated using Equation (1)

$$\text{Loss rate} = \frac{C_{\text{in}} - C_{\text{out}}}{C_{\text{in}}} \times 100\% \quad (1)$$

where  $C_{\text{in}}$  is the initial concentration of HCl before entering the HPDs and  $C_{\text{out}}$  is the concentration of HCl at the outlet of the HPDs. The experiment was repeated three times with an RSD of  $\leq 10\%$ . An ANOVA test and a t-test were conducted to compare the humidity removal efficiency among the HPDs using MATLAB (Version 9.10.0.1684407, MathWorks, Inc.).

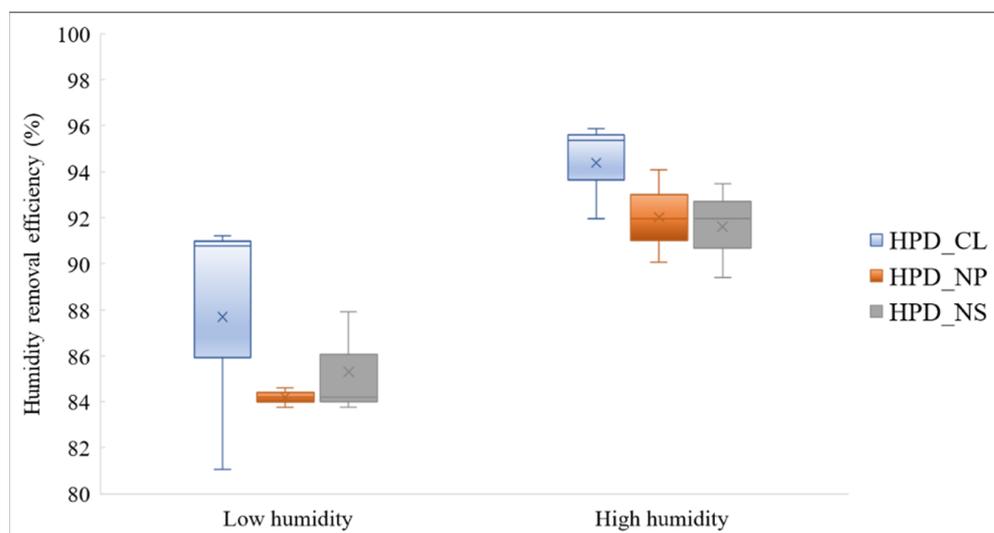
## 3. Results and Discussion

### 3.1. Investigation of Humidity Removal Efficiency of Humidity Pretreatment Devices

In terms of the humidity removal efficiency, the performances of three humidity pretreatment devices—HPD\_CL based on the condensation approach, and HPD\_NP, and HPD\_NS based on the permeation approach—were investigated with respect to various humidity levels based on the stack condition of a solid waste incinerator. The experimental results are depicted in Figure 3.

As shown in Figure 3, all HPDs had a good humidity removal efficiency. Although Figure 3 reveals that the humidity removal efficiency of HPD\_CL seems higher than those of the other devices, an ANOVA test concluded that their average humidity removal efficiencies were not significantly different ( $p$ -values  $> 0.05$ ). Furthermore, it was found that the humidity removal efficiencies in high humidity conditions were higher than those in low humidity conditions because of the higher humidity level, as was also reported elsewhere [24,25]. Although these studies were conducted at ambient humidity levels, the pattern was similar to that found in the current study. In general, all HPDs of concern performed well in terms of humidity removal efficiency. However, the humidity removal efficiencies of the Nafion™ dryers were similar to those of the cooler, although the investment and operating costs associated with Nafion™ dryers were much higher than those

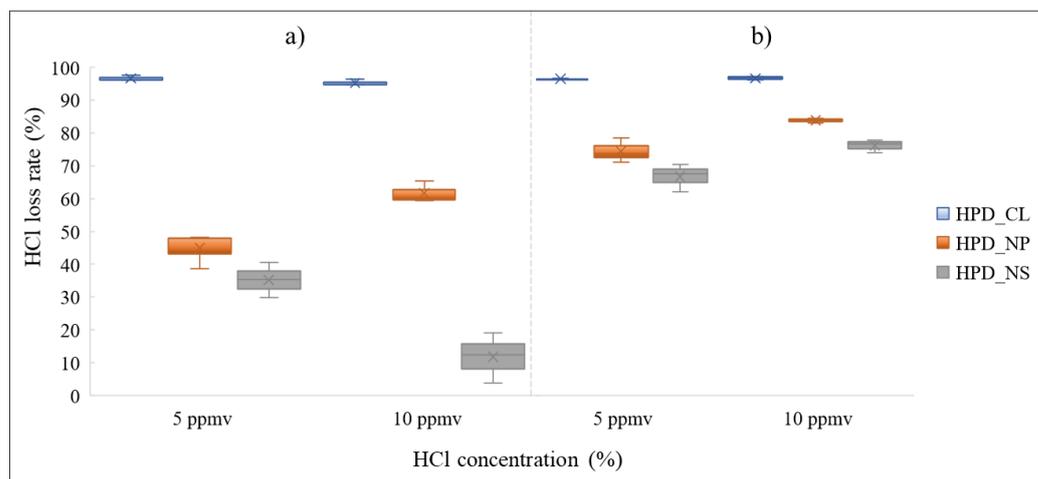
of a conventional cooler. Thus, the loss of HCl should be investigated to determine the advantages of these HPDs.



**Figure 3.** Humidity removal efficiency of humidity pretreatment devices operated at room temperature with respect to low (4%) and high (17%) humidity.

### 3.2. Investigation of the Loss Rates of HCl Caused by Humidity Pretreatment Devices

The performance of HPD\_CL, HPD\_NP, and HPD\_NS was investigated in terms of the loss of HCl target gas. The experiment was also conducted with respect to low and high humidities, as mentioned previously. HCl concentrations were varied as 5 ppmv and 10 ppmv based on the real emissions of a solid waste incinerator. The loss rates of HCl for HPDs in room conditions are shown in Figure 4.



**Figure 4.** Loss rates of HCl caused by humidity pretreatment devices operated in room conditions with respect to various HCl concentrations at a (a) low humidity (4%) and (b) high humidity (17%).

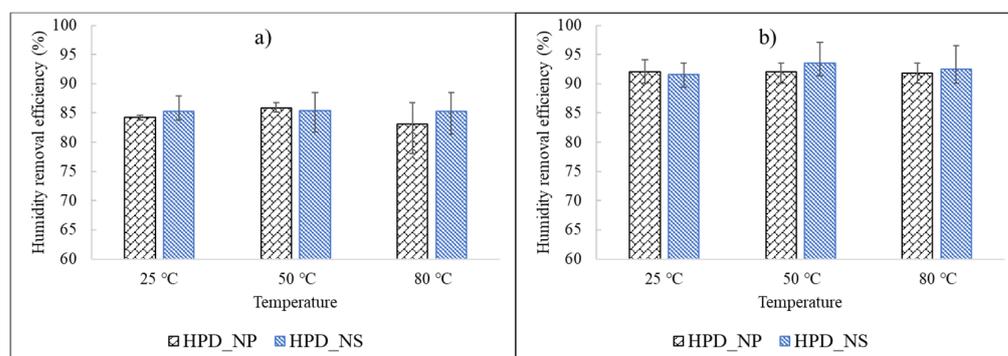
As shown in Figure 4, HPD\_CL revealed the highest loss rates for HCl, followed by HPD\_NP and HPD\_NS ( $p$ -values > 0.05). Higher humidity was associated with a greater loss rate for HCl. Since HPD\_CL removed humidity under the liquid phase, while the water solubility of HCl is 720 g/L at 20 °C [1], HCl was absorbed by water in the HPD\_CL. Although water was continuously removed from the HPD\_CL through a drain pump, water droplets still remained on the inner surface of its impingers, which could influence high water-soluble analytes. This issue was also reported in other studies [24,25]. It was reported that the SO<sub>2</sub> concentration was reduced from 150 parts per billion in volume

(ppbv) to 121 ppbv at 30% relative humidity (RH) and to 57.8 ppbv at 80% RH when a cooler was used to remove humidity in the SO<sub>2</sub> sample gas [24]. When a cooler was applied to remove humidity from the analysis of isobutyl alcohol, the maximum loss of the compound was found to be approximately 40% at 80% RH [25]. For Nafion™ dryers, they caused a significant loss of HCl, especially at a high humidity, although the humidity removal efficiency of these devices was good and the manufacturer reports that HCl can be 100% retained in the sample [26]. Accordingly, the Nafion™ dryers concerned in this study should not be used at a room condition in order to remove humidity at a high level and high temperature; other conditions should be employed instead.

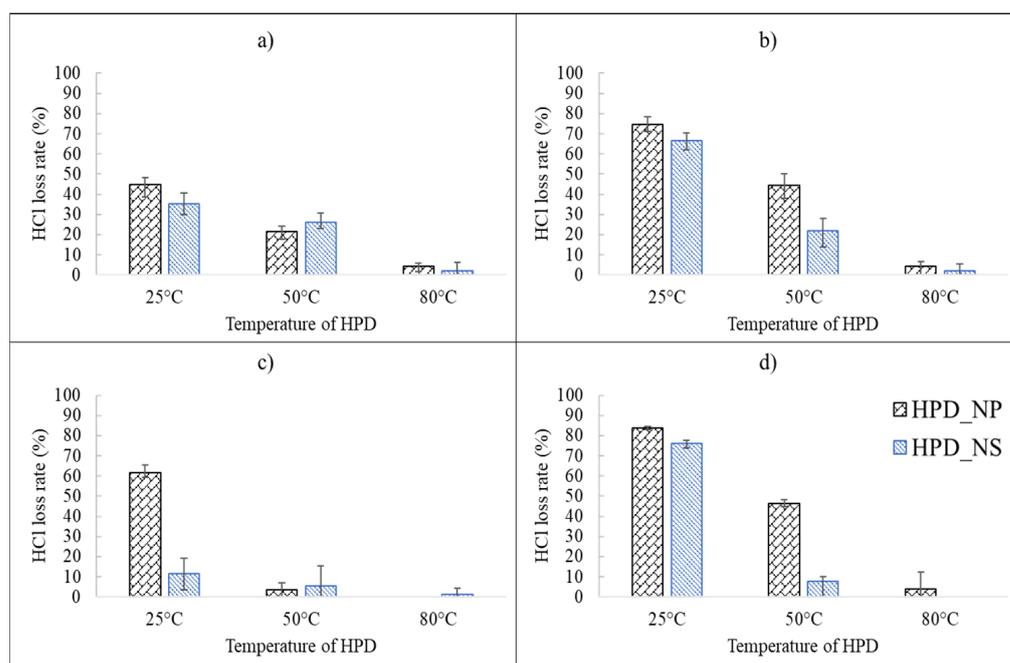
It was reported that HCl gas could be combined with water vapor to generate HCl aerosol depending on the temperature of the system, i.e., the lower the temperature and the higher the humidity, the more HCl aerosol was formed [27]. The relationship between HCl concentration and the formation of aerosol associated with various temperature and humidity is shown in Table 3. As shown in Table 3, at the same temperature, higher RH of the air resulted in lower HCl concentration at which the formation of HCl aerosol is started. It was found that the outlet RH levels of both HPD\_NP and HPD\_NS when they operated at 25 °C were approximately 30–40% at low humidity of inlet and approximately 75–85% at high humidity of inlet. At 80–90% RH (Table 3), the formation of HCl aerosol could be started from only few ppmv at room conditions. Moreover, the target concentrations of HCl in this study were 5 and 10 ppmv. Therefore, this might be the reason for the high HCl loss rates caused by Nafion™ dryers, especially when the inlet humidity was high, although the dryer did not remove water vapor under liquid phase the like the cooler and the humidity removal efficiencies were good. However, this did not seem to be the reason for HCl loss in low humidity inlet samples based on theoretical data, as shown in Table 3. Accordingly, HPD\_NP and HPD\_NS were heated to investigate their performance. Temperature was increased until the loss of HCl was <10%. The humidity removal efficiencies at various temperature are depicted in Figure 5 and the HCl loss rates are presented in Figure 6.

**Table 3.** Starting concentrations of HCl (ppmv) to form aerosol with respect to various air temperature and relative humidity [27].

RH (%)	Air Temperature					
	−10 °C	0 °C	10 °C	20 °C	30 °C	40 °C
30%	>1000	>1000	>1000	>1000	>1000	>1000
50%	200	400	800	1000	>1000	>1000
70%	4.0	10	25	80	200	500
80%	0.0	1.5	2.0	10	25	80
90%	0.0	0.0	0.0	1.5	4.0	10



**Figure 5.** Humidity removal efficiencies of HPD\_NP and HPD\_NS with respect to various operating temperatures and inlet humidity: (a) low humidity (4%); (b) high humidity (17%).

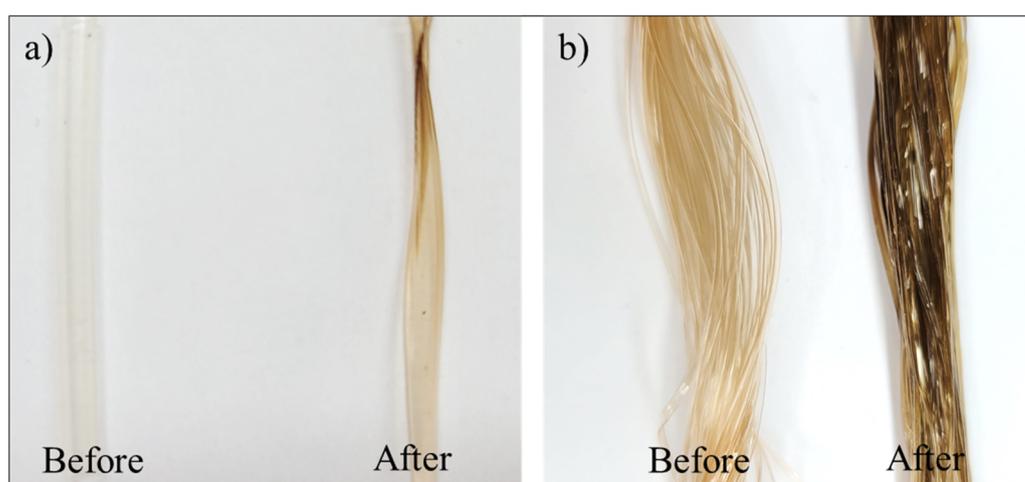


**Figure 6.** HCl loss rates of HPD\_NP and HPD\_NS with respect to various temperatures and other conditions: (a) low humidity (4%) and 5 ppmv HCl, (b) high humidity (17%) and 5 ppmv HCl, (c) low humidity (4%) and 10 ppmv HCl, and (d) high humidity (17%) and 10 ppmv HCl.

As shown in Figure 5, although operating temperatures of HPD\_NP and HPD\_NS were increased from 25 °C to 80 °C, the humidity removal efficiencies of these HPDs were not significantly changed ( $p$ -values > 0.05). This indicates that the humidity removal efficiencies of the Nafion™ dryers in this study were not dependent on dryer temperature.

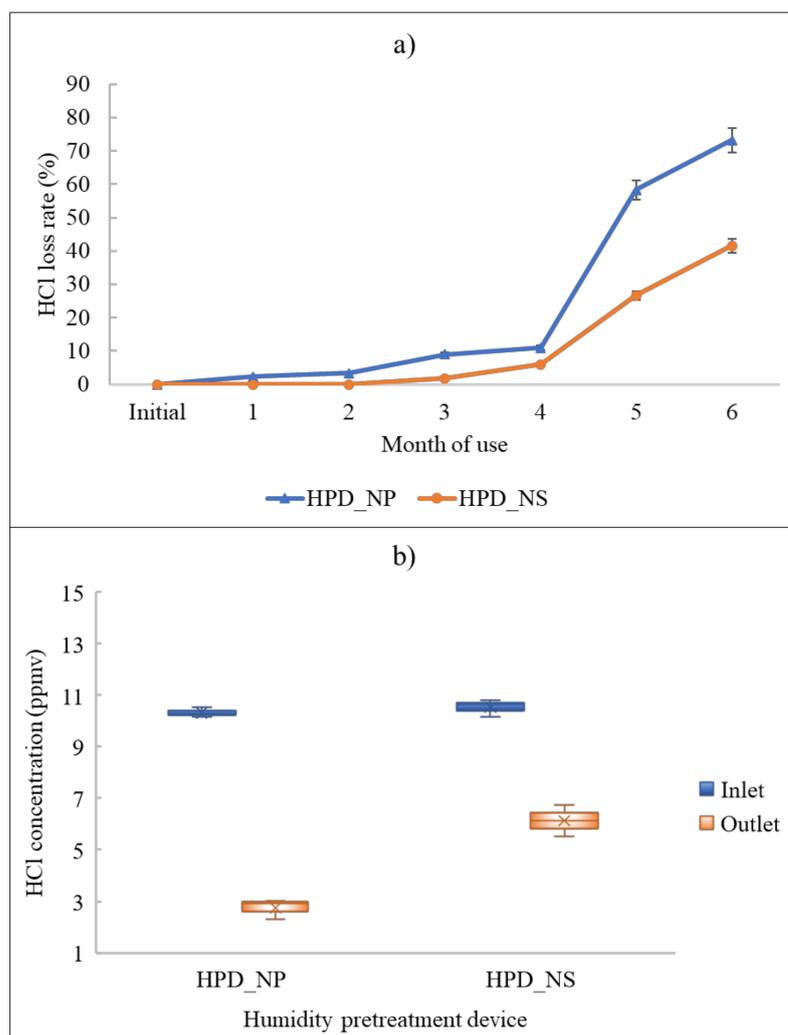
As shown in Figure 6, an increase in the temperature of HPD\_NP and HPD\_NS resulted in a reduction of HCl loss ( $p$ -values < 0.05), although their humidity removal efficiencies were not significantly different, as shown in Figure 5. However, higher inlet humidity brought about a higher HCl loss rate. This indicates that, although the HCl loss rates did not correlate with the humidity removal efficiencies, the inlet humidity was an important effective factor. HPD\_NP showed higher HCl loss rates than HPD\_NS. The gap was higher at higher HCl concentration and temperature. The higher surface area of HPD\_NP (i.e., 47,100 mm<sup>2</sup> of HPD\_NP > 37,680 mm<sup>2</sup> of HPD\_NS) might be the reason why HPD\_NP showed higher loss rates of HCl than HPD\_NS in most cases. In addition, the housing material of HPD\_NP is stainless steel while that of HPD\_NS is Teflon. This might another reason why the HPD\_NP revealed higher HCl loss than the HPD\_NS, because the reaction of HCl solution and HCl gas with stainless steel causing corrosion was reported elsewhere [28,29], and the reaction of HCl gas is accelerated at higher temperature [28]. The lowest HCl loss rates were found at 80 °C for both devices in all conditions. A t-test was implemented to compare the mean HCl concentrations before and after HPD\_NP and HPD\_NS when these devices operated at 80 °C. It was found that the mean HCl concentrations were not significantly different ( $p$ -values > 0.05). The HCl loss rates with respect to HPD\_NS were less than 2%. The HCl loss rates at 5 ppmv HCl were obviously higher than those at 10 ppmv HCl because the loss rate was inversely proportional to the initial concentration based on Equation (1). The reproducibility of HPD\_NP and HPD\_NS was also acceptable because of the RSD < 10%. Consequently, the Nafion™ dryer could operate well in terms of HCl loss at temperatures higher than 25 °C. This result could confirm the assumption of the effect of HCl aerosol formation on the loss of HCl after the dryers because a higher temperature denotes a lower potential of aerosol formation at low HCl concentration (as shown in Table 3). However, it was found that operation at a high temperature for a long time reduced the lifetime of the

Nafion™ membrane. In this study, the lifetime of the membrane was found to be less than six months when it was used at 80 °C, while in a normal condition its lifetime was longer than one year [20,30]. The color of the membrane was changed, as shown in Figure 7. The texture of the membrane also became harder. The performance of the membrane was also worse. As shown in Figure 8, the HCl loss rate of HPD\_NP and HPD\_NS were increased when the use time was longer at 80 °C of operating temperature and 17% of inlet humidity (Figure 8a). The HCl loss rates were significantly high from the fifth month onward. At the sixth month, HCl concentrations at the outlet of HPD\_NP and HPD\_NS were significantly lower than the initial concentrations ( $p$ -values < 0.05) (Figure 8b). The loss rates for HCl with respect to HPD\_NP and HPD\_NS were approximately 73.3% and 41.7%, respectively. Consequently, when the Nafion™ dryers in concern are used at a higher temperature than room temperature, they should be used for less than five months to secure an acceptable HCl loss rate.



**Figure 7.** Color and texture of membranes of (a) NPD\_NS and (b) HPD\_NP before and after being left in the high temperature and humidity environment for around six months.

In general, between the cooler and the Nafion™ dryer, the Nafion™ dryer was found to be more suitable for removing humidity from flue gas and in the measurement of HCl at high temperatures and humidities such as those of the stack gas of a solid waste incinerator. To achieve a good loss rate of HCl, the dryer should be heated (i.e., up to 80 °C in this study) to prevent the formation of HCl aerosol due to the presence of water vapor at a low temperature and low HCl level [27]. However, this operation condition led to the reduction of the membrane's lifetime, which could increase the maintenance costs from exchanging the membrane. This is the disadvantage of the Nafion™ membrane when applied as a humidity pretreatment device for the measurement of HCl at a high temperature and humidity. The Nafion™ membrane was found to cause significant loss of other compounds, at ppbv and ppmv levels and at ambient humidity levels. These compounds included ozone (O<sub>3</sub>), carbon monoxide (CO), methyl ethyl ketone (MEK), methyl isobutyl ketone (MIBK), and isobutyl alcohol [31,32]. The maximum loss of 100 ppbv O<sub>3</sub> was approximately 10%, and that of 25 ppmv CO was approximately 8% [32]. MEK, isobutyl alcohol, and MIBK were found to be lost by more than 80% at 100 ppbv of their concentrations and 80% RH [31]. Therefore, the selectivity of the Nafion™ membrane should be carefully investigated before applying it to an analytical pretreatment device.



**Figure 8.** HCl loss due to contaminated membrane after being left in a high operating temperature ( $80\text{ }^{\circ}\text{C}$ ) and high inlet humidity (17%) environment for around six months: (a) HCl loss rate with respect to elapsed months, (b) inlet and outlet concentrations of HCl after six months of use.

#### 4. Conclusions

The performances of conventional humidity pretreatment devices when measuring the HCl emitted from a stationary source were investigated. The experimental conditions were selected based on the stack conditions of a solid waste incinerator; the flue gas temperature was approximately  $180\text{ }^{\circ}\text{C}$ , the HCl concentration varied at 5 and 10 ppmv, and the humidity varied at 4 and 17% ( $v/v$ ). A cooler and two types of Nafion<sup>TM</sup> dryers were used as the pretreatment devices. It was found that the humidity removal efficiencies of all devices of concern were not significantly different and that all values were over 85%. In terms of HCl loss, the cooler revealed the highest loss rate of HCl because of the HCl absorption of water droplets in the device. Although the HCl loss rates of Nafion<sup>TM</sup> dryers were higher than those of the cooler when they operated at room temperature, there was still significant loss of HCl because of the formation of HCl aerosol at a low temperature. To overcome this issue, the two dryers were heated. They thus performed well at  $80\text{ }^{\circ}\text{C}$  because similar mean HCl concentrations before and after the dryers ( $p$ -values  $> 0.05$ ) were observed. It was found that the HCl loss rates of the two dryers depended on inlet humidity rather than the removal efficiency of the dryers. Consequently, between the cooler and Nafion<sup>TM</sup> dryers, it was determined that a Nafion<sup>TM</sup> dryer could be used as a humidity pretreatment device and for the measurement of HCl emitted from the stack, but its operation temperature must be considered, as the higher the temperature, the lower the HCl loss. However, this operating

condition could shorten the lifetime of the dryer. Therefore, a new humidity pretreatment method for HCl measurement with greater lifetime durability and low investment and operating costs should be proposed in the future. In addition, since this current study was conducted at the ppmv level of HCl, the ppbv level of it should also be investigated in future work.

**Author Contributions:** J.-C.K. and T.-V.D. conceptualized the work; J.-H.L., H.-N.G., I.-Y.K., S.-W.L. and I.-Y.C. performed the experiments; B.-G.P. did data curation, T.-V.D. and H.-N.G. wrote the original draft; J.-C.K. and T.-V.D. performed reviewing and editing. All authors have read and agreed to the published version of the manuscript.

**Funding:** Not applicable.

**Institutional Review Board Statement:** Not applicable.

**Informed Consent Statement:** Not applicable.

**Data Availability Statement:** The data presented in this study are available from the corresponding author upon request. The data are not publicly available due to the confidential policy of our project.

**Acknowledgments:** This paper was supported by Konkuk University Researcher Fund in 2020. This work was also supported by Korea Environment Industry & Technology Institute (KEITI) through R&D Project for Management of Atmosphere environment Program, funded by Korea Ministry of Environment (MOE) (1485018023).

**Conflicts of Interest:** The authors declare no conflict of interest.

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