

DARE2X

Decentralised Ammonia production from Renewable Energy utilising novel sorption-enhanced plasma-catalytic Power-to-X technology

D5.1 - Report on integration of catalyst and sorption materials in lab-scale prototype

Lead beneficiary: DTI

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Abstract: This report summarizes the integration of catalyst materials and sorption materials into the plasma reactor. A single-stage configuration is tested to simultaneously produce and separate NH_3 , where sorption and catalyst materials particles are extruded into particles with a defined size-range to form a packed bed in the plasma-catalytic zone. This report also summarizes the integration of the plasma-catalytic process with adsorption. A two-stage configuration is tested where the sorption separation process occurs predominantly in a separate unit downstream from the plasma-catalytic zone.

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² PU: Public, fully open; SEN: Sensitive.

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ABBREVIATIONS AND ACRONYMS

NiOCat Refers to catalyst powder
developed in WP2

ZeoSorp Refers to sorption material
from deliverable 3.2



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1. Introduction

The DARE2X project is investigating a power-to-x technology for decentralized ammonia based on sorption-enhanced, non-thermal, plasma catalysis. Plasma catalysis has a few unrealized advantages over traditional thermal catalysis. Plasma catalysis functions at ambient conditions (<100 °C) with negligible start/stop times compared to a thermal catalysis that has to heat up the entire mass of a reactor. However, the stability of NH₃ in plasma is very poor, where throughputs can be enhanced if the NH₃ can be protected by being adsorbed to a material (sorption-enhanced).¹ The negligible start/stop times (minutes) of plasma catalysis allows for practical NH₃ adsorption/desorption. In addition, this NH₃ adsorption/desorption can provide efficient separation/up-concentration.

The catalyst materials (from WP2 and WP4) and sorption materials (from WP3) will be integrated into the pilot scale set-up by extruding these powders into particles with a defined size-range to form a packed bed in the plasma-catalytic zone and/or the sorption unit. The pilot scale set-up will test two configurations: single-stage and two-stage (Task 5.1 and Task 5.2, respectively). The single-stage configuration aims to maximize the sorption-enhancement benefits, where both the NH₃ synthesis and NH₃ sorption occur in the plasma-catalytic zone (T5.1). The two-stage configuration aims to optimize the NH₃ separation/up-concentration, where the NH₃ sorption occurs predominantly in a separate sorption unit downstream from the plasma-catalytic zone (T5.2).

2. Pilot scale set-up

The pilot scale set-up is summarized in Figure 1. The plasma generator (A) was supplied by AFS Plasma corona perforators (G05F generator). Brooks Mass Flow Controllers (B) were used to control the flow of gases (GF040CXXC-1036002L-T2PNS4-XXXXAX-00X, GF040CXXC-0136020L-T2PBS4-XXXXAX-00C). The plasma reactor was built in-house (C), with the capacity for 6 quartz tubes supplied by Mirit Glas (diameter: 8 mm, wall size: 1.5 mm, length: 200 mm). The faradaic cage (D) was built in-house (aluminium). The stainless steel tubing and valves (EFHIK) were supplied by Swagelok (Part turn actuator EN ISO 5211). The vacuum pump (G) was supplied KNF (N922STE EX). The sorption unit was supplied by Hulteberg Chemistry and Engineering from WP3 and D3.2 (J) with a heating coil added to it. The sample gas to the analysis unit (L) is quantified by an FTIR spectrometer (Thermo Fisher). The sample gas from the analysis unit (M) is diluted with air (B) before being introduced to a catalytic burner, which burns excess H₂ and NH₃ to avoid releasing them in the exhaust.



Figure 1: The pilot scale reactor set-up with its components: plasma generator (A), mass flow controllers for gases N_2 H_2 and air (B), plasma reactor (C), faradaic cage (D), valve from plasma reactor to analysis unit (E), valve from plasma reactor to vacuum pump (F), vacuum pump (G), valve from vacuum pump to sorption unit (H), valve from pump to analysis unit (I), sorption unit (J), valve from sorption unit to analysis unit (K), sample gas to analysis unit (L), sample gas from analysis unit to exhaust (M).

2.1 Plasma Reactor

The plasma reactor has a reactor body comprised of PEEK (polyether ether ketone) outer electrodes (aluminum) were designed and manufactured in-house (Figure 2 left). It hold 6 quartz tubes that can be loaded with catalyst (Figure 2, right). The quartz tubes (diameter: 8 mm, wall size: 1.5 mm, length: 200 mm) for the plasma reactor have an inner electrode (steel rod, diameter: 3 mm, length: 240 mm) and end pieces comprised of PEEK (polyether ether ketone). Each quartz tube is loaded with glass wool at the ~ 90 mm mark, to support the extrudate material (particle size: 100–315 μm) until the ~ 130 mm mark resulting in a mass loading around 280-330 mg per tube (Figure 2, right).

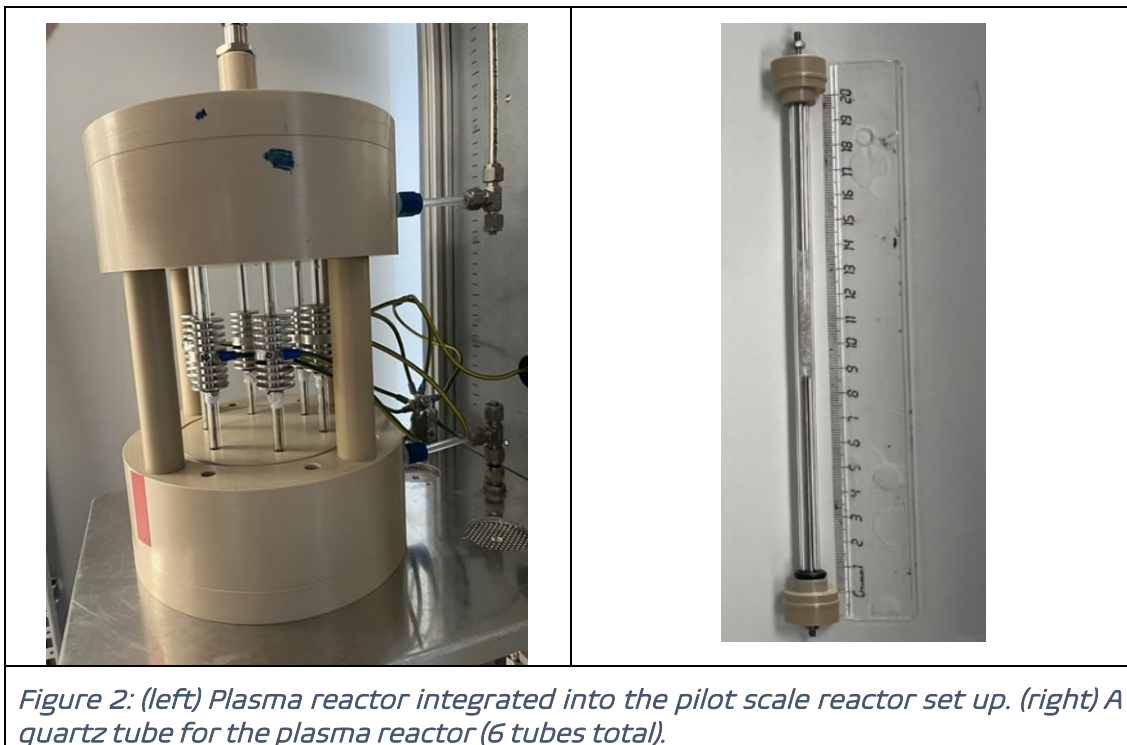


Figure 2: (left) Plasma reactor integrated into the pilot scale reactor set up. (right) A quartz tube for the plasma reactor (6 tubes total).

2.2 Materials

The sorption unit (Figure 1, J) from D3.2 has an internal volume of ~ 250 ml. It was loaded with the sorption material directly from the vacuum sealed bag zeolite based extrudates as described in deliverable 3.2. This sorption material will be referred to as ZeoSorp. The dead space around the sorption material was filled with an inert material on both ends ($\alpha\text{-Al}_2\text{O}_3$, 90 g, 100 ml). The materials were then degassed at 110 °C and 450 torr (0.60 bar) in the sorption unit overnight.

The catalyst used in the plasma reactor was nickel-based and supported on zeolites (different from sorption zeolite) developed in WP2. The synthesised catalyst powder had a Ni loading of 3 ± 1 (wt.%).

The catalyst powder was extruded using a boehmite binder based on methods found in literature.³ Two types of extrudates were made: 50:50 (NiOCat: Al_2O_3) and 25:25:50 (ZeoSorp:NiOCat: Al_2O_3). They were prepared by dry mixing 4 g zeolite powder (0:4 g or 2:2 g ZeoSorp:NiOCat), 160 mg (hydroxypropyl)methyl cellulose (sigma aldrich), and 4.8 g Disperal P2 (pseudo-boehmite from Sasol). Once the mixture was thoroughly homogenized, deionized water (10-11 ml) was added dropwise while continuously mixing to obtain a dough ready for extrusion. The dough was then transferred to a hand extruder (DAS Smart metal clay extruder) for shaping through a 1.6 mm diameter die. The resulting green extrudates were transferred to a muffle oven where they were dried at 323 K for 2 h, then 363 K for 12h, and finally calcined at

823 K for 4 h at with a heating rate of 5 K/min. The calcined extrudates were then gently crushed, sieved and the 100–315 μm fraction was retained.

3. Task 5.1 Integration of sorption and catalyst materials (single stage).

Extrudates are powder samples that have been bound together to form larger particles. This is necessary in order to avoid pressure drops when flowing gases through the reactor bed. The catalyst is mixed with a pseudoboehmite binder ($\text{Al}_x\text{O}_x\text{H}_x$) and calcined to form an extrudate 50:50 (NiOCat: Al_2O_3). In addition, another extrudate is prepared with the inclusion of a sorption material to demonstrate the sorption-enhancement in the plasma-catalytic zone. This results in the extrudate 25:25:50 (ZeoSorp:NiOCat: Al_2O_3).

The single-stage configuration aims to maximize the sorption-enhancement benefits, where both the NH_3 synthesis and NH_3 sorption occur in the plasma-catalytic zone (T5.1). This will be tested by testing two different extrudate samples in the plasma reactor: 50:50 (NiOCat: Al_2O_3) where sorption-enhancement should not occur and 25:25:50 (ZeoSorp:NiOCat: Al_2O_3) where the sorption zeolite should provide sorption enhancement. EDX analysis (Table 1) validates the elemental composition of these two extrudate samples, confirming 50:50 (NiOCat: Al_2O_3) has twice the nickel loading of 25:25:50 (ZeoSorp:NiOCat: Al_2O_3) and confirming the presence of zeolite's Na and K content in 25:25:50 (ZeoSorp:NiOCat: Al_2O_3).

Table 1: EDX analysis of the extrudate samples.

Spectrum Label	O	Na	Al	Si	K	Ni
25:25:50 (ZeoSorp:NiOCat: Al_2O_3)	50,0 \pm 0,2	2,3 \pm 0,1	21,3 \pm 0,3	24,9 \pm 0,2	0,1 \pm 0,0	1,5 \pm 0,1
50:50 (NiOCat: Al_2O_3).	51,1 \pm 0,5	-----	22,5 \pm 1,2	23,8 \pm 0,6	-----	2,6 \pm 0,2

The extrudate samples were tested in 4 experiments each. In each experiment, the plasma was left on for 20 minutes at steady state, and then turned off to monitor the desorption of NH_3 . For 2 experiments, the plasma reaction occurred while keeping the pressure at 0 bar(g), while for the other 2 the pressure was kept at 2.5 bar(g), to assess the influence of the pressure in the reactor. Finally, for each pressure the desorption was investigated in 2 modes. One experiment was performed while keeping flows and pressure constant when turning off the plasma (steady-state), and the other one was performed while switching off the H_2 flow and pumping vacuum in the reactor zone (dynamic operation).

All experiments used a reactor flow of 360 $\text{ml}_{\text{N}_2}/\text{min}$ and 180 $\text{ml}_{\text{H}_2}/\text{min}$. All experiments applied a plasma power of 200 W at 0 minutes which equated to ~ 6 kV high voltage and ~ 200 V DC-voltage at 0 bar, and to ~ 7.5 kV high voltage and ~ 175 V DC-voltage at 2.5 bar. The resulting data (NH_3 concentration over time) was integrated and converted into NH_3 mass ($\text{ppm}_{\text{NH}_3} \times \text{flow} \times \text{time}$).

3.1 Empty tube

Testing these experiments in the plasma reactor without any catalyst or sorption material provides a ‘background’ to estimate what the system is roughly capable of in terms of NH_3 synthesis and sorption. The experiments are summarized in Figure 3.

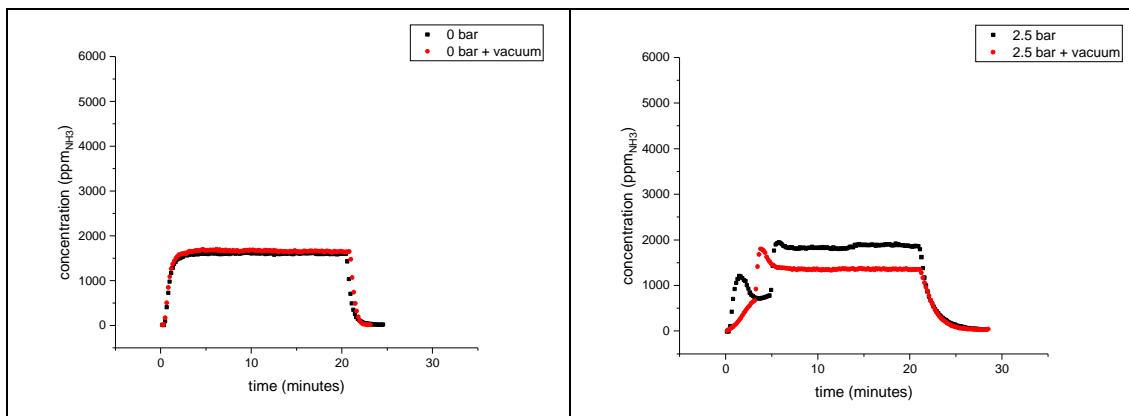


Figure 3: Plasma reactor experiments using empty tubes with 0 bar (left, black), 0 bar + vacuum desorption (left, red), 2.5 bar (right, black), and 2.5 bar + vacuum desorption (right, red).

The 0 bar experiments (Figure 3, left) stabilized at $\sim 1700 \text{ ppm}_{\text{NH}_3}$ after 4 minutes while the 2.5 bar experiments (Figure 3, right) stabilized at $\sim 1800 \text{ ppm}_{\text{NH}_3}$ after 5 minutes. One issue with these experiments was maintaining plasma at 2.5 bar. Therefore, these experiments started at 0 bar and ramped up to 2.5 bar. The 2.5 bar + vacuum experiment (right, red) only maintained plasma in 5/6 of the reactor tubes, hence a lower NH_3 output. Table 2 summarizes the NH_3 output of all 4 experiments.

Table 2: Ammonia output of the empty tube experiments.

		Total	0-5 min	20+ min
		mg NH_3	mg NH_3	mg NH_3
0 bar		12,9	2,5	0,6
0 bar + vacuum		13,5	2,7	0,8
2.5 bar		14,4	1,5	1,7
2.5 bar + vacuum		11,1	1,5	1,4

There doesn't seem to be any significant difference between the 0 bar and 0 bar + vacuum experiments. The difference between the 2.5 bar and 2.5 bar + vacuum experiments can be explained by the lack of the 6th reactor tube being active in 2.5 bar + vacuum. Accounting for this (dividing outputs of 2.5 bar + vacuum by 5/6), suggests there isn't a significant difference the 2.5 bar and 2.5 bar + vacuum experiments, and thus mirroring the 0 bar and 0 bar + vacuum experiments. The total output across all four experiments is similar, and the higher desorption output (20+ min) in the 2.5 bar experiments can be explained by the longer time needed to

exchange the reactor volume with the same mass flow at higher pressures (eg. slower volumetric flow at higher pressures). This would also apply to the longer stabilization time in the initial minutes of the experiments.

3.2 25:25:50 (ZeoSorp:NiOCat:Al₂O₃)

Testing the 25:25:50 (ZeoSorp:NiOCat:Al₂O₃) extrudate in the plasma reactor (~290 mg per tube, ~1740 mg per reactor) should demonstrate a sorption-enhanced effect where roughly 440 mg of Sorption should have the capacity of roughly ~20 mg_{NH3} (from D3.2 result of 44 mg_{NH3} per 1 g_{ZeoSorp-extrudate}). The experiments are summarized in Figure 4.

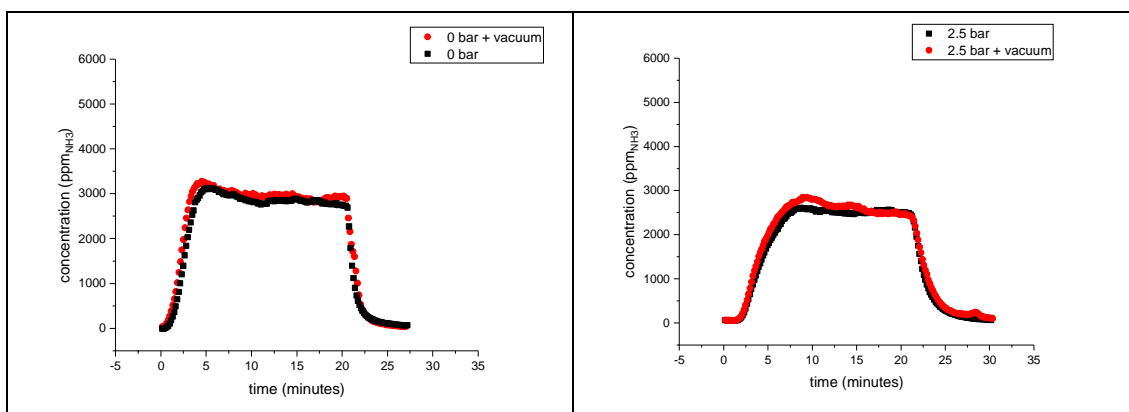


Figure 4: Plasma reactor experiments using 25:25:50 (ZeoSorp:NiOCat:Al₂O₃) extrudate with 0 bar (left, black), 0 bar + vacuum desorption (left, red), 2.5 bar (right, black), and 2.5 bar + vacuum desorption (right, red).

The 0 bar experiments (Figure 4, left) peaked at ~3100 ppm_{NH3} after 4-5 minutes and then stabilized at ~2900 ppm_{NH3} while the 2.5 bar experiments (Figure 4, right) stabilized at ~2600 ppm_{NH3} after 7-8 minutes. This demonstrates that the effective plasma for NH₃ synthesis is compromised by higher pressures, a well-known phenomenon (plasma generated at higher pressures requires more power). This is further outlined by the higher total NH₃ outputs of the 0 bar experiments summarized in Table 3.

Table 3: Ammonia output of the 25:25:50 (ZeoSorp:NiOCat:Al₂O₃) extrudate experiments.

		Total	0-5 min	20+ min
		mg NH ₃	mg NH ₃	mg NH ₃
	0 bar	21,8	2,9	1,4
	0 bar + vacuum	23,2	3,6	1,6
	2.5 bar	19,0	1,2	3,1
	2.5 bar + vacuum	20,3	1,4	3,2

About 13% of the total output is accounted for in the initial 0-5 minutes in the 25:25:50 (ZeoSorp:NiOCat:Al₂O₃) experiments at 0 bar, while ~20% of the total output is accounted for in the initial 0-5 minutes of the empty tube at 0 bar. This suggests there is NH₃ adsorption initially, which is expected to be even more prominent at higher pressures. As expected, about 7% of the total output is accounted for in the initial 0-5 minutes in the 25:25:50 (ZeoSorp:NiOCat:Al₂O₃) experiments at 2.5 bar. About 16% of the total output is accounted for after 20+ minutes in both the 25:25:50 (ZeoSorp:NiOCat:Al₂O₃) experiments at 2.5 bar. This suggests that there is some meaningful desorption being measured compared to the empty tube experiments (10%).

3.3 50:50 (NiOCat:Al₂O₃)

Testing the 50:50 (NiOCat:Al₂O₃) extrudate in the plasma reactor (~290 mg per tube, ~1740 mg per reactor) should demonstrate very little sorption enhancement, since the catalyst support, and the Al₂O₃ binder have negligible sorption capacity (<5 mg_{NH3} per 1 g).

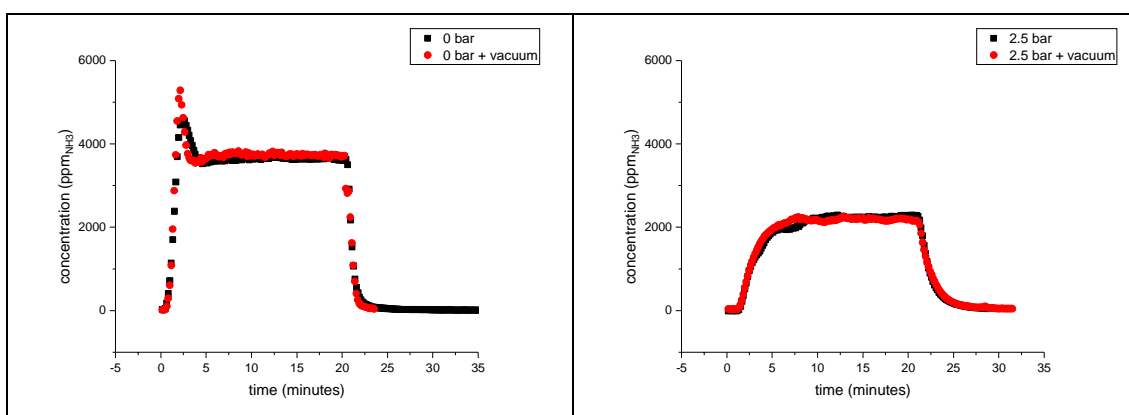


Figure 5: Plasma reactor experiments using 25:25:50 (Sorption:NiOCat:Al₂O₃) extrudate with 0 bar (left, black), 0 bar + vacuum desorption (left, red), 2.5 bar (right, black), and 2.5 bar + vacuum desorption (right, red).

The 0 bar experiments (Figure 5, left) peaked at 4500-5000 ppm_{NH3} after ~2 minutes and then stabilized at ~3600 ppm_{NH3}. Compared to 25:25:50 (Sorption:NiOCat:Al₂O₃) at 0 bar, the faster peak times suggests a lack of initial NH₃ adsorption, and the higher concentrations could be explained by the higher nickel loading. The 2.5 bar experiments (Figure 5, right) stabilized at ~2200 ppm_{NH3} after 7 minutes, replicating the lower outputs at higher pressures from the 25:25:50 (Sorption:NiOCat:Al₂O₃) experiments. Table 4 summarizes the NH₃ output of the 50:50 (NiOCat:Al₂O₃) experiments.

Table 4: Ammonia output of the 50:50 (NiOCat:Al₂O₃) extrudate experiments.

		Total	0-5 min	20+ min
		mg NH3	mg NH3	mg NH3
0 bar		29,5	5,8	1,7
0 bar + vacuum		29,8	6,1	1,4

	2.5 bar	17,1	1,5	2,3
	2.5 bar + vacuum	17,3	1,8	2,3

The higher initial NH₃ outputs (0-5 min) suggests a lack of adsorption: ~20% of the total output is accounted for in the initial 0-5 minutes in both the empty tube and the 50:50 (NiOCat:Al₂O₃) experiments at 0 bar. The higher pressure of 2.5 bar suggests more adsorption: ~10% of the total output is accounted for in the initial 0-5 minutes in both the empty tube and the 50:50 (NiOCat:Al₂O₃) experiments at 2.5 bar. However, it is important to remember that this is confounded with a slower volumetric flow, since the mass flow between the 0 bar and 2.5 bar experiments remains constant.

This lack of adsorption explains the lack of desorption with low outputs after the plasma has been turned off (20+ minutes): ~5% of the total output is accounted for after 20+ minutes in both the empty tube and the 50:50 (NiOCat:Al₂O₃) experiments at 0 bar. About 10% of the total output is accounted for after 20+ minutes in both the empty tube and the 50:50 (NiOCat:Al₂O₃) experiments at 2.5 bar. This suggests that there is no meaningful sorption being measured.

3.4 Conclusions on integrated sorption catalytic reactor

The highest output resulted from 50:50 (NiOCat:Al₂O₃) at 0 bar. However, a sorption-enhanced effect seems to be demonstrated because the 25:25:50 (Sorption:NiOCat:Al₂O₃) performed slightly better than the 50:50 (NiOCat:Al₂O₃) at 2.5 bar. This sorption-enhanced effect remains to be realized in a meaningful way, since the expected outcome was around 20 mg. This sorption-enhancement may not be viable because the NH₃ adsorption kinetics are not fast enough to compete with plasma radicals that would degrade the NH₃. Another possibility is the catalyst powder reaches temperatures that directly inhibits NH₃ adsorption. The lack of a meaningful sorption-enhancement effect could also explain why there was no significant difference between vacuum desorption and normal desorption across the experiments.

4. Sorption unit

A sorption unit was designed and build for tasks 5.2 where the NH₃ separation is done separately from the plasma reactor. The sorption unit design and materials are described in deliverable 3.2 and will therefore not be covered in this deliverable. This will only cover the testing and validation of the unit

4.1 Test setup and protocol

The separation unit was installed in the demonstration setup at DTI. This setup can supply gas at elevated pressure and has pumps to create vacuum in the unit. A heater was also installed on the unit to aid desorption. A typical test run is shown on Figure 6. Before each test run the unit was reset by heating it to 100°C while applying a vacuum (~0.5 bar below ambient) on the unit overnight. Then the unit was cooled to below 30C before starting the next sorption cycle. Sorption was performed with a 2% NH₃/N₂ gas bottle and supplied at 2.5 bar at a flow of 2000

mL/min. A slight heating of the unit was observed during each sorption cycle. After breakthrough had been achieved with a stable NH₃ conc. ~20000 ppm, the gas flow was stopped. The desorption was done by having an inlet gas flow of 200 mL/min N₂ and then varying the outlet flow by a vacuum pump and MFC to keep a vacuum pressure of about 0.5 bar below ambient while heating the unit to 100°C. NH₃ concentration was measured using a FTIR with a gas cell where the gas was diluted with N₂ 10x to keep the concentration within measurement range.

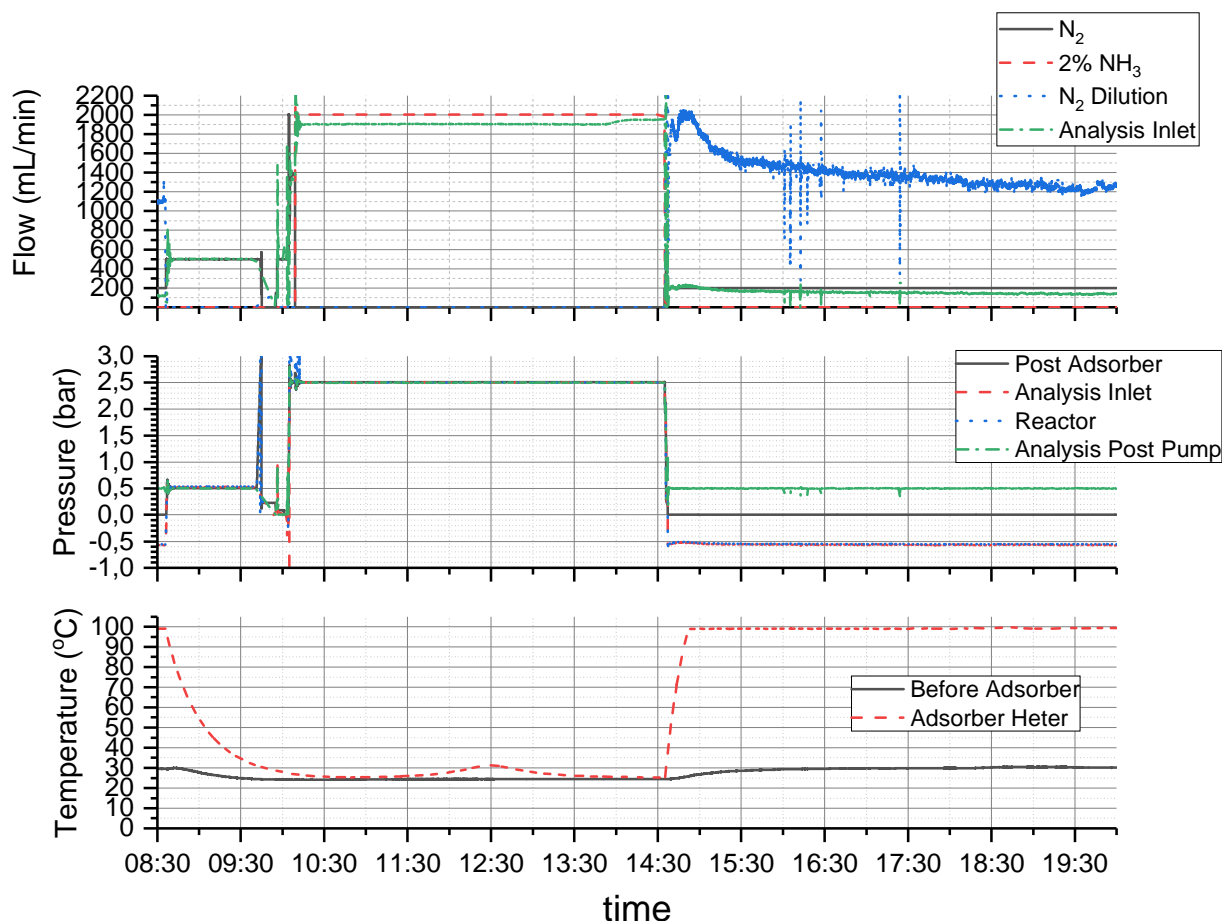


Figure 6: Typical test of sorption unit. This consists of 4 phases. 1) A pretreatment, before 08:30, where the unit was heated in vacuum overnight 2) cool down of the unit 3) sorption with 2% NH₃/N₂ until breakthrough, 4) desorption in vacuum with heating

4.2 Sorption cycles

The sorption unit was able to remove more than 99% of the NH₃ from the gas. The capacity was estimated from the breakthrough time, which is the time it takes before the NH₃ concentration increases on the outlet of the sorption unit. Three comparable sorption experiments on the same unit are shown on Figure 7. None of them are the initial sorption cycle as the unit has been

used using the same sorption material as no degradation had been observed. This can also be seen on the figure where there is a large variation in breakthrough time, but no systematic degradation. Instead, the breakthrough time seem to depend on the duration of the previous desorption cycle, which was not precisely controlled as it happened overnight. Based on data on the sorption material from deliverable 3.2 and the 2.5 bar pressure, the expected break through time should be after 3 hours. However, as stated the available capacity depends on the previous desorption.

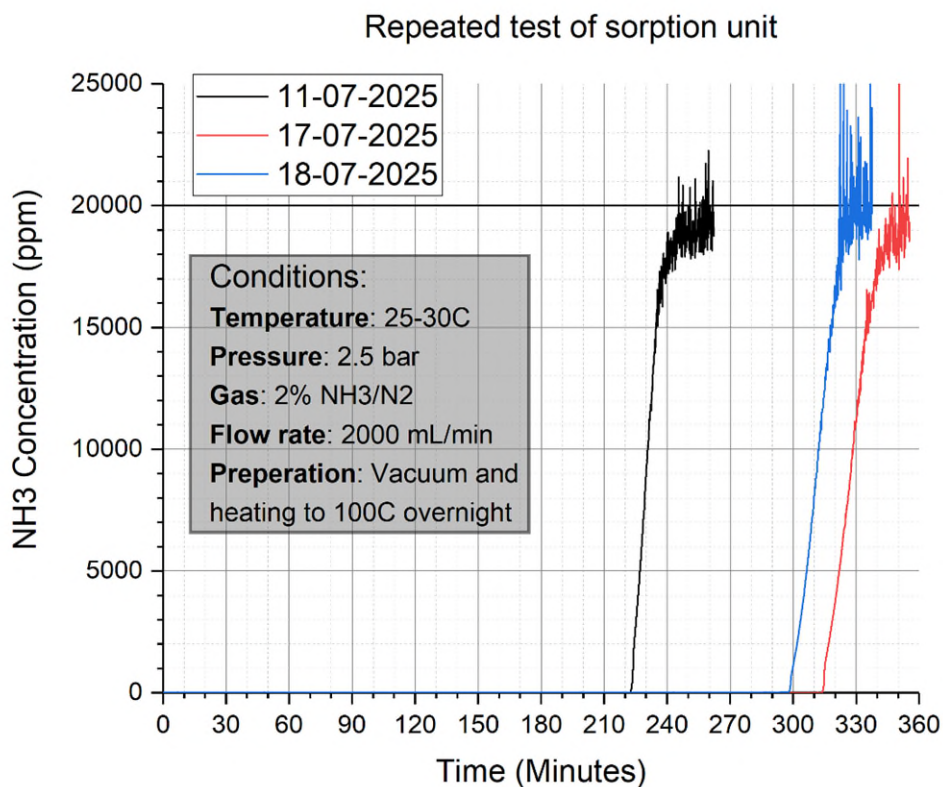


Figure 7: Sorption and breakthrough on three experiments. Most of NH₃ is removed until capacity is reached after which the NH₃ concentration increases to inlet level. Three experiment shown on graph with a large variation in breakthrough time.

4.3 Desorption cycles

Desorption was performed with a vacuum of 0.5 bar below ambient. Three desorption experiments are shown on Figure 8. In two of the experiments (red and blue) the unit was heated to 100°C during the experiment and the third experiment (green) was kept at room temperature during desorption. There is a big effect of heating during desorption. The outlet gas was diluted with N₂ 10 times before FTIR as the FTIR was only calibrated up to 10%. Most of the NH₃ is desorbed within the first hour which suggest that a more efficient operation of the unit would be to use less of its capacity, but which can be desorbed quickly with less energy usage.

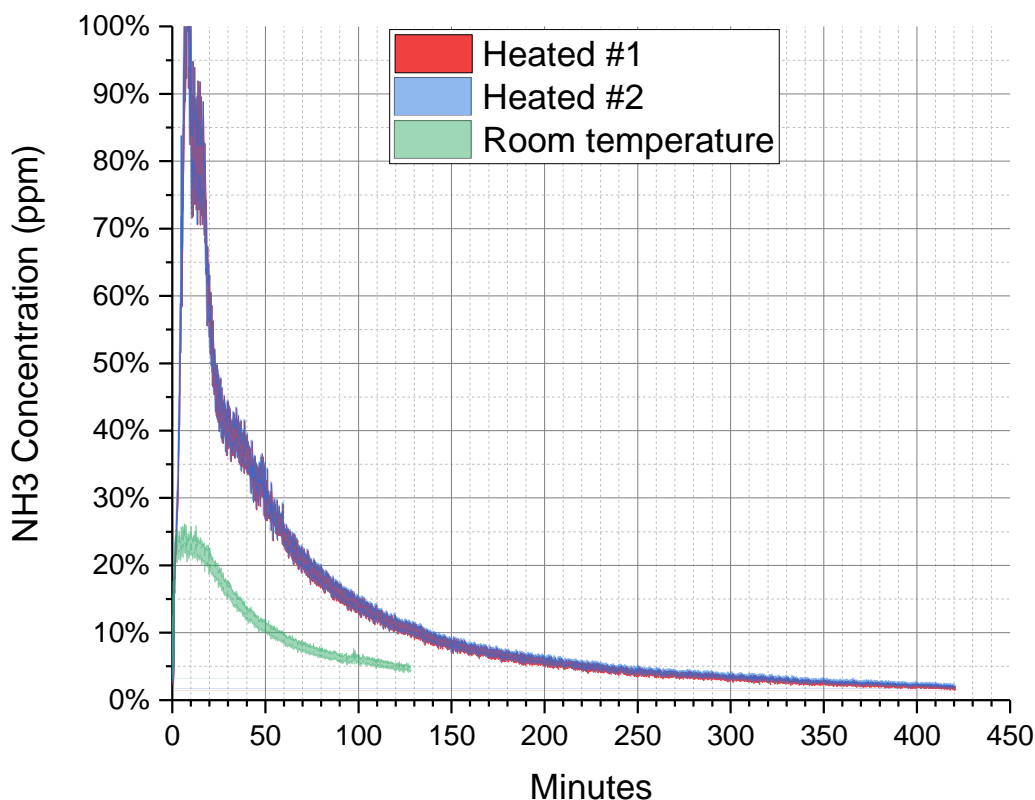


Figure 8: Desorption in three experiments. First two (red and blue) were heated to 100°C during desorption while the green was kept a room temperature during desorption. Desorption experiments performed with 10x dilution and concentration is estimated with that assumption.

5. Conclusions

The concept of combining a plasma catalytic reactor with NH₃ adsorbing material to separate the produced NH₃ from unreacted gasses was compared to having a separation unit after the reactor. The separation unit was able to remove more than 99% of the NH₃ in a 2% NH₃/N₂ stream and the separation unit was able to release most of the adsorbed NH₃ (>70%) in high concentrations (min 10%). The remaining NH₃ was slowly released at lower concentrations and over longer periods and thus for practical applications the full NH₃ capacity would not be used. The NH₃ release was temperature dependent and significant less NH₃ was released if no heating was applied.

Sorption enhancement was demonstrated when adding mixing in sorption material with the catalyst in the plasma reactor. The effect is however limited. Based on the temperature

sensitivity seen in the separation unit, the lack of temperature control inside the plasma might inhibit the effect. Ideally, the temperature should be close to room temperature when adsorbing material and then elevated to around 100°C when desorbing. The current reactor design does not have temperature measurement inside the plasma and can only cool from the outside. Future developments could include the ability to cool the inner electrode and add optical fibre based temperature sensors to enable more precise temperature control.

6. References

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