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SAND2004-0022

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Printed January, 2004

Quantification of Ammonia Binding Sites in Davison (Type 3A) Zeolite Desiccant: A Solid-State Nitrogen-15 MAS NMR Spectroscopy Investigation

Todd M. Alam, Greg P. Holland and Brian R. Cherry

Prepared by
Sandia National Laboratories
Albuquerque, New Mexico 87185 and Livermore, California 94550

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Todd M. Alam*, Greg P. Holland and Brian R. Cherry
Department of Organic Materials
Sandia National Laboratories
P.O. Box 5800
Albuquerque, NM 87185-0888

Abstract

The quantitative analysis of ammonia binding sites in the Davison (Type 3A) zeolite desiccant using solid-state ^{15}N MAS NMR spectroscopy is reported. By utilizing ^{15}N -enriched ammonia ($^{15}\text{NH}_3$) gas, the different adsorption/binding sites within the zeolite were investigated as a function of NH_3 loading. Using ^{15}N MAS NMR multiple sites were resolved that have distinct cross-polarization dynamics and chemical shift behavior. These differences in the ^{15}N NMR were used to characterize the adsorption environments in both the pure 3A zeolite and the silicone-molded forms of the desiccant.

* Author to whom correspondence should be addressed: tmalam@sandia.gov

Acknowledgements

Sandia is a multiprogram laboratory operated by Sandia Corporation, a Lockheed Martin Company, for the United States Department of Energy's National Nuclear Security Administration under Contract DE-AC04-94AL85000. The authors are also indebted to Jim Schicker from Honeywell/FM&T for providing samples of the W76 AF&F silicone-molded desiccant.

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Nomenclature

| | |
|------|----------------------------|
| NMR | nuclear magnetic resonance |
| MAS | magic angle spinning |
| DP | direct-polarization |
| CP | cross-polarization |
| TPPM | two pulse phase modulation |

Quantification of Ammonia Binding Sites in Davison (Type 3A) Zeolite Desiccant: A Solid-State Nitrogen-15 MAS NMR Spectroscopy Investigation

Introduction

Zeolites are extensively used in a wide variety of applications including catalysis, gas separation, and as drying agents.¹⁻³ Small molecules will adsorb in the cage-like structures of these aluminosilicate materials if the adsorbate has a critical diameter smaller than the pore size of the zeolite.³ Varying the cations present in the cage can control this pore size.² In the case of zeolite A molecular sieves (which are typically used as drying agents) the sodium (Na) form has a pore size of ~4 angstroms (Å) while the potassium (K) exchanged material has a 3Å pore diameter.³ The crystal structures⁴ and physical properties⁵ of zeolite A molecular sieves have been known for decades; however, the local environment of adsorbed species is still not well understood, particularly for the 3A molecular sieve.

Solid-state ¹⁵N NMR spectroscopy has been successfully employed in the study of adsorption environments on solid surfaces.⁶⁻¹⁴ Enriched NH₃ and N₂ gas have been implemented to quantitatively study the adsorption sites in various zeolites.^{6-8,10} The ¹⁵N chemical shift is a valuable tool for distinguishing between complexes, binding environments, gaseous and even liquid environments in the zeolite cage.^{6,7,10} Further information regarding Lewis and Brønsted acidity can also be determined from ¹⁵N NMR data.^{9,11,13,14}

In this SAND report we utilized solid-state ¹⁵N cross polarization magic angle spinning (CP-MAS) NMR spectroscopy to directly determine the different ammonia

adsorption/binding sites in the Davison 3A molecular sieve. The relative concentrations of these different ammonia adsorption sites as a function of loading were analyzed for both the unmolded 3A zeolite desiccant and silicone-molded desiccant samples.

Experimental Details

A. Silicone-molded Desiccant

The silicone-molded desiccant samples were obtained from FM&T (J. Schicker), and received at SNL on 4/11/02. These samples were produced as 6" x 6" x 1/8" molded slabs through the combination of two materials as detailed below. The molded materials utilized a RTV silicone resin obtained from General Electric (GE Silicones, Waterford, NY) designated GE615 (material standard 2070091-01) plus Davison brand (Davison Chemical Division, W. R. Grace and Company, Baltimore, MD) type 3A molecular sieve (material standard 8500042-01-810). The desiccant was added to silicone resin by vacuum mixing in a 50:50 weight percent ratio. The slabs were poured in a mold then mold cured for 4 hours at 67 °C.

B. Desiccant Activation

Activation of both the native Davison 3A molecular sieve (Lot # SMR 4-052085) and the silicone-molded desiccants was achieved by drying the samples at 176 °C under vacuum for 48 hours. The samples were immediately transferred from the oven to a nitrogen-filled glove box to avoid moisture contamination.

C. Ammonia Loading

Dried desiccant samples were then packed and sealed in a stainless steel container. For all experiments described, approximately 300 mg of sample was packed in a nitrogen-filled glove box to avoid moisture contamination. Desiccant samples under vacuum were exposed to a given pressure (100-700 torr) of ^{15}N -labelled ammonia gas (Cambridge Isotopes). The system was allowed to equilibrate to some final pressure after 2-5 hours. The total amount of adsorbed gas was calculated from the initial and final pressures implementing the ideal gas law. Ammonia loading is expressed in mmol of ammonia per gram of Davison 3A molecular sieve. In the case of the silicone-molded desiccants the weight of the polymer is not included in this calculation.

D. NMR Analysis

The solid-state ^{15}N MAS NMR spectra were obtained at a resonance frequency of 40.55 MHz, on a Bruker Avance400 NMR spectrometer using a 7 mm CP-MAS probe spinning at 5 kHz. Typical ^{15}N MAS NMR conditions for DP experiments used an 8.5 μs $\pi/2$ pulse, 170 s recycle delay and 128 scan averages. The ^{15}N CP-MAS NMR spectra were obtained using 1024 scans, an 8.5 μs ^1H $\pi/2$ pulse, a 4.5 ms contact pulse, and a 2 s recycle delay. TPPM ^1H decoupling was applied during acquisition in both DP and CP experiments. The ^{15}N NMR spectra were referenced to nitromethane ($\delta = 0.0$ ppm) by calibrating to a secondary standard of ^{15}N -labelled glycine ($\delta = -347.6$ ppm). Spin-lattice

(T_1) relaxation measurements were performed with the saturation recovery method.¹⁵ Spectral deconvolutions of the observed NMR resonances were performed using the dmfit program.¹⁶

Results

A. NH₃ Binding to Non-molded Desiccant

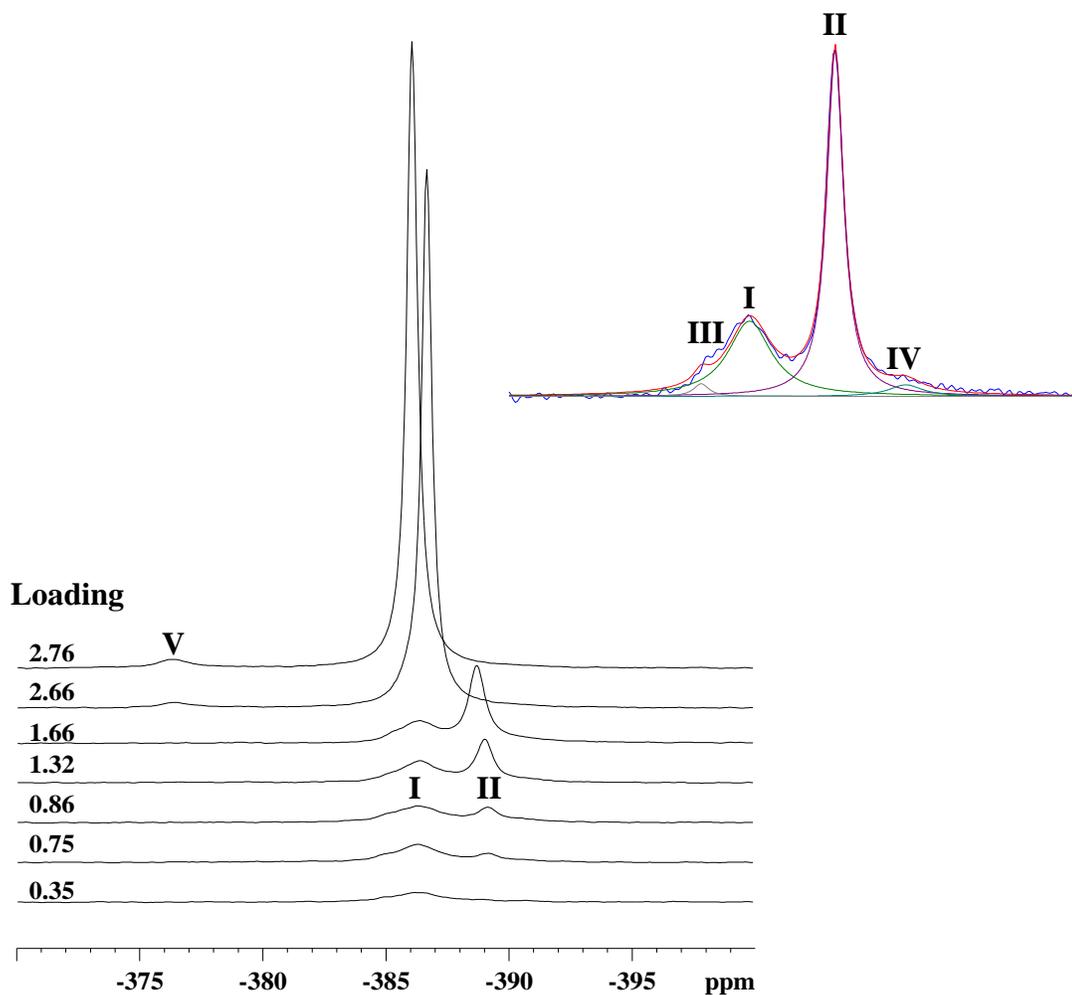
The solid-state ¹⁵N MAS NMR spectra for the Davison 3A zeolite desiccant as a function of ammonia loading are shown in **Figure 1A**. There are two primary resonances resolved in the spectra. These two resonances, labeled **I** and **II**, comprise a minimum of 85% of the observed signal. However, five distinct environments are extracted upon spectral deconvolution as shown in **Table I**. This report will focus on understanding resonances **I** and **II** since they represent the vast majority of the ammonia adsorption sites in this material.

Table I. Chemical shift (δ) in ppm, % fraction (F), and concentration (Conc.) of different ammonia sites obtained from ¹⁵N MAS NMR spectra of 3A Davison zeolite desiccant

| 3A Zeolite | I | | | II | | | III | | |
|----------------------------|-----------|----------|--------------------------|-----------|----------|--------------|------------|----------|--------------|
| Loading^a | δ | F | Conc.^b | δ | F | Conc. | δ | F | Conc. |
| 0.35 | -386.4 | 76.5 | 0.27 | -388.8 | 8.4 | 0.03 | -385.0 | 11.4 | 0.04 |
| 0.75 | -386.3 | 77.7 | 0.58 | -389.2 | 15.0 | 0.11 | -385.1 | 2.7 | 0.02 |
| 0.86 | -386.4 | 64.9 | 0.56 | -389.1 | 25.4 | 0.22 | -385.1 | 5.8 | 0.05 |
| 1.32 | -386.4 | 52.2 | 0.69 | -389.1 | 45.2 | 0.60 | -385.1 | 1.4 | 0.02 |
| 1.66 | -386.4 | 33.9 | 0.56 | -388.7 | 63.6 | 1.06 | -385.1 | 2.5 | 0.04 |
| 2.66 | -386.4 | 17.4 | 0.46 | -386.7 | 77.0 | 2.05 | -385.1 | 2.3 | 0.06 |
| 2.76 | -386.4 | 7.0 | 0.19 | -386.1 | 89.2 | 2.46 | -385.1 | <1.0 | - |
| | IV | | | V | | | VI | | |
| Loading | δ | F | Conc. | δ | F | Conc. | δ | F | Conc. |
| 0.35 | -390.7 | 3.7 | 0.01 | - | - | - | - | - | - |
| 0.75 | -390.6 | 4.6 | 0.03 | - | - | - | - | - | - |
| 0.86 | -390.6 | 3.9 | 0.03 | - | - | - | - | - | - |
| 1.32 | -390.6 | 1.2 | 0.02 | - | - | - | - | - | - |
| 1.66 | -390.6 | <1 | - | - | - | - | - | - | - |
| 2.66 | -390.6 | <1 | - | -376.5 | 3.4 | 0.09 | - | - | - |
| 2.76 | -390.6 | <1 | - | -376.4 | 3.8 | 0.10 | - | - | - |

^aLoading in mmol of NH₃ per gram of activated desiccant

^bConcentration given by mmol species per gram of activated desiccant



(B)

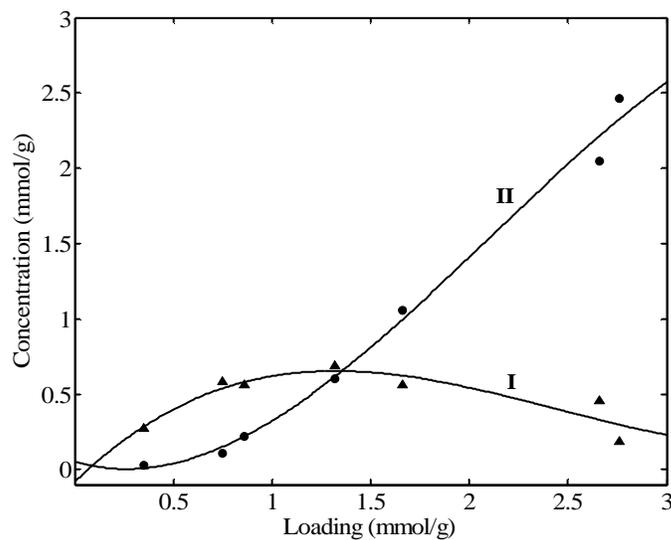


Figure 1: High-resolution ^{15}N MAS NMR for 3A zeolite desiccant with various amounts of adsorbed NH_3 in mmol/g desiccant (A). A spectral deconvolution of the zeolite sample with 1.66 mmol/g is included to show the multiple NH_3 environments. Concentration of NH_3 at sites I and II as a function of total loading (B).

The ^{15}N species giving rise to Resonance **I** has a consistent chemical shift of -386.3 ± 0.1 ppm across the entire ammonia loading range and appears to be occupied first since resonance **I** dominates the spectrum at the lowest loading of 0.35 mmol/g. This adsorption environment appears saturated at a loading of 1.32 mmol/g while, resonance **II** continues to populate continuously (see **Figure 1B**) with increased loading. The main contrast between resonances **I** and **II** is the continuous chemical shift displayed by resonance **II** with increasing NH_3 loading. This behavior has been observed previously in the ^{15}N MAS NMR spectra of NH_3 in various zeolitic materials and was unambiguously attributed to a close packing of the ammonia molecules resulting in a shift towards that of liquid ammonia (-381.9 ppm).⁷ The constant chemical shift displayed by resonance **I** indicates that this adsorption environment is isolated from associating with other NH_3 molecules in the concentration range studied.

The solid-state $^1\text{H} \rightarrow ^{15}\text{N}$ CP-MAS NMR spectra for the Davison 3A zeolite desiccant as a function of ammonia loading are shown in **Figure 2**. Similar environments are observed compared to the DP experiments depicted in **Figure 1**, but differences in relative intensities are observed. In the CP spectra presented in **Figure 2** resonance **I** displays a more intense signal than resonance **II** and therefore, is more effectively cross-polarized by neighboring protons. This is most obvious comparing the DP spectrum of the sample containing 1.32 mmol/g in **Figure 1** where resonance **II** displays the more intense resonance while, the CP spectrum in **Figure 2** the opposite is observed and **I** has a more intense signal. These differences in CP efficiency typically indicate differences in mobility between the two environments since the proximity of protons is similar.

Therefore, resonance **I** likely corresponds to NH_3 molecules occupying a site that is more strongly bound than those represented by resonance **II**. This is also consistent with relaxation data that measured a T_1 relaxation time of 34 and 4 s for resonance **I** and **II**, respectively. Longer relaxation times are typically characteristic of a more rigid environment. Based on the chemical shift behavior, CP efficiency, and T_1 relaxation times we assign resonance **I** to bound NH_3 which is probably associated with the cations (Na and K) present in the cage and resonance **II** to “free” ammonia trapped in the zeolite cage.

Resonances **III**, **IV**, and **V** represent defect binding sites within the zeolite material, but since they comprise a minor concentration (<15%) they will not be discussed further.

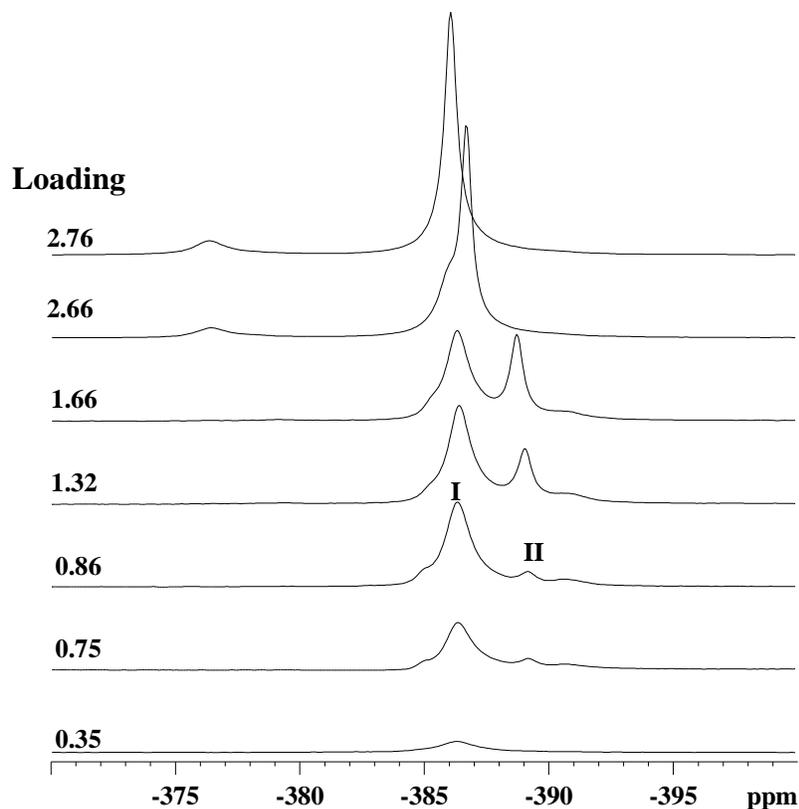


Figure 2: High-resolution ^{15}N CP-MAS NMR for 3A zeolite desiccant with various amounts of adsorbed NH_3 . Loading is represented as mmol NH_3 per gram of activated desiccant.

B. NH_3 Binding in Silicone-Molded Desiccant

The ^{15}N MAS NMR spectra of the silicone-molded 3A zeolite desiccant as a function of NH_3 loading are displayed in **Figure 3**. These NMR spectra (see **Figure 3**) show some differences compared to the spectra of the native 3A zeolite desiccants (see **Figure 1**). A larger contribution from the resonances at -365 ppm and -376 ppm is observed as shown in **Table II**. The resonance at -365 ppm is attributed to NH_4^+ species consistent with ^{15}N NMR chemical shift of aqueous NH_4NO_3 .¹⁷ A similar environment

has been observed previously in HY type zeolites where it was assigned to NH_4^+ formed due to NH_3 interactions with hydroxyl environments on the aluminosilicate framework.⁷ The production of this NH_4^+ species in the 3A zeolite studied here could result due to an analogous mechanism; however the possibility of interactions between NH_3 and proton sites on the polymer can not be discounted. It is also interesting to note that this NH_4^+ does not appear in any of the NMR spectra of the native 3A zeolite. The resonance at –376 ppm is ascribed to “free” NH_3 interacting with NH_4^+ species based on a previous study which showed that the ^{15}N resonance of NH_3 demonstrates a 25-35 ppm higher chemical shift than NH_3 gas (-399.9 ppm) when in contact with NH_4^+ . Similar observations have been reported in zeolite Y.⁸

Table II. Chemical shift (δ) in ppm and % fraction (F) obtained from fitting ^{15}N NMR spectra of silicone-molded 3A zeolite desiccant samples containing different amounts of ammonia

| 3A Pad | I | | | II | | | III | | |
|----------------------------|----------------------------|----------|--------------------------|----------------------------|----------|--------------|----------------------------|----------|--------------|
| Loading^a | δ | F | Conc.^b | δ | F | Conc. | δ | F | Conc. |
| 0.55 | -386.2 | 50.6 | 0.28 | -389.9 | 35.2 | 0.19 | - | - | - |
| 1.05 | -386.0 | 29.7 | 0.31 | -387.8 | 61.9 | 0.65 | - | - | - |
| 1.35 | -385.6 | 38.8 | 0.52 | -385.9 | 54.0 | 0.73 | - | - | - |
| 2.00 | -384.1 | <1 | - | -385.4 | 90.7 | 1.81 | - | - | - |
| | IV | | | V | | | VI | | |
| Loading | δ | F | Conc. | δ | F | Conc. | δ | F | Conc. |
| 0.55 | -391.3 | <1 | - | -379.9 | 3.8 | 0.02 | -367.0 | 9.8 | 0.05 |
| 1.05 | -388.4 | <1 | - | -378.5 | 2.5 | 0.03 | -366.1 | 5.9 | 0.06 |
| 1.35 | - | - | - | -375.8 | 2.9 | 0.04 | -365.6 | 4.4 | 0.06 |
| 2.00 | - | - | - | -375.8 | 5.6 | 0.11 | -365.3 | 3.3 | 0.07 |

^aLoading in mmol of NH_3 per gram of activated desiccant

^bConcentration given by mmol species per gram of activated desiccant

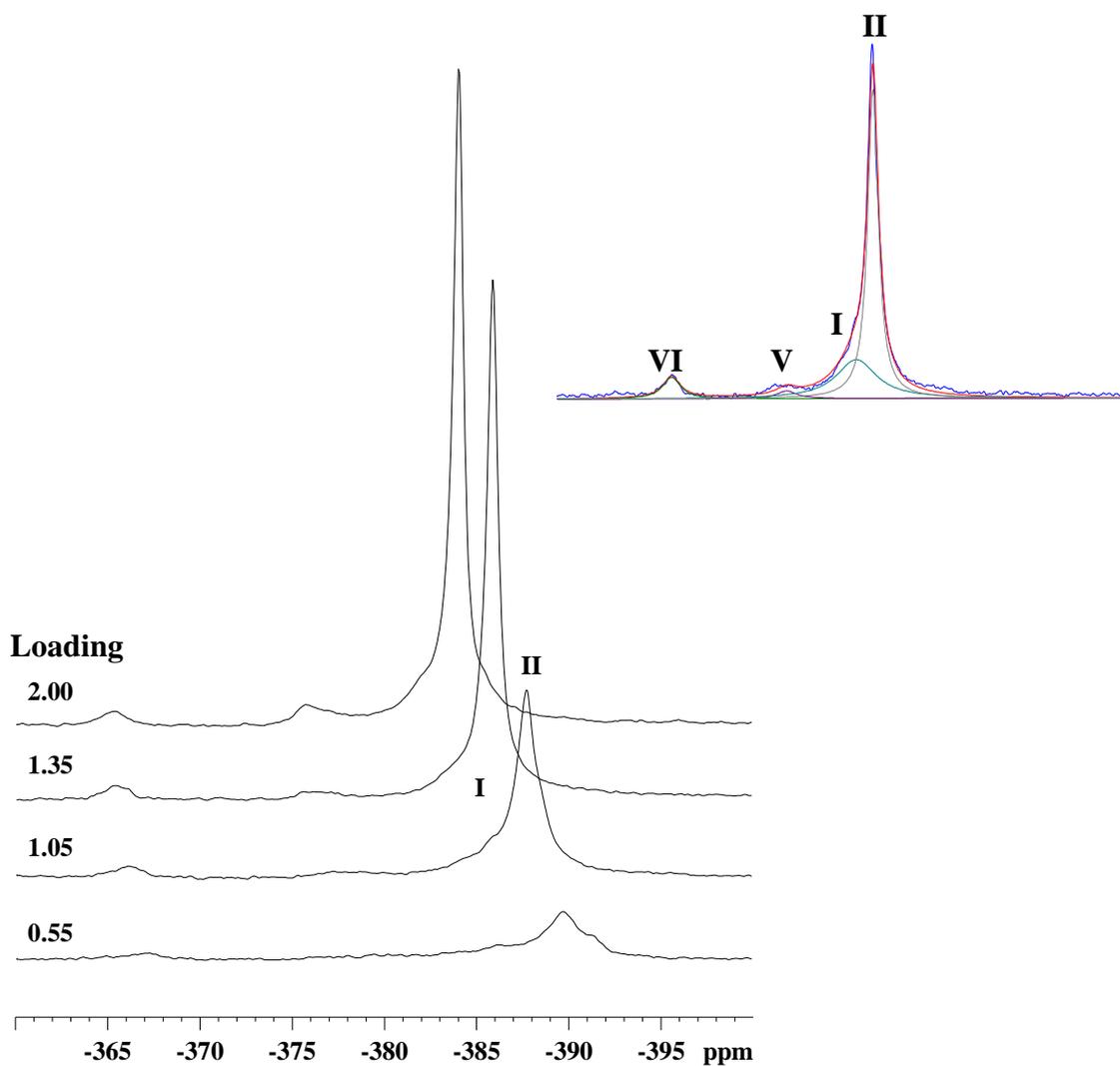


Figure 3: High-resolution ^{15}N MAS NMR spectra for 3A zeolite silicone-molded desiccant pad with various amounts of adsorbed NH_3 . Loading is represented as mmol NH_3 per gram of activated desiccant. A spectral deconvolution of the silicone-molded desiccant with 1.05 mmol/g is included to show the multiple NH_3 environments.

Another main difference between the ^{15}N NMR spectra of the pure 3A zeolite desiccant and the spectra of the silicone-molded desiccant samples is the larger ^{15}N NMR chemical shift observed for resonance **II** that was previously attributed to “free” NH_3 molecules. This indicates a stronger association between the NH_3 molecules in the

molded desiccant or a greater amount present in the large cage. This observation will be explained further in the discussion below.

The ^{15}N CP-MAS NMR spectra of the silicone-molded desiccant as a function of NH_3 loading is shown in **Figure 4**. Similar results are observed compared to the pure 3A zeolite desiccant, where resonance **I** displays a more enhanced intensity than resonance **II**. This has been linked to a more rigid, bound environment of the former species. In addition, the two resonances at -365 ppm and -376 ppm display a signal enhancement compared resonance **II** indicating that neighboring protons more effectively cross-

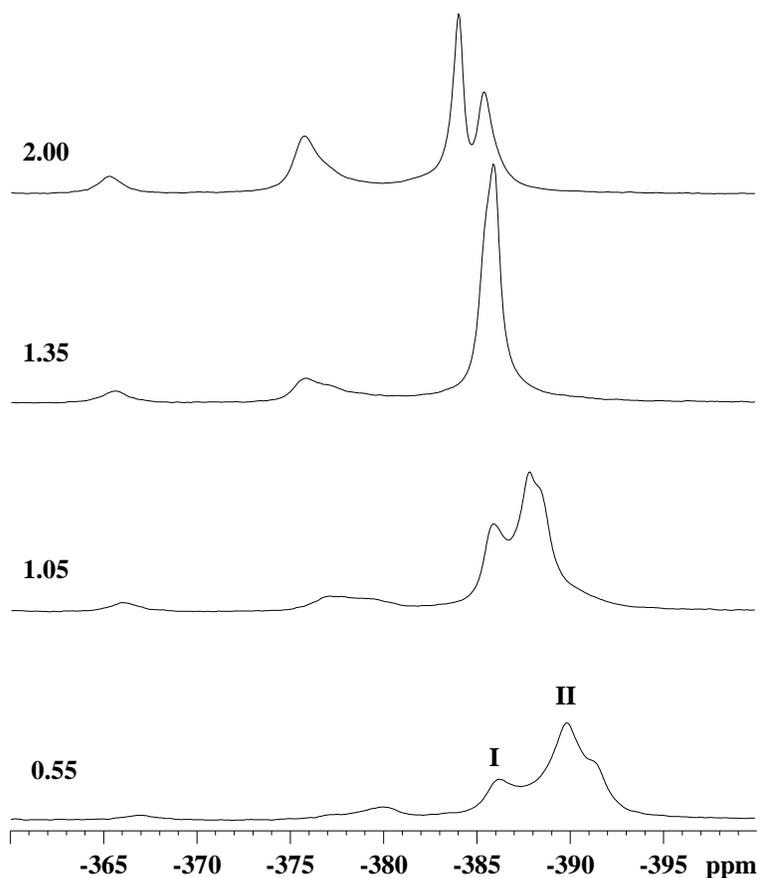


Figure 4: High-resolution ^{15}N CP-MAS NMR for zeolite 3A zeolite silicone-molded desiccant with various amounts of adsorbed NH_3 . Loading is represented as mmol NH_3 per gram of desiccant.

polarize these environments. This is consistent with the assignment of these two environments to NH_4^+ species formed at hydroxyl groups on the framework and NH_3 interacting with these NH_4^+ environments, respectively. The latter occurs presumably through hydrogen bonding.

Discussion

Two main resonances corresponding to two distinct adsorption species are observed in both the native 3A zeolite and silicone-molded forms of the desiccant. The main contrast between the solid-state ^{15}N NMR results of the pure 3A zeolite and the silicone-molded desiccant is the significantly larger chemical shift observed for the “free NH_3 ” (resonance **II**). This chemical shift towards liquid NH_3 demonstrates that this environment is becoming more liquid-like indicating interaction between multiple NH_3 molecules in the zeolite cage. The chemical shift is plotted in **Figure 5** as a function of ammonia loading. The chemical shift of the pure zeolite begins shifting when the number of NH_3 molecules per large cage (N) is greater than 1 while, the silicone-molded desiccant displays an immediate variation in chemical shift.⁴ The results for the pure desiccant are consistent with previously observed ^{15}N chemical shifts in zeolites.⁷ The chemical shift behavior observed for the silicone-molded forms indicate that there is more NH_3 per large cage than expected, based on the amount of zeolite present. This could indicate that all the zeolite embedded in the silicone-molded polymer are not equally active and that some could possibly be inactive or not accessible to NH_3 binding.

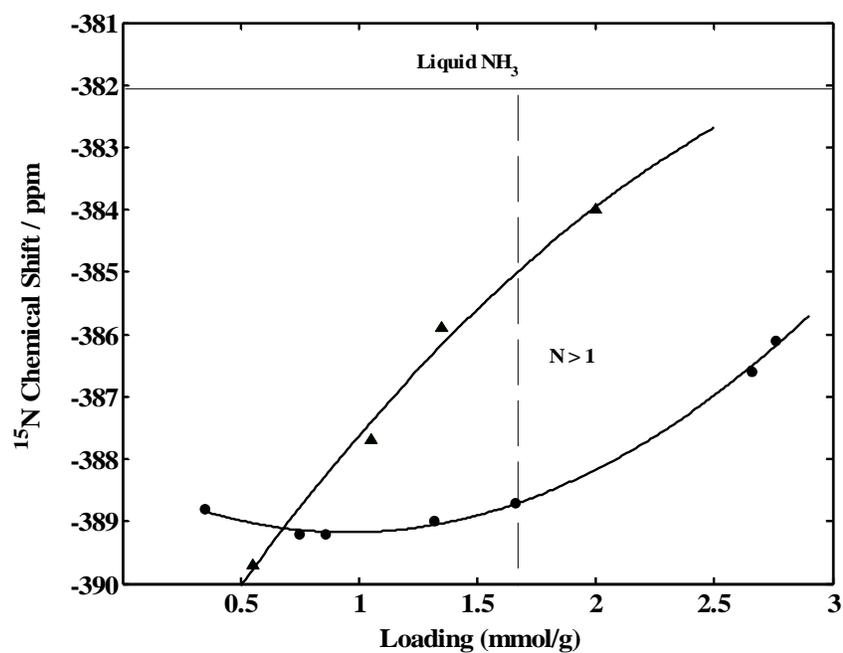


Figure 5: ^{15}N chemical shift of “free NH_3 ” as a function of adsorbed NH_3 for the molded (▲) and native (●) 3A Davison zeolite. N denotes the number of NH_3 molecules per large cage.

Conclusions

It has been demonstrate that high-resolution ^{15}N NMR can be used to elucidate multiple NH_3 adsorption environments in zeolite desiccants. These ^{15}N MAS NMR experiments show that there are 5 or 6 distinct adsorption sites present in the desiccant materials. However, site populations are dominated by two primary environments. These sites are assigned based on chemical shift, CP behavior, and T_1 relaxation times to “bound NH_3 ” and “free NH_3 ” environments in the zeolite cage. The bound environment is thought to be associated with the cations in the cage. These NMR studies also show that there are some significant differences between the pure and silicone-molded forms of the desiccant that possibly indicate that not all the desiccant embedded in the silicone mold are active for guest adsorption.

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