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Metal loaded zeolite adsorbents for hydrogen cyanide removal

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Abstract

Metal (Cu, Co, or Zn) loaded ZSM-5 and Y zeolite adsorbents were prepared for the adsorption of hydrogen cyanide (HCN) toxic gas. The results showed that the HCN breakthrough capacity was enhanced significantly when zeolites were loaded with Cu. The physical and chemical properties of the adsorbents that influence the HCN adsorption capacity were analyzed. The maximal HCN breakthrough capacities were about the same for both zeolites at 2.2 mol of HCN/mol of Cu. The Cu2p XPS spectra showed that the possible species present were Cu2O and CuO. The N1s XPS data and FT-IR spectra indicated that CN would be formed in the presence of Cu+/Cu2+ and oxygen gas, and the reaction product could be adsorbed onto Cu/ZSM-5 zeolite more easily than HCN.

Key words: hydrogen cyanide; zeolite; removal

DOI: 10.1016/S1001-0742(12)60138-7

Introduction

Hydrogen cyanide (HCN) is a colorless, very volatile and very poisonous liquid, which boils at 26°C (Seredych et al., 2009). It is commonly released to the air from various sources including vehicle exhaust emissions (Baum et al., 2007; Karlsson, 2004), chemical processing, coal gasification (Chen et al., 2009; Lin et al., 2010), and petroleum refineries (Oliver et al., 2005; Kotdawala et al., 2008). Furthermore it is a major precursor of N2O, which is an important greenhouse gas with a long atmospheric lifetime (120 ± 30 years) (Dagaut et al., 2008). Therefore it can easily induce several health and environmental hazards (Dagaut et al., 2008; Yang et al., 2011).

Many strategies have been developed to remove HCN from gas, including catalytic hydrolysis (Kröcher and Elsener, 2009), oxidation (Dagaut et al., 2008; Giménez-Lpez et al., 2009; Zhao et al., 2006) and adsorption (Seredych et al., 2009; Oliver et al., 2005; Nickolov and Mehandjiev, 2004; Barnes et al., 2002; Ye et al., 2009). Catalytic oxidation of HCN is an alternative approach, and the process typically operates at 150°C or higher; N2, N2O, NO, NO2, CO2 and H2O were found as reaction products (Giménez-Lpez et al., 2009; Zhao et al., 2006). Hydrolysis is one of the possible approaches to remove HCN from gas; HCN is usually removed at temperatures in the range of 200–500°C, and NH3 and CO are formed during catalytic hydrolysis (Kröcher and Elsener, 2009). Direct removal of HCN is a particularly attractive method nowadays. Different types of removal materials have been investigated in recent years. Hudson et al. (2004) reported that Cu-BBOP-SBA-15 had an improved breakthrough time for HCN (36 min) at 22°C and 80% relative humidity. Seredych et al. (2009) reported that when BPL carbon was tested as an HCN adsorbent, the adsorbed amount in dry conditions at room temperature was only 0.5 mg/g. The adsorption capacity of the activated carbon can be enhanced through additional modification; for example, impregnation with metal oxides (e.g., Cr, Cu, Ag, Mo, Zn, Co) (Seredych et al., 2009; Nickolov and Mehandjiev, 2004; Barnes et al., 2002; Ye et al., 2009). Unfortunately, some shortcomings with present activated carbons have been identified, including thermal instability (Hudson et al., 2004). On the other hand, zeolite materials are characterized by their high surface area, large pore volume, and high thermal stability (Li et al., 2008). However, research on the possibility of abating HCN by zeolite dry adsorption has never been studied.

In this study, two types of zeolite, ZSM-5 and Y zeolites, were loaded with metals (Cu, Co, or Zn) to act as chemical adsorbents, and their performance in removing HCN from a gas stream were compared. The correlation between the physical/chemical properties of the adsorbents and their adsorption behaviors were discussed. The performance of modified zeolites was evaluated by HCN breakthrough data. Nitrogen adsorption at 77.350 K was used to char-
acterize the structure of the adsorbents, including pore volume and surface area. The surface chemical structures of the Cu/ZSM-5 zeolites were analyzed with X-ray photoelectron spectroscopy (XPS) and Fourier transform infrared spectroscopy (FT-IR).

1 Materials and methods

1.1 Adsorbent preparation

Two commercial zeolites, ZSM-5 and Y zeolites (99.99%, Nankai University Catalyst Co., Ltd., Tianjin, China), were used as support materials for the metal adsorbents. Table I compares the physical and chemical properties of these two zeolites prior to the metal loading process. It was observed that Y zeolite has a higher BET surface and pore volume than the ZSM-5 zeolite. Moreover, ZSM-5 is a high silica zeolite with a SiO$_2$/Al$_2$O$_3$ molar ratio of 50 as provided by the manufacturer, while the Y zeolite has a SiO$_2$/Al$_2$O$_3$ molar ratio of only 5.2 (Li et al., 2008; Weitkamp, 2000).

A total of 10 g of each type of zeolite and 100 mL of metal precursor solution, either Cu(NO$_3$)$_2$·3H$_2$O, Co(NO$_3$)$_2$·6H$_2$O, or Zn(NO$_3$)$_2$·6H$_2$O, were stirred individually at room temperature for 12 hr. After filtration and washing, the adsorbents were dried in an oven at 120°C for 12 hr and then calcined in a furnace at 550°C for 6 hr. The amount of metal loaded on the zeolite was varied by using solutions with different concentrations of metal precursor, and the actual mass of metal loaded on the zeolite was determined by atomic adsorption spectrometry (spectrAA220FS Varian Inc.).

1.2 Adsorbent characterization

A multi-spot nitrogen adsorption meter (NOVA2000e, Quantachrome Corp.) was used to determine nitrogen adsorption isotherms under 77.350 K. Photoelectron spectra were obtained using X-ray photoelectron spectroscopy (XPS) analyses, which were carried out using a Physical Electronics PHI5600 spectrometer. FT-IR measurements were performed on a Thermo Nicolet AVATAR FT-IR 360 instrument.

1.3 HCN breakthrough measurements

The model gas flow was composed of nitrogen with 100 ppm HCN; they were mixed in the mixer evenly with micro-oxygen and introduced into the adsorption bed unit. HCN breakthrough curves for the HCN removal were assessed in dynamic tests at 30°C with an oxygen content of 1.0%. Each sample (5.0 g) was packed in a quartz column and was exposed to gas containing 100 ppm HCN at a flow rate of 250 mL/min. The concentration of HCN was analyzed using a spectrophotometric method, permitting the detection of HCN as low as 0.0050 mg/m$^3$. HCN was absorbed readily in sodium hydroxide to avoid safety problems. The “breakthrough time” was determined to be the point at which the HCN removal efficiency dropped to below 90%. The HCN adsorption capacity was calculated with the corresponding integral according to following equation under various conditions of the breakthrough curves (Wang et al., 2009).

$$X = \frac{Q_{0}t - \int_{0}^{t} C dt}{m}$$

where, $X$ (mg/g) is the adsorption capacity, $Q$ (m$^3$/min) is the gas flow, $t$ (min) is the adsorption time, $C_0$ (mg/m$^3$) and $C$ (mg/m$^3$) are the adsorption column entrance and outlet mass concentrations, and $m$ (g) is the adsorbent weight.

1.4 Measurements of HCN

A Shimadzu UV-240 UV-Vis recording spectrophotometer was used with 10-mm cells. HCN was measured with the iso-nicotinic acid-3-methyl-1-phenyl-5-pyrazolone spectrophotometric method at 638 nm. Colorimetry was based on the conversion of HCN absorbed by sodium hydroxide to cyanogen chloride with an aqueous solution of chloramine-T. Reaction of the cyanogen chloride with pyridine and then with a 3-methyl-1-phenyl-5-pyrazolone reagent results in a colored complex, which was analyzed by a spectrophotometer (Jiang et al., 1999; Zhang et al., 2011; Motier et al., 2010).

2 Results and discussion

2.1 Effects of modifiers on HCN adsorption

Three metal precursors, Cu(NO$_3$)$_2$·3H$_2$O, Co(NO$_3$)$_2$·6H$_2$O, and Zn(NO$_3$)$_2$·6H$_2$O, with a concentration of 0.5 mol/L, were evaluated for their HCN adsorption capacities on the zeolite adsorbents, and the results are shown in Fig. 1 for ZSM-5 zeolite. One can see that the HCN removal efficiency for the Cu-loaded zeolite adsorbent was much higher than those of Zn- and Co-loaded zeolites (Nickolov and Mehandjiev, 2004; Barnes et al., 2002). Therefore, Cu was chosen as the metal species for later study. The breakthrough capacities for Cu/ZSM-5, Zn/ZSM-5, Co/ZSM-5 were 1.979, 0.662, 0.2070, 0.3996.

<table>
<thead>
<tr>
<th>Zeolite type</th>
<th>Pellet form</th>
<th>SiO$_2$/Al$_2$O$_3$ (mol ratio)</th>
<th>Cation form</th>
<th>BET surface area (m$^2$/g)</th>
<th>Pore size (nm)</th>
<th>Pore volume (mL/g)</th>
</tr>
</thead>
<tbody>
<tr>
<td>ZSM-5</td>
<td>Strip</td>
<td>50</td>
<td>H$^+$</td>
<td>245.4</td>
<td>1.687</td>
<td>0.2070</td>
</tr>
<tr>
<td>Y</td>
<td>Strip</td>
<td>5.2</td>
<td>H$^+$</td>
<td>437.4</td>
<td>1.685</td>
<td>0.3996</td>
</tr>
</tbody>
</table>

Table 1  Physical and chemical properties of zeolites used in this study
0.5842 mg HCN/g adsorbent, respectively.

2.2 Characterization of Cu/zeolite adsorbents

Copper nitrate solutions of various concentrations ranging from 0.1 to 1.5 mol/L were used to obtain Cu/zeolite adsorbents with various metal loading amounts. The relationships between the precursor concentration of Cu(NO$_3$)$_2$ and the actual loading amounts of Cu metal for both ZSM-5 and Y zeolite adsorbents are shown in Fig. 2.

It was observed that, as the Cu(NO$_3$)$_2$ precursor concentration was increased, the rate of increase of the actual Cu content loaded on the Y zeolite was much higher than that on the ZSM-5 zeolite. The maximum Cu loadings on Y and ZSM-5 zeolites were 6.306% (w/w) and 3.924% (w/w), respectively, with a Cu(NO$_3$)$_2$ precursor concentration of 1.5 mol/L. This may be due to both the relatively higher Al content of Y zeolite available for Cu ion exchange and its higher BET surface area (pore volume), which provide more impregnation sites for Cu.

The surface area and pore volume of ZSM-5 and Y zeolite adsorbents before and after modification were calculated from N$_2$ adsorption-desorption isotherms. The variations in the BET surface area and pore volume of both Cu/ZSM-5 and Cu/Y zeolite adsorbents with respect to the precursor concentration are shown in Fig. 3. One can see that the variations of the BET specific surface area ($S_{BET}$) for both Cu/ZSM-5 zeolite and Cu/Y zeolite adsorbents follow trends similar to the variations of their pore volumes ($V_{total}$). The BET specific surface area and pore volume of Cu/Y zeolite adsorbents decreased from 456.5 to 371.9 m$^2$/g and from 0.3996 to 0.3503 mL/g respectively when the actual Cu loading concentration was increased from 0 to 6.306%. For Cu/ZSM-5 zeolite adsorbents, it was interesting that the BET specific surface area decreased from 258.8 to 243.1 m$^2$/g and the pore volume increased from 0.2232 to 0.2131 mL/g when the actual Cu loading concentration was increased from 0 to 3.924%.

2.3 Adsorption capacity

The effective adsorption capacities for achieving over 90% HCN removal with Cu/ZSM-5 and Cu/Y zeolite adsorbents are shown in Fig. 4 as a function of actual Cu loading mass concentration. As observed, the adsorption capacity of Cu/ZSM-5 zeolite adsorbent increased to 3.851 mg HCN/g adsorbent (mg/gads) as the Cu loading concentration is increased to 3.924% (w/w). Similarly, the adsorption capacity of Cu/Y zeolite adsorbent increased with increasing Cu loading and reached its maximum value of 5.056 mg/gads at a Cu loading concentration of 5.498% (w/w); then it decreased as the Cu loaded concentration was further increased. The maximum adsorption capacities were about the same for both zeolites at 2.2 mol HCN/mol Cu. In comparison with the HCN capacity of Cu-modified activated carbon, the Cu/ZSM-5 and Cu/Y zeolite provided comparable HCN capacity (Brown et al., 1989). The maximum adsorption capacity per metal mass loaded obtained in this study was much higher than literature data using CuO for adsorbing hydride gases. Previous publication reported that a maximum adsorption capacity of only 0.15
Table 2  Elemental analysis of Cu/ZSM-5 before and after adsorption using XPS

<table>
<thead>
<tr>
<th>Sample</th>
<th>Before adsorption</th>
<th>After adsorption</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>C1s</td>
<td>O1s</td>
</tr>
<tr>
<td>Area (cts-eV/s)</td>
<td>5014</td>
<td>34822</td>
</tr>
<tr>
<td>Sensitivity factor</td>
<td>16.52</td>
<td>39.89</td>
</tr>
<tr>
<td>Concentration (%)</td>
<td>25.57</td>
<td>73.53</td>
</tr>
</tbody>
</table>

The Cu/ZSM-5 zeolite was prepared using 1.5 mol/L Cu(NO₃)₂ precursor concentration.

HCN adsorption capacities of Cu/ZSM-5 and Y zeolite adsorbents are shown in Fig. 4 by their percentages relative to maximal values, i.e., 258.8 m²/g and 3.851 mg/g_ads, respectively, for specific surface area and adsorption capacity. The BET surface area represents the physical adsorption properties of the adsorbent, while the actual Cu loading amount is the major chemical adsorption factor. It is seen clearly from Fig. 4 that the specific surface area of the zeolite decreased slightly; however, the adsorption efficiency increased as the Cu loading was increased. Thus the HCN adsorption is governed by the chemical factor of Cu content for Cu/ZSM-5 zeolite.

The percentages relative to the maximum of BET surface area and effective adsorption capacity (i.e., 456.5 m²/g and 5.056 mg/g_ads) as functions of Cu loading are shown in Fig. 5a for the Cu/ZSM-5 zeolite adsorbent. The adsorption capacity increased with increasing Cu loading mass, while the specific surface area decreased to about 81% of its maximal value at Cu loading of 6.306% (w/w). However, as the Cu loading continuously increased to beyond 5.498% (w/w), the HCN adsorption capacity decreased significantly. This indicates that the decrease of HCN adsorption efficiency of Y zeolite at higher Cu loading is mainly due to the loss of its specific surface area. Below 5.498% (w/w) of actual Cu loading, the chemical property (Cu content) dominates the available adsorption sites, and beyond that the physical property (specific surface area or pore volume) is the major factor responsible for the HCN adsorption capacity.

Fig. 5  Variations in percentages relative to maximal values of HCN adsorption capacity and specific surface area for Cu/ZSM-5 zeolite (a) and Cu/Y zeolite (b) adsorbents as a function of Cu loading content (% by weight).
2.5 X-ray photoelectron spectroscopic analysis

The chemical states of the elements on the Cu/ZSM-5 adsorbent before and after adsorption were examined by XPS. Table 2 shows the major surface element concentrations of Cu/ZSM-5 before and after adsorption obtained by XPS analysis. The concentrations of C, O and Cu on the Cu/ZSM-5 before adsorption were 25.57%, 73.53% and 0.91%, respectively. The concentrations of C, O, Cu and N on the Cu/ZSM-5 after adsorption were 15.48%, 82.33%, 0.85% and 1.35%, respectively. Freshly prepared Cu/ZSM-5 adsorbent had no N species. After adsorption, N was observed. The increase of N content after adsorption should be ascribed to the chemical fixation of Cu(NO$_3$)$_2$ groups onto the surface of ZSM-5 and therefore confirms the occurrence of chemical reactions between HCN and the surface functional groups of ZSM-5.

Figure 6 shows the XPS spectra of the Cu2p core level binding energy before adsorption and after adsorption/oxidation. It has been reported that the copper impregnated onto samples is mainly amorphous copper oxide (CuO). After aging treatment, the amorphous copper oxide may reduce to Cu$_2$O or form crystallized CuO (Ehrburger et al., 1986; Huang et al., 2006; Yi et al., 2011). The major Cu 2p3/2 peaks centered at 932.52 and 932.74 eV, with the presence a small satellite peaks at 951.61 and 952.74 eV, were assigned to Cu$_2$O. In addition, the other Cu 2p3/2 peaks centered at 934.42 and 934.71 eV with the presence of small satellite peaks at 954.74 and 956.01 eV were assigned to CuO.

XPS data from the Cu 2p spectra and the possible Cu species and relative percentages are shown in Table 3. The relative percentage of Cu$_2$O was 66.62%, and the ratio of CuO was 33.38%. After adsorption, it was observed that the relative percentage of Cu$_2$O was 71.74%, and the ratio of CuO was 28.26%; the ratio of Cu$_2$O was higher than that before adsorption.

Figure 7 shows the relevant detailed N1s XPS spectra of the used Cu/ZSM-5 zeolite. As shown in Fig. 7, the peaks in the curve can be fit with N1s signals at 397.54, 399.67 and 401.50 eV, and show the presence of CN$^-$ ions on the adsorbent surface. It is known that the copper added to the adsorbent, reacts chemically with HCN with the formation of a precipitate. The removal of HCN can be attributed mainly to chemical interaction with CuO and Cu$_2$O in the active phase.

2.6 FT-IR analysis results

FT-IR provides information on the chemical structure of the adsorbent material. The FT-IR spectra (between 400 and 4000 cm$^{-1}$) of the zeolite adsorbents before and after adsorption are shown in Fig. 8. We are able to identify twelve bands at about 449, 545, 796, 1100, 1225, 1633, 1714, 1875, 2179, 2853, 2920, and 3454 cm$^{-1}$

A wide absorption band at 3200–3600 cm$^{-1}$ with a maximum at 3445–3450 cm$^{-1}$ can be assigned to the O–H stretching mode of hydroxyl groups and adsorbed water
(Yi et al., 2011; Puziy et al., 2002). Another broad peak within the 700–1200 cm\(^{-1}\) region with a maximum at about 1100 cm\(^{-1}\) and a weak shoulder peak appearing at about 1225 cm\(^{-1}\) can be assigned to C–O vibrations in alcohol groups (Shenderova et al., 2011).

A weak adsorption band at about 2800–3000 cm\(^{-1}\) may be ascribed to C–H stretching vibrations. The three weak absorption bands at about \(\nu = 1633.4\) and 1714.0 cm\(^{-1}\) are characteristic of carbonyls in ketones, carboxylic acids, acid anhydrides, esters and lactones (Yi et al., 2011; Shenderova et al., 2011; Maillard et al., 2011).

When the ZSM-5 zeolite adsorbents adsorb HCN, the bands that appear at 1875.5 and 2179.4 cm\(^{-1}\) are characteristic of the stretching vibration of C=O and C≡N. Nanba et al. (2007) stated that the band appearing at 2240 cm\(^{-1}\) has been assigned to the stretching vibration of C≡N in various nitrile compounds. Poignant et al. (2001) reported that the bands correspond to an acrylonitrile species coordinated to Cu\(^{2+}\) ions and are characterized by a \(\nu(C≡N)\) band at 2279 cm\(^{-1}\). Woo et al. (2011) reported that the FT-IR spectra of the SCN\(^{-}\) stretching mode had strong intensity and thus was a good candidate to study the effect of ion association; FT-IR bands of CN stretching in the NH\(_2\)SCN system appear in the region 2030–2090 cm\(^{-1}\). It can be concluded that the FT-IR absorption band difference between the fresh Cu/ZSM-5 zeolite and the exhausted Cu/ZSM-5 zeolite shows that CN\(^{-}\) would be formed in the presence of Cu\(^{2+}\)/Cu\(^{2+}\) and oxygen gas, and the reaction product could be adsorbed onto Cu/ZSM-5 zeolite more easily than HCN.

### 3 Conclusions

Zeolite adsorbents modified by impregnation, especially by Cu(NO\(_3\))\(_2\) impregnation, have exhibited significantly enhanced adsorption purification ability. The specific surface area had a minimal effect on the HCN adsorption when Cu/ZSM-5 zeolite was used, but it could govern the HCN adsorption when Cu/Y zeolite was used. The maximal HCN breakthrough capacities were about the same for both zeolites at 2.2 mol HCN/mol Cu.

According to XPS data, freshly prepared Cu/ZSM-5 adsorbent had no N species, while after adsorption, a N species was observed, and the increase of N content after adsorption indicated the occurrence of chemical reactions between HCN and the surface functional groups of ZSM-5. The N1s XPS data and FT-IR spectra showed that CN\(^{-}\) would be formed in the presence of Cu\(^{2+}\)/Cu\(^{2+}\) and oxygen gas, and the reaction product could be adsorbed onto Cu/ZSM-5 zeolite more easily than HCN.

### Acknowledgments

This work was supported by the National Natural Science Foundation of China (No. U1137603, 51268021), the Hi-Tech Research and Development Program (863) of China (No. 2012AA062504) and the Applied Basic Research Program of Yunnan (No. 2011FB027, 2011FA010).

### References


### Table 3 Cu2p XPS results and possible Cu species before and after HCN adsorption

<table>
<thead>
<tr>
<th>Cu species</th>
<th>Before adsorption</th>
<th>After adsorption</th>
</tr>
</thead>
<tbody>
<tr>
<td>Binding energy of Cu2p3/2 (eV)</td>
<td>932.52</td>
<td>934.42</td>
</tr>
<tr>
<td>Binding energy of Cu2p1/2 (eV)</td>
<td>951.61</td>
<td>954.74</td>
</tr>
<tr>
<td>Fwhm (eV)</td>
<td>2.60</td>
<td>2.80</td>
</tr>
<tr>
<td>Calculated Cu percentage (%)</td>
<td>66.62</td>
<td>33.38</td>
</tr>
<tr>
<td>CuO</td>
<td>932.74</td>
<td>934.71</td>
</tr>
<tr>
<td>Cu</td>
<td>952.78</td>
<td>956.01</td>
</tr>
</tbody>
</table>

The Cu/ZSM-5 zeolite was prepared using 1.5 mol/L Cu(NO\(_3\))\(_2\) precursor concentration.


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