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BTA Frontier Application Note 1301

Trace Ammonia Capture on Zeolite 13X Under Dry and Humid Conditions

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Ammonia (NH_3) accumulation in enclosed environments including livestock facilities, industrial workspaces, wastewater treatment plants, and commercial or residential settings poses persistent risks to occupant health and air quality. Sorbent-based gas treatment systems offer a practical, energy-efficient route to controlling indoor ammonia concentrations, but selecting the right sorbent demands more than simple equilibrium measurements under idealised conditions. Materials must be evaluated under atmospheres that genuinely reflect the target environment including realistic ammonia concentrations, competing gases, and the humidity levels typical of occupied indoor spaces. This application note demonstrates how the BTA Frontier breakthrough analyser enables precise, multi-component characterisation of ammonia adsorption performance under conditions directly simulating indoor air streams. Taking zeolite 13X as a representative test material, we show how a breakthrough experiment can yield the key performance metrics that matter most for sorbent selection: usable breakthrough capacity, equilibrium saturation capacity, and the quantified impact of humidity on ammonia uptake.

Introduction

Ammonia (NH_3) is a hazardous atmospheric pollutant, contributing significantly to the formation of fine particulate matter ($PM_{2.5}$) and posing direct risks to human health at concentrations as low as 25 ppm [1]. A particularly acute and industrially significant source of ammonia emissions is the global poultry farming industry, where ammonia accumulates continuously inside housing units as a natural by-product of microbial breakdown of uric acid and undigested protein in litter and manure [2]. Left uncontrolled, in-house ammonia concentrations can exceed safe thresholds rapidly: regulatory bodies recommend levels be kept below 25 ppm to protect bird health and welfare, with the EU setting a stricter limit of 20 ppm and best-practice guidelines targeting below 10 ppm [3,4]. Prolonged exposure above these thresholds causes respiratory irritation in humans thus directly affecting the health of handlers, besides reducing growth rates, egg production, and increasing bird mortality thereby directly impacting farm profitability and animal welfare [1].

Different ammonia mitigation strategies, from dietary manipulation and litter additives to ventilation management, have been used to mitigate the emitted ammonia from poultry exhaust air [1,5,6]. As regulatory pressure intensifies particularly in densely farmed regions where ammonia reduction is now a condition for operation permits, the adoption of solid-phase gas adsorption technologies as part of air treatment systems is gaining traction. Adsorbent-based

approaches offer a clean, straightforward, energy-efficient, and scalable route to capturing ammonia from ventilation exhaust streams, making sorbent selection and performance characterisation a critical step in system design.

As the broader landscape of candidate sorbents ranging from zeolites, activated carbons, aluminas, metal oxides, and metal-organic frameworks continues to expand, selecting the optimal material requires accurate, process-relevant characterisation of adsorption capacity, breakthrough behaviour, and competitive uptake in the presence of humidity and other competing gases under more realistic conditions.

Traditional unary volumetric or gravimetric equilibrium isothermal measurements provide valuable data, but they cannot capture the dynamic, real-world conditions under which adsorbents must perform. Packed bed breakthrough analysis bridges this gap by passing a gas stream of defined composition directly through a column of the sorbent material and measuring the point at which the target species (in this case, ammonia) begins to elute. Key performance indicators such as usable breakthrough capacity and impact of other components such as humidity can all be extracted from a single experiment.

The BTA Frontier from Surface Measurement Systems is a purpose-built breakthrough analyzer for exactly such type of measurements. With up to 5 gas inlets and two vapor generators, its advanced multi-sensor architecture supports precise ammonia measurements from sub ppm level to thousands of ppm along with the accurate measurement of other components including humidity and CO_2, thus enabling precise quantification of ammonia uptake under competitive multi-component conditions, directly mimicking the humid, multi-component air streams found in poultry housing environments. Importantly, the instrument's high-performance gas-blending system and precise temperature and flow control minimise dead volume artefacts and ensure reproducible, process-relevant results across a broad concentration range. The dual-sample column configuration further maximises experimental throughput, allowing sequential sorbent activation and testing with minimal downtime.

In this application note, we demonstrate the capability of the BTA Frontier to characterise the dynamic ammonia adsorption performance of a representative zeolite 13X sorbent under simulated poultry house ammonia and humidity levels, providing the quantitative breakthrough data essential for adsorbent screening and process scale-up in similar air-treatment applications.

Methodology

All breakthrough experiments were performed on the BTA Frontier. As illustrated in the instrument schematic (Figure 1), the instrument operates on a packed bed chromatographic principle, a precisely blended gas mixture is introduced at one end of a sample-packed column, and the effluent concentration is continuously monitored at the outlet by a dedicated sensor train.

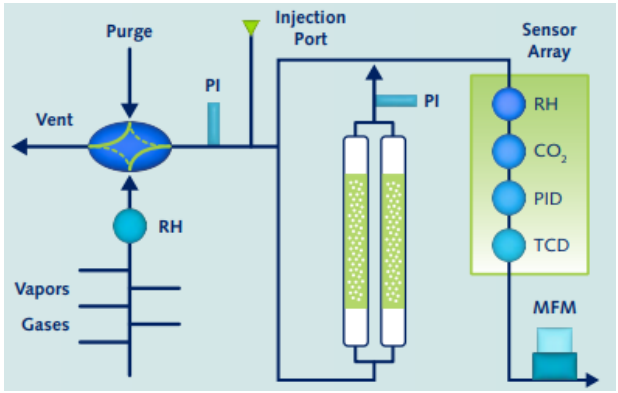

Figure 1. Simplified schematic of BTA Frontier.

The gas and vapor generation and mixing system feature up to five high turn-down mass flow controllers (MFCs), a dedicated inert purge inlet and up to two solvent reservoirs, enabling accurate multi-component mixtures across a wide concentration range. Humidity or other solvent vapors are generated by bubbling a controlled gas fraction through a heated liquid reservoir, with wet and dry streams blended in a custom manifold to deliver a stable, condensation-free vapour. An upstream switching manifold allows the column inlet to be toggled between the inert purge and the

target gas stream, enabling sharp concentration front generation and minimising dead volume artefacts particularly important for small sample masses. Pressure transducers upstream and downstream of the column monitor pressure drop across the bed, supporting accurate mass balance calculations. The outlet sensor train includes CO_2 Non-Dispersive Infrared (NDIR) sensor, capacitive Relative Humidity (RH) sensors, photoionization detectors (PID) for detection of a wide range of Volatile Organic Compounds (VOC's), NH_3 and H_2S and a general purpose Thermal Conductivity Detector (TCD) for binary and quasi binary mixture analysis all housed within a temperature-controlled incubator to ensure stable vapour generation and prevent condensation throughout the experiment. A dedicated resistive column oven with independent temperature control enables in-situ activation and regeneration.

For analysis, 18 mg of zeolite 13X 0.4 mm beads (Zeochem) were packed into a silanized glass column between two glass wool plugs. Prior to each experiment, the sample was activated in situ at 350 °C under a 200 sccm nitrogen flow, then cooled to the analysis temperature of 25°C under the same flow. Breakthrough adsorption experiments were then initiated by switching the feed from pure nitrogen to a blended gas mixture containing 50 ppm NH_3 and/or 55% RH at a flow rate of 100 sccm. Ammonia concentration at the column outlet was continuously monitored using calibrated PID sensors, while humidity was tracked using integrated capacitive sensors. Each experiment was run until near-saturation or for a maximum of 300 minutes, whichever came first. Upon completion, the feed was switched to a 100 sccm purge gas (N_2) and the bed temperature ramped to 350°C at 3 °C/min, held for 300 minutes to fully desorb retained species. The bed was then cooled under same nitrogen flow to target temperature before next breakthrough experiment was conducted. Blank experiments were performed under identical conditions using an inert bed material in place of the adsorbent, allowing dead volume contributions and sensor response delays to be accounted for in all subsequent calculations. The adsorbent column was reweighed after the final regeneration step to determine the dry mass of sorbent, used as the basis for all capacity calculations.

Results

Dry Conditions (50 ppm NH_3, 0 % RH)

To evaluate the regenerability and cyclic stability of the adsorbent, two consecutive adsorption–desorption cycles (BT-1, BT-2) were performed under identical conditions (50 ppm NH_3 inlet, 100 sccm), and the resulting breakthrough profiles are presented in Figure 2. A blank experiment (no adsorbent) was also conducted to characterise the system dead volume and instrument response time.

Both breakthrough experiments exhibited well-defined, sigmoidal breakthrough curves characteristic of favourable adsorption with the breakthrough onset observed at approximately 100 minutes, after which the outlet concentration rose gradually through the mass transfer zone before approaching saturation (~50 ppm) at approximately 230–250 minutes.

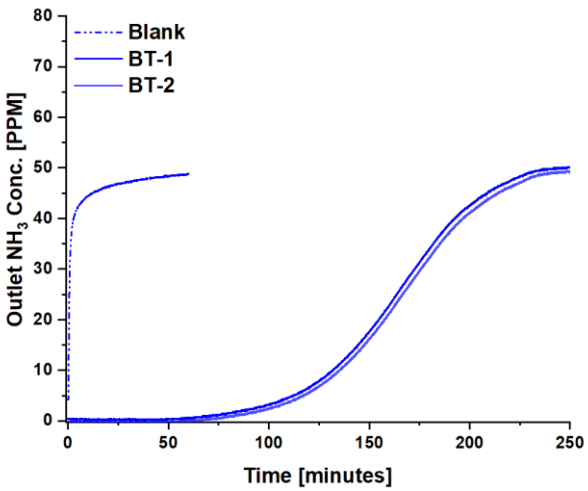

Figure 2. Ammonia breakthrough curves on Zeolite 13X

Notably, both successive cycles produced largely overlapping breakthrough adsorption profiles, demonstrating excellent cyclic reproducibility. Blank corrected saturation capacities calculated from the breakthrough curves indicate capacities of 1.96 and 1.98 mmol/g for BT-1 and BT-2 respectively. This behaviour indicates that the regeneration protocol employed between cycles was effective in restoring the adsorption capacity of the material. The consistent dynamic uptake

capacity across the two experiments confirms the structural and chemical stability of the adsorbent under repeated NH_3 adsorption–desorption cycling, a critical requirement for practical deployment in continuous gas-phase NH_3 removal applications.

Figure 3 presents the outlet NH_3 concentration profiles during temperature-programmed regeneration (R-1 and R-2) alongside the sample temperature profile and a blank reference. Regeneration was achieved by applying a linear temperature ramp (3°C/min) from 25 to 350°C, at which point the temperature was held isothermally for the remainder of the experiment.

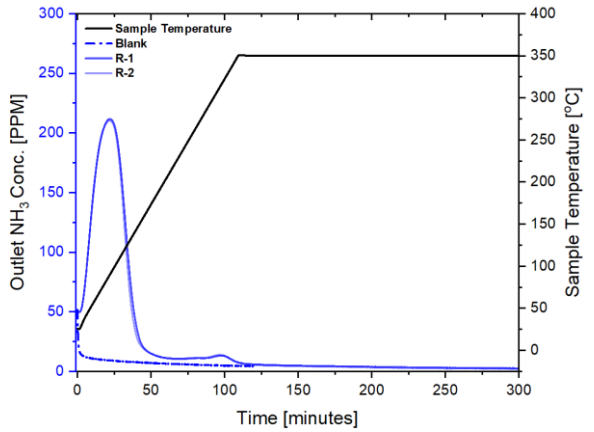

Figure 3. NH_3 desorption profile after adsorption under dry conditions.

Upon initiation of the temperature ramp, both regeneration cycles (R-1 and R-2) exhibited a sharp, well-defined NH_3 desorption peak centred around approximately 25–30 minutes, reaching a maximum outlet concentration of ~210 ppm (R-1) and slightly lower for R-2, coinciding with a sample temperature of approximately 75–100°C. This low-temperature desorption peak is consistent with the release of weakly or physisorbed NH_3 species from the adsorbent surface. Following this primary desorption event, the outlet NH_3 concentration declined sharply toward baseline levels as the temperature continued to rise. A minor secondary desorption feature was observed at approximately 100 minutes (~315°C), likely attributable to the release of more strongly bound NH_3 species or chemisorbed surface species at higher thermal energy. Beyond ~125 minutes, the outlet NH_3 concentration dropped below 5 ppm and continued tailing further for the duration of the isothermal hold at 350°C, confirming near complete desorption and successful regeneration of the adsorbent. The specific desorbed ammonia amounts calculated from the desorption curves stand at 1.78 and 1.75 mmol/g which are largely consistent with the preceding adsorption uptakes (Figure 4). The close agreement between R-1 and R-2 desorption profiles further corroborates the cyclic reproducibility of both the adsorption and regeneration steps, consistent with the breakthrough reproducibility demonstrated in Figure 2.

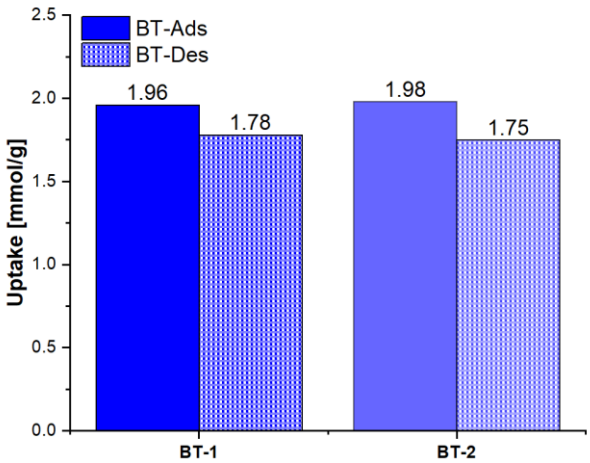

Figure 4. Adsorption and desorption capacities under dry conditions.

Humid Conditions (50 ppm NH_3, 55% RH)

Figure 5 presents the NH_3 and H_2O breakthrough curves obtained when the adsorbent was exposed to a humid (55% RH) 50 ppm NH_3 feed stream. In contrast to dry conditions, ammonia breakthrough occurred within a few minutes, nearly simultaneously with the water breakthrough, followed by a momentary concentration spike. This transient overshoot likely reflects displacement of weakly adsorbed NH_3 by the advancing water front, consistent with competitive co-adsorption between NH_3 and H_2O on the hydrophilic zeolite 13X surface. Following the spike, the effluent NH_3 concentration rises only gradually, as evidenced by the shallow slope of the breakthrough curve, indicating that NH_3 continues to adsorb on the humidity-saturated adsorbent, albeit at a substantially reduced rate.

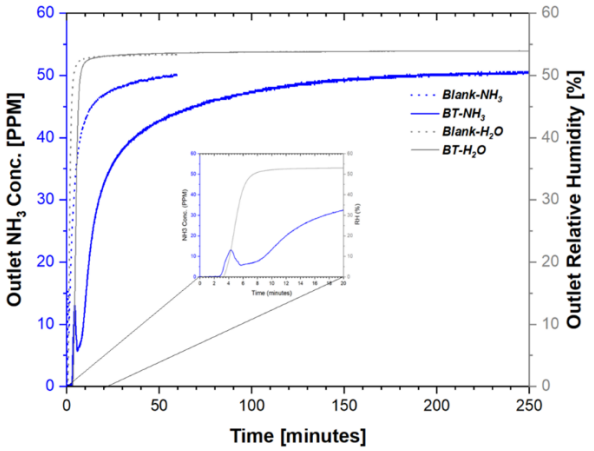

Figure 5. NH_3 and H_2O breakthrough curves on Zeolite 13X.

The extended approach to saturation and the absence of a well-defined plateau within the 250-minute experimental window suggest that some dynamic NH_3 uptake capacity is retained under humid conditions, though it is markedly reduced relative to dry breakthrough performance.

Adsorption capacities calculated from integration of the breakthrough curves yield an NH_3 uptake of 0.32 mmol/g and a substantially higher H_2O capacity (14.82 mmol/g), as expected for the strongly hydrophilic zeolite 13X framework. The reduced NH_3 capacity under humid conditions is consistent with competitive adsorption, whereby preferential occupation of active sites by water limits available sites for NH_3 uptake.

Regeneration of the NH_3 and H_2O-saturated adsorbent was performed using the same temperature-programmed protocol applied following dry NH_3 adsorption experiments (Figure 6). Both ammonia and water were successfully desorbed during the temperature ramp, confirming that thermal regeneration is effective under humid operating conditions. The NH_3 desorption profile closely resembles that observed after dry saturation; however, the peak desorption concentration and integrated area under the desorption curve are substantially lower, consistent with the reduced NH_3 uptake capacity measured under humid conditions.

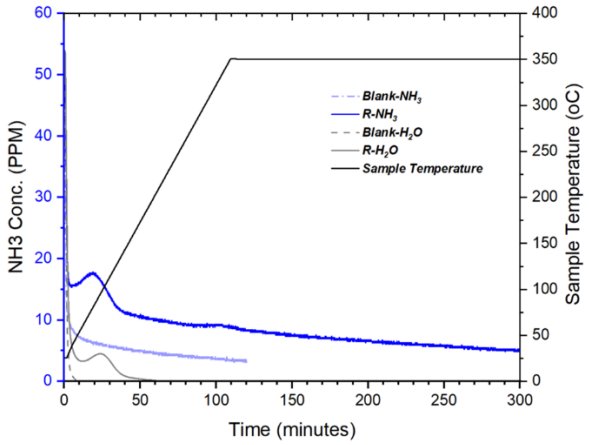

Figure 6. NH_3 and H_2O desorption profiles of Zeolite 13X

Desorption capacities derived from integration of the desorption curves are in good agreement with the corresponding adsorption capacities determined in the preceding breakthrough experiment, again confirming complete adsorbent regeneration. A general comparison of the adsorption and desorption uptakes at both dry and humid conditions is shown in Figure 7 indicating an 80 % drop in ammonia capacity under humid conditions as compared to dry conditions.

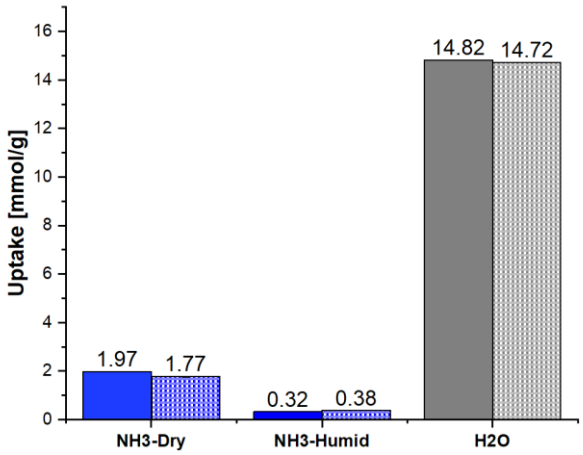

Figure 7. Adsorption (solid) and desorption (patterned) uptake from breakthrough experiments under dry and humid conditions.

Conclusions

This application note has demonstrated the capability of the BTA Frontier to deliver precise, process-relevant characterisation of ammonia adsorption performance under conditions directly simulating ppm ammonia concentrations as in poultry house exhaust air. Using only 18 mg of zeolite 13X as a representative adsorbent, the instrument generated reproducible, quantitative breakthrough profiles across both dry and humid operating conditions, extracting the key performance metrics about the adsorbent.

Under dry conditions zeolite 13X exhibited high NH_3 capacity at 50 ppm of approximately 1.97 mmol/g, alongside excellent cyclic reproducibility across two consecutive adsorption–desorption cycles. Regeneration at 350°C achieved near-complete desorption of retained ammonia. Under humid conditions, competitive co-adsorption between NH_3 and H_2O significantly altered the breakthrough behaviour. Ammonia breakthrough occurred within minutes, nearly simultaneously with the water front, and the NH_3 uptake capacity was reduced by 80% from dry conditions reflecting preferential occupation of active adsorption sites by water. Despite this reduction, a residual dynamic NH_3 uptake capacity was retained beyond water saturation, and thermal regeneration confirmed complete recovery of both NH_3 and H_2O with no evidence of irreversibly bound species.

These results illustrate how the BTA Frontier's multi-sensor architecture, precise gas blending, and independent thermal control combine to enable rapid, milligram-scale characterisation of adsorbent performance under realistic multi-component conditions. The ability to simultaneously quantify competitive NH_3 and H_2O uptake, assess cyclic stability, and validate regeneration completeness within a single experimental workflow positions the BTA Frontier as a powerful tool for accelerating adsorbent screening and development across ammonia removal, air quality, and broader gas treatment applications.

References

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