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Short Communication

Adsorption of nitrosamines in acidic solution by zeolites

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Abstract

Adsorption of *N*-nitrosopyrrolidine (NPYR) by zeolite NaZSM-5, Hβ and NaZSM-11 in acidic solution with pH of 1 was investigated. The NPYR uptake process obeyed the first-rate Lagegren kinetic equation describing adsorption, and the equilibrium data were fitted to the Langmuir or Freundlich adsorption equation. Zeolite ZSM-5, Hβ and NaZSM-11 possessed a higher adsorption capacity than amorphous silica or ordered mesoporous siliceous SBA-15, implying their potential application for adsorption of nitrosamines in gastric juice to anti-cancer.

Keywords: *N*-nitrosopyrrolidine; Acidic solution; Adsorption; Zeolite; Anti-cancer

1. Introduction

Development of modern industry causes increasingly serious pollution in the environment where human live in, constituting a catastrophic health risk including cancer. Anti-cancer is thus one of the challenges faced scientists in 21st century in the realm of life science, and removal of carcinogen from environment is an important step. Nitrosamines are probably the most widespread carcinogens, existing in workplace, processed meats, cigarette smoke and beer, and even are produced in the stomach by reaction of secondary amines and nitrite (NO_2^-) both taken from foods (Hiramoto et al., 2001). A report of German scientists showed in 1976 that the uptake of nitrosamines for a person was to 700 ng everyday. In another study, the mean total nitrosamines levels were 0.7 or 1.3–1.4 μM for fasting gastric juice at pH 1.0–1.5 or pH 3.6–7.0, correlated with bacterial mutagenicity of juice (Pignateli et al., 1987). The occurrence of gastric cancer was mostly related to the nitrosamines in stomach, as reviewed in 1983 (Mirvish, 1983) and 1989 (Preston-Martin and Correa, 1989). Many carcinogenic agents like nitrosamines or their precursors enter human stomach through diet and drinking, no matter how carefully human treat their food. Environmental pollution makes this hidden trouble more serious, because of the contaminated food and atmosphere. However, although nitrosamines are well-known carcinogenic substances, they require metabolic activation before reaction with DNA to cause mutation and cancer. Therefore, it is possible and necessary to trap the nitrosamines in gastric juice provided a selective adsorbent material is employed. To seek this functional material, zeolites are considered as the best candidates.

Zeolites and molecular sieves have been employed in slow release drugs, enzyme mimetic drugs (Mitchell, 1991), anti-tumor drugs (Weiner, 1997), and additive in cigarette to remove carcinogenic agents like nitrosamines (Meier and Siegmann, 1999; Xu et al., 2002, 2003a,b). Apart from the trapping in gaseous phase,

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zeolites can also adsorb volatile nitrosamines like NDMA (*N*-nitrosodimethylamine) and NPYR (*N*-nitrosopyrrolidine) in organic or aqueous solutions (Zhu et al., 2001), which is beneficial to eliminate the nitrosamines in beer (Wang et al., 2001). Among these zeolites used NaY possesses the largest adsorption capacity in organic solution while ZSM-5 is the best adsorbent in aqueous solution. However, those aqueous experiments were done in neutral solution with pH of 7, so that their results cannot be simply applied to the gastric juice that appears acidic (Pignateli et al., 1987). Moreover, a lot of zeolites cannot be used in acidic solution because of their inherent feature. This prompts us to investigate the adsorbing characteristics of zeolites in acidic solution. We chose the pH = 1 hydrochloric acidic solution, similar to the gastric juice, as the experimental standard solution, and NPYR is selected as the adsorbate because it widely exists in environment such as beer and cigarette smoke as well as gastric juice, emphatically on the adsorption kinetics and isotherm properties of NPYR on ZSM-5 zeolites.

2. Experimental

NaZSM-5 (12.5) or NaZSM-5 (26), with Si/Al ratio of 12.5 or 26, NaY, NaA zeolites and silica were commercially available powders (Xu et al., 2003b; Zhu et al., 2001). NTY (Si/Al = 7) is a dealuminated NaY zeolite sample with a surface area of 860 $m^2 g^{-1}$. Sample Hβ (Si/Al = 14.5) with the surface area around 600 $m^2 g^{-1}$ was provided by BASF. Zeolite NaZSM-11 (Si/Al = 25) and mesoporous siliceous molecular sieve SBA-15 were prepared in our laboratory according literature (Fyfe et al., 1989; Zhao et al., 1998). Sample HZSM-5 was obtained by ion exchange with NH_4Cl solution from parent NaZSM-5 zeolite, and NPYR was purchased from Sigma (Xu et al., 2003b).

To check the stability of zeolite in acidic solution, all the samples were shaken in the hydrochloric acid solution with pH value of 1 at 30 °C for 3 h. The dunked samples were filtrated and dried at 100 °C for 5 h prior to examination of X-ray diffraction that performed on a D/max-γ A X diffractmeter using Cu Kα radiation.

Adsorption of NPYR by zeolite in acidic solution was studied using batch method (Zhu et al., 2001); distilled water and the hydrochloric acid were employed throughout the experiments. To investigate the adsorption of NPYR on different mass of zeolite in acidic solution, 10 mg, 20 mg, 50 mg, 100 and 200 mg NaZSM-5 (12.5) zeolites samples were added into the 25 ml solution with an initial concentration of 4.4×10^{-4} M in colored cone flasks. The mixtures were shaken in a thermostatic shaker waterbath at 20 °C for 1 h, and then centrifuged at 3000 rpm for 15 min to separate the solution and the solid. The concentrations of NPYR in acid solution were determined at different times (Xu et al., 2003b). For the adsorption of Hβ, ZSM-11, NTY zeolite, SBA-15 and silica samples, the amount of adsorbent was fixed to 100 mg.

In another experiments to examine the influence of NPYR content on the adsorption, the NPYR solution with an initial concentration of about 0.44, 0.88, 1.32, 1.56, 2.2 mM were used. 0.01 g HZSM-5 (12.5), NaZSM-5 (12.5) and NaZSM-5 (26) zeolites samples were added into the 25 ml NPYR solution with known concentration in colored cone flasks, and the test was performed in the similar procedure as mentioned above.

Series of experiments were undertaken to study the influences of initial concentration, mass and types of adsorbent for the kinetics feature of adsorption. When the impact of one specific parameter was determined, all the other parameters kept constant. The flasks were shaken for 1 h to reach the adsorption equilibrium, because NPYR adsorption usually finished within this period.

For these uptake curves, the distribution coefficient k_d was estimated: $k_d = rV/(100 - r)m$ k_d expresses the ratio of NPYR adsorbed in the solid over that in the liquid phase (Metaxas et al., 2003). It defines the selectivity of the adsorbents towards NPYR. Additionally, the kinetic data of these curves was treated with the following first-rate Lagegren equation (Namasivayam and Kadiverlu, 1997): $\log(q_e - q_{ads}) = 1/2.303 \times \log q_e - (K_{ads} \times t)$ where q_e and q_{ads} are the amount of total NPYR adsorbed per gram of adsorbent at the equilibrium and at any time t, respectively, and K_{ads} is the adsorption rate constant.

3. Results and discussion

3.1. The stability of samples in acidic solution

Fig. 1 shows the patterns of zeolite NaZSM-5 (12.5), Hβ, and NaZSM-11 as well as NTY before and after

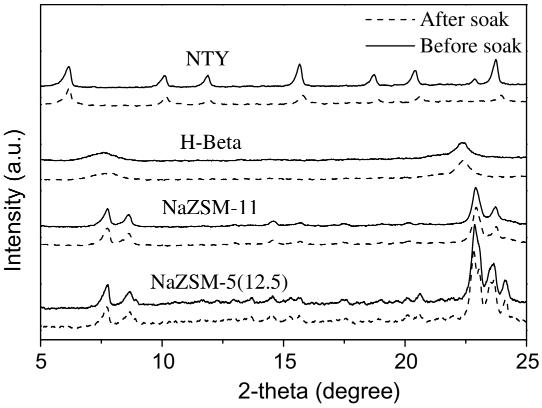

Fig. 1. XRD patterns of NaZSM-5 (12.5), NaZSM-11, Hβ and NTY before and after soaked in acidic solution for 3 h.

shaken in acidic solution at pH of 1, in which the characteristic XRD peaks of these zeolites can be clearly distinguished. Based on these results, it is very likely that NaZSM-5 (12.5), ZSM-11 and Hβ are stable in the acidic solution. The intensity of their major XRD peaks was almost invariable in comparison with the original samples. The peak intensity of NTY zeolites was obviously reduced after shaken in acidic solution, indicating the partially framework collapsing of NTY zeolites. The XRD results of NaZSM-5 (26) and HZSM-5 (12.5) were similar to that of NaZSM-5 (12.5) so their XRD results are omitted. Both amorphous silica and the ordered mesoporous SBA-15 are also stable in the acidic solution, hence these materials are also selected to carry out the succedent adsorption experiments. Unfortunately, both NaY and NaA zeolite were dissolved in the solution within 30 or 15 min and only a clear solution remained. Thus, they cannot be used in such acidic solution as the adsorbents.

The stability of NPYR in the acid solution was also examined. No degradation or volatilization was found after the compound dissolved in the solution for 3 h and centrifuged at 4000 rpm for 15 min. The determined concentration kept unchangeable, as illustrated in Fig. 2.

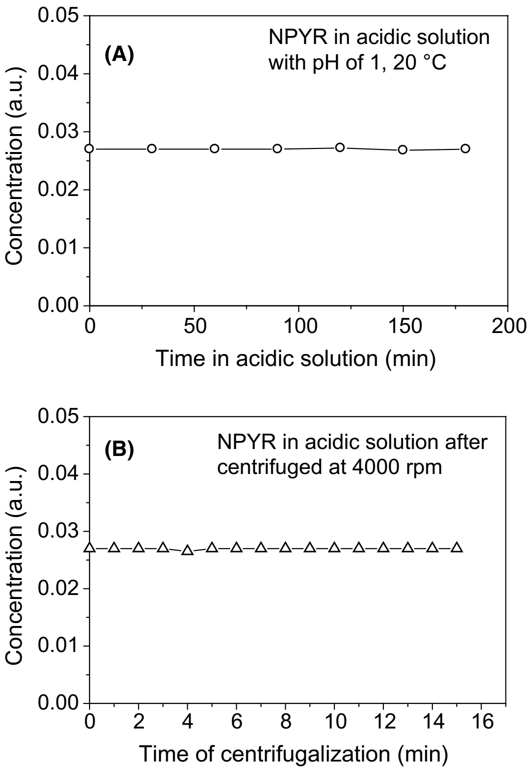

Fig. 2. Stability of NPYR in acidic solution and centrifugalization.

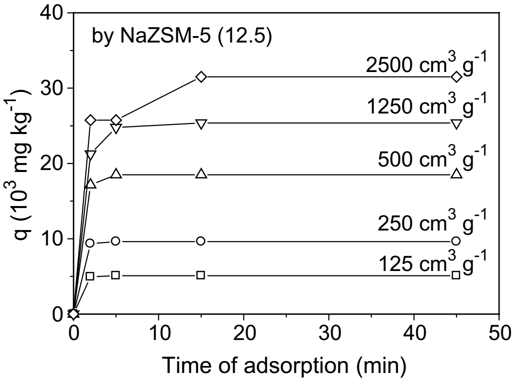

Fig. 3. Impact of V/m value on the adsorption of NPYR over zeolite NaZSM-5 (12.5) at 20 °C.

3.2. Adsorption kinetics

(a) Effect of adsorbent mass. Series of experiments were undertaken on NaZSM-5 (12.5). The variation of NPYR uptake, q, with time, t, during the non-equilibrium adsorption of different adsorbent mass is shown in Fig. 3. Adsorption rate of ZSM-5 zeolites was enough rapidly to reach saturation within 1 h, indicating the potential application of zeolites for removal of nitrosamines in the stomach. From the curves, k_d and K_{ads} were calculated by the front equation and presented in Table 1.

k_d depended on the ratio V/m and r value. Changes in the ratio of V/m affect the uptake efficiency r value and subsequently k_d value (Metaxas et al., 2003). From the results of Table 1, k_d expressed an apex value at the ratio liquid–solid-phase volumes of 500 varying of V/m value. Although the increase of zeolites mass made q_e value decreased, the percentage removal value was increased at the same time. K_{ads} value, which is a constant related to the V/m ratio, rose as the adsorbent mass increased. The smaller the V/m value, the higher the percentage removal r-value and K_{ads} value.

(b) Effects of initial NPYR concentration. NaZSM-5 (26) was used in experiments and the liquid–solid ratio was oriented at 2.5 cm^3 mg^{-1}. Fig. 4 illustrates the

Table 1
Distribution coefficients and Lagegren constants of NPYR adsorption by NaZSM-5 (12.5) with different values of V/m

V/m ($cm^3 g^{-1}$)	k_d ($cm^3 g^{-1}$)	r (%)	K_{ads} (min^{-1})
2500	0.99	22.4	0.039
1250	1.07	46.1	0.075
500	2.65	84.1	0.076
250	1.75	87.5	0.098
125	1.54	92.5	0.105

Adsorption conditions: $T = 20\,°C$, $C_0 = 44\,mg\,dm^{-3}$.

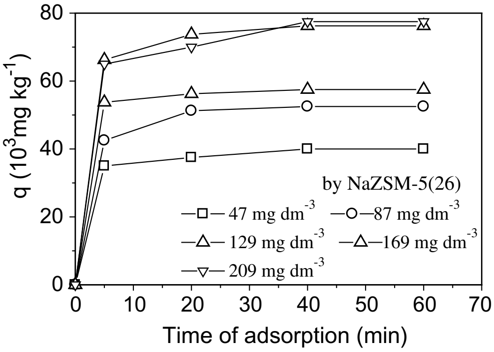

Fig. 4. Effect of initial concentration on the adsorption of NPYR by NaZSM-5 (26) in acidic solution at 30 °C.

Table 2
Distribution coefficients and Lagegren constants of NPYR adsorption by NaZSM-5 (26) with different initial concentration

C_0 (mg dm^{-3})	R (%)	k_d (cm^3 mg^{-1})	K_{ads} (min^{-1})
47	34.0	1.29	0.030
87	24.1	0.79	0.041
129	17.8	0.54	0.042
169	18.8	0.55	0.037
209	14.8	0.43	0.025

Adsorption conditions: $T = 30$ °C, $V/m = 2.5$ cm^3 mg^{-1}.

variation of NPYR uptake, q, with time, t, during the non-equilibrium adsorption of different initial concentration. From the curves, k_d and K_{ads} were calculated by the front equation and presented in Table 2.

The NPYR uptake process obeyed the first-rate Lagegren equation describing adsorption with all $R > 0.95$ correspondingly for solutions containing different initial NPYR concentration. K_{ads} was related to the initial NPYR concentration in acid solution. k_d value decreased with increasing the initial NPYR concentration. At the same time, r value also decreased with increasing the initial NPYR concentration. The changes of maximum adsorption amount at the different NPYR initial concentration was shown in Fig. 4.

(c) Effects of Si/Al ratio and ion of zeolites. The adsorption amount of all sample rose with the initial NPYR concentration; especially it increased with the ratio Si/Al of NaZSM-5 zeolites raised from 12.5 to 26. The Al content in the zeolite governing the polarity of internal voids and the polarity of pore was highly relevant to the hydrophilicity or hydrophobocity of zeolites (Camblor et al., 1996). NPYR was hydrophobic though the solution for adsorption was aqueous. Since the NaZSM-5 zeolites with a high Si/Al ratio were also hydrophobic, it firstly adsorbed the hydrophobic NPYR in acidic aqueous solution. Accordingly, the equilibrium adsorption amount of NaZSM-5 (12.5) was 70×10^3 mg kg^{-1}, and less than that of the NaZSM-5 (26) on which the corresponding value reached 77.5×10^3 mg kg^{-1}.

ZSM-5 zeolites with the same Si/Al ratio but different type of ion presented the same equilibrium adsorption amount and their maximum uptakes both were 70×10^3 mg kg^{-1} when the V/m value was equal to 2.5 cm^3 mg^{-1}. Metal ion of ZSM-5 zeolite seems not play a determinative role in the adsorption of volatile nitrosamines in acid solution. One reason for this phenomenon is the acidic aqueous solution, in which Na^+–H^+ ion exchange of zeolite may occur, making the similar adsorptive capacity appeared on NaZSM-5 and HZSM-5 samples.

3.3. Adsorption isotherms

As seen in Fig. 5, all ZSM-5 zeolites can selectively adsorb NPYR in acidic solution, and NaZSM-5 (26) sample exhibits the largest capability. All adsorption isotherms in Fig. 5 exhibit an initial steep slope followed a gentle upward growth and finally arrive to saturation trend. For an overall analysis of adsorption isotherm, we try to correlate the experimental equilibrium data with Langmuir or Freundlich equation that in its linear form $\ln q = \ln k_F + 1/n \times \ln C_e$ where $1/n$ is an exponent relate to adsorptive strength as well as the favorability (Slejko, 1985). The adsorption data were fitted to Freundlich equation at first and Table 3 lists the isotherm parameters calculated with the method of least squares. For all the investigation systems the exponent of $1 < n < 10$ indicates the favorable nature of adsorbent-NPYR system. The smallest value of n, meaning a strong affinity existed between zeolites and NPYR, was observed on HZSM-5 (12.5) sample while the affinity between NaZSM-5 (26) and NPYR seemed weaker. The zeolites with high Si/Al ratio contain the fewer ions in channels, forming the weaker electrostatic fields in the pores. NPYR is a polar compound, thus the affinity be-

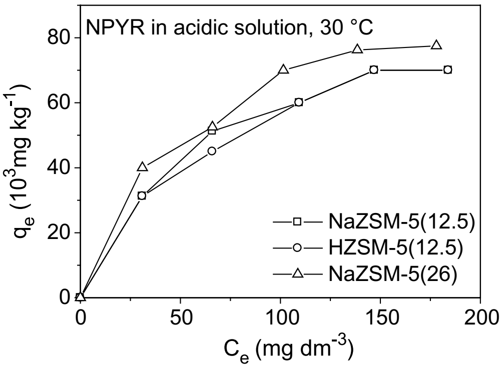

Fig. 5. Adsorption isotherm of NPYR on zeolite in acidic solution with pH of 1 at 30 °C.

Table 3
Regression equations of $\log q_e$ vs. $\mathrm{Log}\, C_e$ for Langmuir and Freundlich isotherms conditions: 30 °C, in acidic solution

Samples	Freundlich equation			Langmuir equation		
	k_F	n	R	k_L	$q_m (\times 10^3\,\mathrm{mg\,kg^{-1}})$	R
NaZSM-5 (26)	9.92	2.45	0.98	0.020	102.0	0.990
NaZSM-5 (12.5)	6.80	2.17	0.98	0.017	94.3	1
HZSM-5 (12.5)	6.1	2.08	0.99	0.014	99.0	0.99

tween it and NaZSM-5 (26) is less than that with NaZSM-5 (12.5). Both affinity for nitrosamines and hydrophobocity of zeolite are the important factors beneficial for the selective adsorption of volatile nitrosamines in acidic solution, the former provides an electronic inducing force to attract the adsorbate while the latter prevents from the competitive adsorption of water solvent. However, some times they are incompatible. Low Si/Al ratio is helpful to enhance the affinity but makes the zeolite adsorbent hydrophilic, so the influence of aluminum component in zeolite ZSM-5 on the adsorption is complex.

Intensity of electrostatic fields in zeolite increased with the decrease in cation size (Rabo et al., 1966). Since diameter of proton was smaller than that of sodium ion, the electrostatic intensity in HZSM-5 would be greater than that of NaZSM-5 with the same Si/Al ratio. Thus, the n value of HZSM-5 with a ratio Si/Al of 12.5 was the largest. As listed in Table 3, all *R*-values, correlation coefficients, exceed 0.98, implying that these adsorption curves obeyed the Freundlich adsorption law.

For these curves showing a clear saturation tendency in higher concentration, the applicability of the Langmuir equation $q = q_m k_L C/(1 + k_L C)$ was tested, where constant k_L and the saturation capability q_m can be evaluated by the regression analysis of the linear form of the foregoing equation. In particular, the linear form of Langmuir equation was expressed as the followed: $C/q = C/q_m + 1/(q_m \times k_L)$.

The isotherm parameters for Langmuir model were calculated by use of least squares method and the results are also listed in Table 3, in which all correlation coefficients (*R*) for Langmuir equation exceed 0.99. That is to say, these adsorption isotherms were more suitable to be fitted to Langmuir equation that can help us to evaluate the interaction energy and to predicate the saturation value of each zeolite. The q_m values indicated the amount of NPYR to form a monolayer over adsorbents. For zeolite NaZSM-5 (26) this value was $102 \times 10^3\,\mathrm{mg\,kg^{-1}}$, but the real uptake was $77.5 \times 10^3\,\mathrm{mg\,kg^{-1}}$, probably due to the geometric limitation in adsorption of NPYR provided by the pore size of ZSM-5 that is closely matched to the molecular diameter of NPYR (Zhu et al., 2001). However, the calculated data has a similar tendency to the experimental data. For instance the calculated uptake of NaZSM-5 (26) and NaZSM-5 (12.5) is 102 and $94 \times 10^3\,\mathrm{mg\,kg^{-1}}$, in fact the former adsorbed indeed more NPYR ($77.5 \times 10^3\,\mathrm{mg\,kg^{-1}}$) than the latter ($70 \times 10^3\,\mathrm{mg\,kg^{-1}}$).

3.4. Adsorption capacity of other porous materials

The adsorption capacity of other porous materials was presented in Fig. 6, among them Hβ zeolites had the largest value and its uptake was $55 \times 10^3\,\mathrm{mg\,kg^{-1}}$, originating from its big pore size and surface area. ZSM-11 has the pore aperture similar to that of ZSM-5 but the channel structure is straight instead of "Z" type, its equilibrium adsorptive amount reaches $42.5 \times 10^3\,\mathrm{mg\,kg^{-1}}$, similar to that of NaZSM-5 (26) ($40 \times 10^3\,\mathrm{mg\,kg^{-1}}$). So, the channel camber is not crucial for the adsorption of volatile nitrosamines in acidic solution. Although NTY zeolites possesses a larger surface area ($860\,\mathrm{m^2\,g^{-1}}$) than ZSM-5 or β zeolite, its equilibrium adsorptive amount was only $15 \times 10^3\,\mathrm{mg\,kg^{-1}}$, resulting from the partially collapse of structure that blocks channel and the competitive adsorption of water solvent (Zhu et al., 2001). Besides, dealumination caused many mesoporous pores to form in Y zeolite, which was not beneficial for the adsorption of volatile nitrosamines because mesoporous MCM-48 and SBA-15 were inferior to zeolite for adsorption of NDMA or NPYR (Zhu et al., 2003; Xu et al., 2003a).

Silica had further smaller adsorptive capacity for nitrosamines in acidic solution and its equilibrium uptake was only $6.3 \times 10^3\,\mathrm{mg\,kg^{-1}}$, though its surface area is close to that of NaZSM-5 (26). Mesoporous

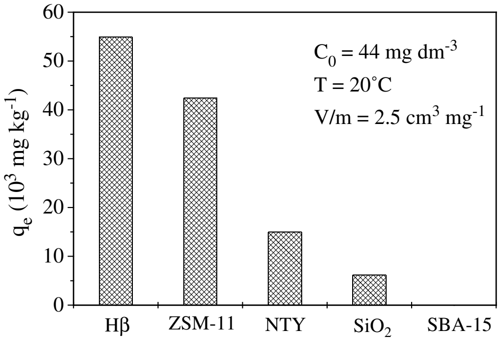

Fig. 6. Amount of NPYR adsorbed on different porous materials in acidic solution at 20 °C.

SBA-15 did not adsorb NPYR in the acidic solution at all (Fig. 6). The huge pore size of SBA-15 was impossible to provide the suitable adsorbent–adsorbate interaction to trap those nitrosamines with small molecular size like NPYR. Additionally, siliceous SBA-15 has no ion in its channels so it cannot possess any electrostatic fields like that of zeolites to attack the adsorbate.

4. Conclusion

1. ZSM-5 zeolites can adsorb NPYR in the acid solution with pH of 1. Higher Si/Al ratio is beneficial for the adsorption. HZSM-5 and NaZSM-5 zeolites with the same Si/Al ratio possessed the same adsorptive capacity.
2. The NPYR uptake process in acidic solution obeys the first-rate Lagegren kinetic equation, while the isotherms on ZSM-5 were preferably obeys the Langmuir equation rather than Freundlich equation.
3. Hβ zeolite has the highest adsorption capacity other than ZSM-5, and zeolites are superior to silica and siliceous SBA-15 for the adsorption of nitrosamines in acidic solution.

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