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TransMembraneChemiSorption of ammonia from sealing water in Hungarian powder metallurgy furnace

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ABSTRACT

Ammonia recovery from industrial wastewater is gaining more and more attention. Due to the environmental impact the regulations for ammonia emissions are becoming stricter, and the need for improved processes is growing. An alternative to conventional technology is TransMembraneChemiSorption, using hydrophobic membranes for controlled mass transfer from liquid through gas-filled pores into liquid absorption fluid. This study presents some information about operating data, basic layout and the restrictions of a full-scale industrial system, as well as an analysis of this new technology compared with common processes. Carrying out plant-scale measurements, a model has been evaluated which makes it possible to establish mass transfer calculations for hollow fiber membrane contactors. The resulting total mass transfer coefficient equals existing ones from literature.

Keywords: Ammonia removal; Chemisorption; Hollow fiber; Membrane contactors; Water treatment

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1. Introduction

An industrial plant owned by a multinational company located in a populated area of Budapest (Hungary) wanted to solve a serious environmental problem: treating wastewater effluent, sealing water, which has a high NH_3 content. Early in the planning phase, three primary goals were established. The first goal was to safely reduce the NH_3 concentration in a process wastewater stream from a maximum of 8,000 mg/L down to 75 mg/L in the final wastewater effluent. Although 100 mg/L is the official NH_3 concentration limit for a wastewater stream discharged in Budapest [1], internal company regulations required at least 25% less than the state-mandated limit.

The second goal was to select a technology that helped reduce the risks associated with environmental impact, safety and public health in accordance with the company's compliance and sustainability policies. The third goal was to make use of an existing structure. The area of Budapest where the installation took place is densely populated, with limited opportunities for growth and expansion. To accomplish this goal, a small footprint was necessary.

A detailed review of literature was conducted to learn about available technologies, processes and their features.

Stripping and burning NH_3 from wastewater is considered an effective method to reduce ammonia concentration, and one that produces little disposable waste. However, because the plant was located in a residential area, burning was not a suitable option due to local environmental

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regulations [2] and the likelihood of resistance from the community. Another disadvantage of stripping and burning is the large footprint. For NH_3 stripping from coke wastewater, Ozyonar et al. [3] reported a volumetric mass transfer coefficient of $K{\cdot}a = 23/h$. The resulting packing was estimated with a diameter of $d_p = 0.7$ m and a height of $h_p = 0.43$ m.

Ion exchange is regarded as a general treatment for charged compounds, but here it is not considered effective due to the high ammonium (NH_4^+) concentration (>400 mg/L) in the wastewater. The most commonly used ion exchangers with natural or synthetic resins have shown capacity limits with a concentration of <45 meq/L NH_4^+ [4]. In the ion-exchange process, a sodium (Na^+) ion is replaced by an NH_4^+ ion, thereby increasing the Na^+ ion concentration in the liquid phase. Once the resin capacity is used up, regeneration is needed, and this is typically accomplished using a flush with an alkaline sodium chloride (NaCl) solution. The resulting ammonium chloride (NH_4Cl) solution is an additional waste stream that must be treated or disposed of [5].

Biological treatment can be applied, but it was not considered here as a suitable option for handling a manufacturing process with frequent stops and starts. Controlling the process in the industrial environment is difficult enough, but coping with variables such as fluctuating inlet NH_3 levels, heavy metal concentrations, and temperature changes that are prevalent in this non-continuous process would make process control exceedingly difficult [6]. Moreover, according to Anthonisen [7], high pH and high NH_3 concentrations inhibit both ammonia and nitrite oxidation. Results from Abeling [8] suggest that ammonia oxidation processes are inhibited at concentrations above 7 mg/L NH_3 and are blocked at concentrations above 20 mg/L NH_3. Nitrite oxidation processes are also inhibited at concentrations over 24 mg/L NH_3 [9].

The reverse osmosis treatment of NH_4^+ solution in such a high concentration (>8,000 mg/L) would require more than 40 bar of feed pressure resulting in a 2 $kW/h/m^3$ energy consumption to reach a concentration of 120 g/L ammonium sulfate [10]. Another disadvantage of this technology is that any heavy metal content in the raw water could be concentrated with NH_4^+, thereby creating a waste stream that would be more toxic.

A membrane contactor is another membrane separation technique. Membrane contactors are commonly designed with hydrophobic, microporous hollow fiber membranes [11] allowing free gas to transfer from an aqueous phase to a liquid receiving phase through air-filled pores. When a chemical reaction also takes place simultaneously by applying a suitable receiving phase, we call the process TransMembraneChemiSorption (TMCS), where mass transfer can be closely controlled. Its operating principle has been published in a couple of studies [12–16].

Comparing the available techniques, TMCS seemed the most promising so it was selected for the study. Sulfuric acid is a proper receiving phase, thus TMCS enables the direct transport of gaseous NH_3 from wastewater to a sulfuric acid solution through a hydrophobic, microporous hollow fiber membrane in a single compact device. As a by-product of this membrane-based process, an ammonium sulfate ($(NH_4)_2SO_4$) solution with a concentration close to the solubility level of 30%–40% is generated. In the wastewater, >95% of incoming NH_3 can potentially be removed depending on operating conditions. Heavy metal transport from the wastewater side (aqueous phase) to the by-product side (liquid receiving phase) is lower compared with other methods because the air-filled pores of the hollow fiber membrane act as a barrier to dissolved, non-volatile components in the water.

Although the process has been described in literature, it has not been used in large-scale applications where the initial ammonia content is so high (8,000 mg/L). Therefore, the main aim of our project was to confirm that the TMCS process can be applied to successfully treat wastewater effluent with an extremely high ammonia content, and to provide a detailed mass transfer analysis for a real industrial process.

2. Theory

2.1. Process description

According to Henry's law, the molar fraction of a gas in liquid x_i (mol/mol) is proportional to the partial pressure of gas p_i (Pa) above the liquid. The solubility (Henry) coefficient K_H (Pa mol/mol) is different for each gas and influenced by temperature and pressure. By chemisorption of the gas in liquid, its concentration becomes zero and its solubility infinite. In the TMCS process, a partial pressure difference of gas on both sides of a membrane is generated, creating the driving force for continuous gas transfer. To improve membrane contactor efficiency, wastewater flows countercurrent to the sulfuric acid (H_2SO_4) solution inside the module. Gas transport takes place through air-filled pores of the wall of the hydrophobic hollow fiber membrane as shown in Fig. 1.

In the process, wastewater is circulated on the outside (shell side) of the hollow fiber at specified temperature and pH ranges and at a specified flow rate, while H_2SO_4 is circulated on the inside (lumen side or receiving phase) at a specified pH range and flow rate. The wastewater pH is increased using 50% sodium hydroxide (NaOH) to exceed pH 11, which causes the NH_3 to shift to a free gas state, while 98% H_2SO_4 is used to keep the pH of the receiving phase always under pH 2 in order to maintain sufficient absorption capacity. The NH_3 chemically reacts with the H_2SO_4 in a two-stage reaction through ammonium hydrogen sulfate $(NH_4)HSO_4$ into ammonium sulfate $(NH_4)_2SO_4$, depending on the pH of the solution. This process is expressed and simplified in Eq. (1):

$$2NH_3 + H_2SO_4 \rightarrow (NH_4)_2SO_4 \quad (1)$$

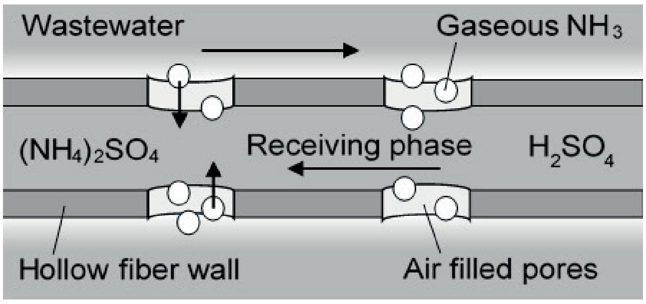

Fig. 1. TMCS process for NH_3 removal from industrial wastewater using a hydrophobic, microporous hollow fiber membrane.

At pH > 4 the resulting $(NH_4)_2SO_4$ solution is a fertilizer which can possibly be sold or disposed of free-of-charge from the factory.

The concentration of the $(NH_4)_2SO_4$ by-product was set during the process. The higher the product concentration, the lower the volume of disposable liquid created. The final pH of the product was adjusted by dosing with potassium hydroxide (KOH). If the target use for the by-product is agricultural purposes, the use of KOH instead of NaOH may be beneficial to avoid sodium contamination. System control is fully automated, so operating activity is normally limited to handling $(NH_4)_2SO_4$ and H_2SO_4 containers and for periodic observation and maintenance checks.

2.2. Mass transfer considerations

The mass concentration C of ammonia in water is influenced by the ionization equilibrium between ammonia (NH_3) and ammonium (NH_4^+), and can be expressed by Eq. (2):

$$\mathrm{NH_3 + H_2O \leftrightarrow NH_4^+ + OH^-} \tag{2}$$

The NH_3 fraction (dimensionless) depends on the pH and temperature T (K) of the solution and is readily derived from Eq. (3) [4]:

$$\alpha_{\mathrm{NH_3}} = \frac{C_{\mathrm{NH_3}}}{C_{\mathrm{NH_3}} + C_{\mathrm{NH_4^+}}} = \frac{10^{\mathrm{pH}}}{e^{6244/T} + 10^{\mathrm{pH}}} \tag{3}$$

If the pH value exceeds 11 and the treatment temperature is 20°C –50°C, $\alpha_{\mathrm{NH_3}}$ is approximately 1.

2.3. Simple diffusion approximation

Taking this into consideration the mass flux J_t of transferred species is derived from film theory and can be expressed by Eq. (4) [2,3,17]:

$$J_t = K \cdot (C_t - C_{S,t}) \cong K \cdot C_t \tag{4}$$

The rate constant K is the overall mass transfer coefficient related to the liquid (feed) side. The driving force for the process is the difference in NH_3 concentration C_t across the membrane, while the NH_3 concentration in equilibrium with stripping solution $C_{S,t}$ is essentially zero for excess acid (low pH) conditions. The $\alpha_{\mathrm{NH_3}}$ value was included to show that only free NH_3 is transferred across the air-filled pores.

In the case of a simple diffusion approximation, a constant ammonia concentration is assumed along the length of membrane (C_t average concentration). Then, using the mass balance, the flux can be written as follows:

$$-J_t = \frac{V}{A_T} \cdot \frac{dC_t}{dt} \tag{5}$$

where V is the liquid (wastewater) volume, A_T is the mass transfer area of a membrane, which correlates with installed membrane area A_M [18]. Comparing Eq. (4) with Eq. (5) for the wastewater, a differential equation can be given whose solution will be straight with a slope $m = \frac{A_T}{V} \cdot K$ [2]:

$$\ln \frac{C_0}{C_t} = \frac{A_T}{V} \cdot K \cdot t \tag{6}$$

where C_0 is the initial NH_3 concentration.

In the membrane contactor, C_t is constant along the length of the membrane, if Q_F (m/min) $\rightarrow \infty$. With a finite flow rate feed a concentration difference has formed between two ends of the membrane. In this case, the mass transfer is extended into the next form [2]:

$$K_{\exp} = -\frac{Q_F}{A_T} \cdot \ln\left(1 - \frac{V}{Q_F \cdot t} \cdot \ln\left(\frac{C_0}{C_t}\right)\right) \tag{7}$$

where the volume of liquid (wastewater) now is about equal to the volume of the feed reservoir.

Finally, for the concentration ratio of the NH_3 as a function of time, using transformation (7) gives the following formula:

$$\ln \frac{c_0}{c_t} = \frac{Q_F}{V} \cdot \left[1 - \exp\left(-\frac{A_T}{Q_F} K_{\exp}\right)\right] \cdot t \tag{8}$$

This will be a straight line with a $m_Q = \frac{Q_F}{V} \cdot [1 - \exp(-\frac{A_T}{Q_F} \cdot K_{\exp})]$ slope, depending on the flow rate feed, Q_F, if $Q_F \rightarrow \infty$ then $m_Q \rightarrow m$.

3. Materials and methods

Two series 10 × 28 inch Liqui-Cel™ Membrane Contactors were integrated by Hidrofilt Ltd. (Magyar str. 191 Hungary) into a complete industrial wastewater treatment plant. An average cartridge has an outer/inner diameter of $D_{\mathrm{CO}} = 0.245$ m, $D_{\mathrm{CI}} = 0.114$ m offers space for roughly 224,000 fibers with an outer diameter of roughly $D_{\mathrm{FO}} = 300 \times 10^6$ m and an effective fiber length of $L_f = 0.61$ m [17]. The properties of the membrane used are summarized in Table 1.

The TMCS process to remove NH_3 was added between the furnace's overflow sump and the neutralization step in an environment with limited space. The full size of the TMCS system including storage tanks, by-product tanks, and an electrical cabinet with a control panel is 1,000 mm × 14,000 mm × 4,000 mm. Fig. 2 shows the membrane contactors with the operation panel.

Table 1
The properties of the membrane

Material	Polypropylene
Configuration	Microporous hollow fibers
Wall thickness (nominal)	40 μm
Pore dimensions	0.04 × 0.10 μm
Porosity (nominal)	40%
Tortuosity	2.25

The process design consists of two circulating loops: the wastewater loop (feed side) L1 and the by-product loop (stripping side) L2. The two loops are separated by the hollow fiber membrane inside the membrane contactors. The two modules MC1 and MC2 are connected in series for the wastewater stream and in parallel for the by-product stream. Circulation on both sides is ensured by centrifugal pumps P1 and P2. The membrane dosing pumps P3 and P4 adjust the pH on both sides, according to simplified process and instrumentation drawing (P&ID) in Fig. 3.

At start-up the wastewater tank (feed reservoir) T1 holds a total volume of roughly 1 m^3, while the acid tank (stripping reservoir) T2 is initially filled with roughly 0.12 m^3. The filling level shifts due to water vapor transport from the water loop to the acid loop because of the difference in water vapor pressures between the two liquid phases. The temperature is controlled by an electrical heating element. After the NH_3 is transferred, the cleaned wastewater is released. The product is circulated until a specified $(NH_4)_2SO_4$ concentration or the maximum filling level in the acid tank is reached.

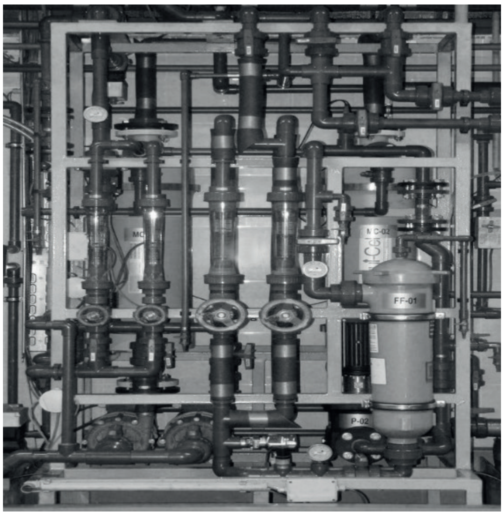

Fig. 2. Liqui-Cel™ Membrane Contactors with operation panel at Budapest's TMCS plant.

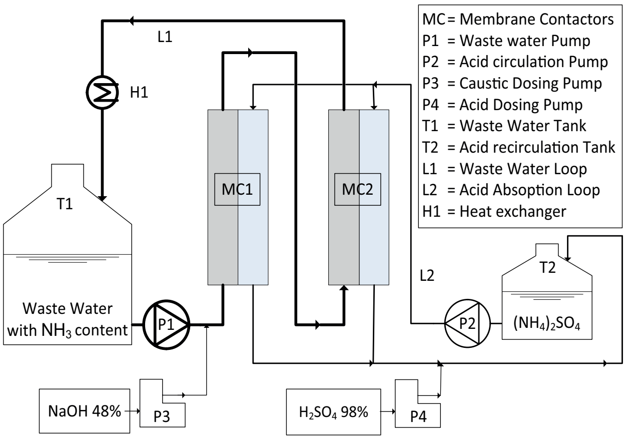

Fig. 3. Simplified P&ID of TMCS system.

4. Results and discussion

Experiments were carried out to study the progress of the process under the conditions given above. The experimental data were based on industrial wastewater with parameters before and after treatment listed in Table 2. The effluent from the furnace liquid seal is first collected in a storage tank to cool down to 40°C. The wastewater is pumped directly from this tank to the TMCS process feed side T1 process tank.

The data in the results section were generated for $V = 1$ m^3 and $Q_F = 5$ m^3/h. Two series 10 × 28 inch Liqui-Cel™ Membrane Contactors with an approximate membrane area of $A_T = 260$ m^2 were used (each contactor has $A_M = 130$ m^2) in a membrane package volume of $V_P = 0.045$ m^3.

Wastewater of a typical (average) composition was selected for the specific measurements, where the ammonia concentration of wastewater was 5,950–6,100 mg/L (average initial concentration $C_0 = 6{,}000$ mg/L), the treatment temperatures were low (24°C–27°C) medium (30°C–35°C) and high (38°C–42°C), averaging at 25°C, 32°C and 40°C, and the pH was set between 11 and 11.5. Three parallel samples were measured at 0, 15, 30, 45, 60, 90 and 120 min treatment times in the feed reservoir, and the average was calculated. The temperature was controlled by cooling the reservoir. Finally, the concentration logarithmic ratios were constituted and plotted against treatment times (Fig. 4).

4.1. Mathematical method

The values of m_Q in Eq. (8) were calculated by linear regression (EXCEL). The correlation coefficient square, R^2 is determined [19], furthermore the Student's t-test is used which is $t_f = \sqrt{fR^2/(1-R^2)}$ [20], where f is the variability, $f = n - p - 1$, n ($n = 6$) is the number of measured points (times) and p ($p = 1$) is the number of parameters to be calculated and the significance levels, P belonging to t_f was extracted from the Student's t-test table. The 0-hypothesis is that the results can be written by Eq. (8) at a 95% probability level ($1 - P = 0.95$), if $P \leq 0.05$. The slopes, m_Q and (experimental) mass transfer coefficient K_{exp} calculated from those are summarized in Table 3.

According to Eq. (8) straights can be fitted to the measured points ($1 - P = 0.999 > 0.95$), from whose slopes experimental mass transfer coefficients $K_{exp} = 0.011$–0.017 m/h were

Table 2
Process data of TMCS system in Budapest

	Wastewater	Treated water[b]	Product $(NH_4)_2SO_4$
Wastewater production[a] (m^3/d)	2.50	2.25	0.65
NH_3 concentration (mg/L)	3,300–8,600	60–300	75,000
Temperature (°C)	25–40	30–40	35–40
pH	10–11.3	6.5–9.0	5.0–8.0

[a] Wastewater is preliminary reverse osmosis desalinated urban water, which downstream of the metallurgical furnace gets contaminated with NH_3 and heavy metals.

[b] Following 2 h of treatment.

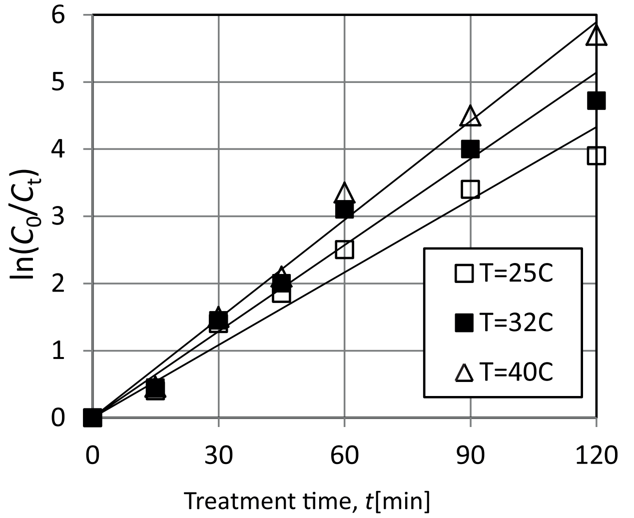

Fig. 4. Correlation between logarithmic ratio of NH_3 concentration and treatment time, t.

Table 3
The slopes, m_Q and (experimental) mass transfer coefficient K_{exp} calculated from Fig. 4 with regression characteristics

Temperature (°C)	m_Q (1/h)	K_{exp} (m/h)	R^2	t_f	P
25	2.18	0.011	0.9750	12.5	0.001
32	2.56	0.014	0.9712	11.6	0.001
40	2.97	0.017	0.9846	16.0	0.0005

calculated. These values are higher, but still in the range of numbers calculated by Semmens et al. [3], for NH_3 removal on a laboratory scale with acid on the outside and NH_3–water on the inside of the hollow fibers. Higher K_{exp} values are to be expected in this case because NH_3–water flows on the outside of fibers in a transverse direction [11].

Comparing a desorption column using the Onda [21] prediction model, a feed side mass transfer coefficient of K_F = 0.5 m/h was calculated with Eq. (A1) and is larger compared with the K_{exp} value above of a membrane system. However, the membrane contactors offer a larger specific area a_T = 5,769 m²/m³ (A_T/V_P) compared with Pall ring packing, e.g., with a calculated specific surface area of a_W = 91 m²/m³ with Eq. (2). Therefore, the volumetric transfer coefficient ($K·a$) of TMCS $K_{exp}·a_T$ = 84/h is equal to or larger than the calculated stripper $K_F·a_W$ = 53/h (or $K·a$ = 23/h from [21]). It must also be considered that the TMCS combines a desorption step with a chemical absorption step in a single column. The reduction of the required space and energy due to the missing air circulation creates an additional advantage for membrane operation.

The experimental mass transfer coefficient, K_{exp}, depends on the temperature; it increases if the temperature goes up. The mass transfer process is based on diffusion processes of thermoactivation, whose rate increases with temperature and according to the rate of mass transfer process increases too.

The ammonia content of the industrial samples dropped to 60–300 mg/L after 2 h of treatment, while the same value for the experimental samples was 20–115 mg/L. The reason for the difference is primarily the lower pH (<11) of the industrial samples than desired. Based on the model, the calculated value for the industrial samples is 10–260 mg/L, which drops to 10–165 mg/L with a pH of >11, and to 10–70 mg/L if T > 30°C–35°C.

The required treatment time (C_t < 75 mg/L) depends on the initial concentration (C_0) and the treatment temperature (T). Both can be measured online, so the treatment time can be optimized by using the model. Since the values of the two parameters can sometimes change from sample to sample, and the treatment time (K material transfer coefficient) can depend on other features to some extent, this can be considered a probability variable. We elaborated a model for a similar case in another work [22], which can also be used here to determine the minimum treatment time necessary to ensure the given safety.

Further performance trials with this system under different conditions were completed, making it possible to estimate the time demand and expected NH_3 outlet value of treated water under different initial NH_3 inlet concentrations. However, data and results of the additional trials were not shared at the time the paper was written. In this paper, the main focus is on ammonia measuring and observing membrane contactor technology in the TMCS process in a working production environment. A general study on factors that can influence the process, like pH, temperature, etc., can be tested further in a lab-scale environment.

5. Conclusion

The main scope of the project was to reduce NH_3 concentrations below regulatory limits. It was also important to limit any discharge that could negatively impact the environment by creating a sustainable process that produced a manageable by-product that was easy to dispose of, or was fit for secondary use (reuse). It was also important for the selected technology to be flexible enough to handle frequent starts and stops, and there was a preference for an installation where there was limited space.

These goals were achieved by using membrane contactors. The 2-h treatment at pH > 11 and T > 30°C–35°C reduced ammonia concentrations significantly by 98%–96%, which proved to be suitable to achieve the final goal of a 75 mg/L outlet ammonia concentration in the final effluent. It has been proved that a temperature increase exerts a significant effect on treatment time. Based on the model it can be stated that an increase in temperature from 25°C to 50°C can reduce the treatment time from 2 to 1 h. Above 50°C it is not possible to employ the membrane technology due to the heat tolerance of the membrane elements. Owing to the exponential nature of the model it can also be stated that a further reduction of the final effluent concentration from 75 to 30 mg/L should increase the treatment time by at least 25% in the case of a linear approximation, and would double the time demand in the case of non-linear approximation.

The TMCS process has been proven on an industrial scale as a viable alternative to conventional technologies under the operating environment explored in this paper. The results create a basis for future wastewater recirculation projects.

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Symbols

A_M — Membrane area, m^2
A_T — Transfer area, m^2
C — Inlet gas concentration, kg/m^3 and mg/L
C_0 — Initial gas concentration, kg/m^3 and mg/L
C_t — Gas concentration after some time, kg/m^3 and mg/L
$C_{S,t}$ — Stripping gas concentration, kg/m^3 and mg/L
D_{CI} — Average cartridge inner diameter, m
D_{CO} — Average cartridge outer diameter, m
D_{FO} — Outer diameter of fibers, 10^{-6} m
D_P — Diameter of packing, m
Fr — Froude number
H_P — Height of packing, m
J_t — Mass flux after some time, kg/m^2 h
K — (Overall) mass transfer coefficient, m/h and m/min
$K{\cdot}a$ — Volumetric transfer coefficient, 1/h
K_{exp} — Experimental mass transfer coefficient, m/h
K_F — Feed side mass transfer coefficient, m/h
K_H — Henry coefficient, Pa mol/mol
L_f — Effective fiber length, m
Q_F — Flow rate feed, m^3/h
R — Correlation coefficient
Re — Reynolds number
Sc — Schmidt number
T — Treatment temperature, K and °C
V_P — Volume of packing, m^3
V — Volume liquid/reservoir, m^3
We — Weber number
a_T — Total surface area of packing, m^2/m^3
a_W — Wetted surface area of packing, m^2/m^3
d_P — Nominal size of packing material, m
f — Variability
g — Gravitational constant, m/h^2
n — Number of measured points
p — Number of calculated parameters
p_i — Partial pressure, Pa
t — Time, min
t_f — Random variable of Student test
x_{i} — Molar fraction of a gas in liquid, mol/mol
α_{NH_3} — NH$_3$ fraction
μ — Viscosity of liquid, kg/m/h
ϱ — Density of liquid, g/cm^3
σ — Surface tension, kg/h^2
σ_c — Critical surface tension, kg/h^2

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Appendix

Comparison data for NH_3 stripper

A commonly used prediction model for designing a packed column was published by Onda et al. [21]. The feed side mass transfer coefficient K_F of a randomly packed desorption column is described by Eq. (A1):

$$K_F \cdot \left(\frac{\rho}{\mu g}\right)^{1/3} = 0.0051 \cdot (\mathrm{Re})^{2/3} \cdot (\mathrm{Sc})^{-1/2} \cdot \left(a_T \cdot d_P\right)^{2/5} \quad \text{(A1)}$$

The wetted surface area (a_w) for calculating Reynolds number in Eq. (A1) is given by Eq. (A2):

$$\frac{a_W}{a_T} = 1 - \exp\left\{-1.45\left(\frac{\sigma_c}{\sigma}\right)^{0.75} \cdot (\mathrm{Re})^{0.1} \cdot (\mathrm{Fr})^{-0.05} \cdot (\mathrm{We})^{0.2}\right\} \quad \text{(A2)}$$

For the comparison study a packing of Pall rings (PVC) with a nominal size of D_P = 25 mm, a specific surface area of a_T = 220 m^2/m^3 under a liquid velocity of u = 13 $m^3/m^2/h$ was used. From a volume calculation of a cylindrical packing shape, the packing diameter (D_P) can be calculated by Eq. (A3):

$$D_P = \sqrt{\frac{4}{\pi} \cdot \frac{Q_F}{u}} \quad \text{(A3)}$$

while the packing height (H_P) is calculated by Eq. (A4), transposed Eq. (7):

$$H_p = \frac{Q_F}{A_P \cdot K_F \cdot a_w} \cdot \ln\left(1 - \frac{V}{Q_F \cdot t} \cdot \ln\left(\frac{c_0}{c_t}\right)\right) \quad \text{(A4)}$$