Copper Loaded on Sol-Gel-Derived Alumina Adsorbents for Phosphine Removal

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Published online: 24 Jan 2012.

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ABSTRACT

The hydride gas of phosphine (PH_3) is commonly used for semiconductor and optoelectronic industries. The local scrubbers must immediately abate it because of its high toxicity. In this study, copper (Cu) loaded on the sol-gel-derived γ-alumina (Al_2O_3) adsorbents are prepared and tested to investigate the possibility of PH_3 removal and sorbent regeneration. Test results showed that during the breakthrough time of over 99% PH_3 removal efficiency, the maximum adsorption capacity of Cu loaded on the sol-gel-derived γ-Al_2O_3 adsorbent is 18 mg-PH_3/g-adsorbent. This is much higher than that of Cu loaded on the commercial γ-Al_2O_3 adsorbent—8.6 mg-PH_3/g-adsorbent. The high specific surface area, narrow pore size distribution, and well dispersion of Cu loaded on the sol-gel-derived γ-Al_2O_3 could be the reasons for its high PH_3 adsorption capacity. The regeneration test shows that Cu loaded on the sol-gel-derived γ-Al_2O_3 adsorbent can be regenerated after a simple air purging procedure. The cumulative adsorption capacity for five

regeneration cycles is 65 mg-PH_3/g-adsorbent, which is approximately double that of the Cu/zeolite adsorbent demonstrated in the literature.

INTRODUCTION

The hydride gas of phosphine (PH_3) is used in large quantity for semiconductor, liquid crystal display (LCD), and light-emitting diode (LED) manufacturing industries in various processes. The unutilized PH_3 gas must be abated right after the process tool because of its high toxicity and flammability. In addition, the PH_3 gas is thought to be an airborne molecular contaminant (AMC), which decreases the product yield. Dry chemical adsorption is a preferred method used for hazardous gas removals from these manufacturing factories. It can treat PH_3 effectively,[1–3] especially when the PH_3 coexists with arsine (AsH_3) in the exhaust gases. However, commercialized dry chemical adsorbents cannot be reused and they must be treated as hazardous wastes. Thus it is essential to develop a regenerative adsorbent for reducing the amount of hazardous waste production.

Earlier researchers[4] treated PH_3 by carbon dry adsorption. The capacity of activated carbon (AC) can reach approximately 12 mg-PH_3/g-AC at 140 °C[5]; however, its disadvantage is its inflammability.[6] The spent carbon might react exothermically with air and result in spontaneous combustion. Thus the AC adsorbent is not practically acceptable in abating the toxic hydride gases for the semiconductor and optoelectronic industries. Li et al.[7] used metal (Cu, Zn, or Mn)-loaded ZSM-5 and Y zeolite adsorbents for the adsorption of PH_3 toxic gas. Their results showed that more than 99% PH_3 adsorption efficiency was achieved when zeolites were loaded with Cu. However, they also revealed that PH_3 removal efficiencies

(REs) can only be up to 97% for the regenerated adsorbent, which cannot meet the more than 99% requirement for toxic PH$_3$ removal from the semiconductor and LCD industries. Recently, some new materials (e.g., titanium dioxide [TiO$_2$] nanotubes or MCM-41) with high specific surfaces were developed and could be used as supports of Cu to enhance the activity of Cu as a catalyst/adsorbent.[8] Unfortunately, these new porous materials with very high specific areas are too expensive thus far to treat the hazardous air pollutant (HAP) in large quantities. It seems that the adsorbents with cost-effectiveness and better regenerative performance are worthy of investigation.

The Cu/γ-alumina (γ-Al$_2$O$_3$) catalyst/adsorbent is commonly used for air pollution control.[9] Conventionally, the γ-Al$_2$O$_3$ catalyst support is prepared from bauxite directly or from the monohydrate by dehydration or recrystallization at elevated temperature.[10] Relatively small surface area and poor attrition resistance are the two major drawbacks of the adsorbents prepared by conventional methods. The small surface area limits the amount of active metal species that can be dispersed on the support surface and thus restricts the adsorption capacity of the adsorbent. Sol-gel-derived adsorbent with the characteristics of high surface area and uniform pore-size distribution has the potential to improve this demerit. Use of the sol-gel-derived Cu/γ-Al$_2$O$_3$ adsorbent for treating oxides of sulfur (SO$_x$) has been attempted.[11] The results indicated that the sol-gel-derived adsorbents offered higher surface area, more uniform pore size distribution, and higher crush strength, thus they showed superior sulfation and regeneration properties than those of the commercially available adsorbents. Studies on simplifying the synthesizing step and improving the performance of adsorbent/catalyst have also been performed.[12,13]

To the best of the authors' knowledge, the performance of Cu catalyst loaded on the sol-gel-derived mesoporous γ-Al$_2$O$_3$ for removal of HAPs such as PH$_3$ has not been investigated. In this study, adsorbents of Cu loaded on mesoporous γ-Al$_2$O$_3$ were prepared and used to investigate the possibility for PH$_3$ removal, and the relationship between adsorption capacity and physical/chemical characteristics of the adsorbents are established. In addition, the possibility of regenerating this mesoporous adsorbent is tested.

EXPERIMENTAL PROCEDURES
Sample Preparation
The sol-gel-derived mesoporous γ-Al$_2$O$_3$ was prepared from 1 M boehmite sol (γ-AlOOH) and it acted as the support of adsorbents. The sol was synthesized by the Yoldas process,[10,14–16] which included dissolution of aluminum (Al) tri-sec-butoxide in deionized water and peptization of the resulting precipitates with nitric acid. The γ-Al$_2$O$_3$ support was then prepared by drying a given amount of γ-AlOOH in a Petri dish followed by calcination at 450 °C for 3 hr. Finally, the product was crushed and sieved into granules with sizes of 420–840 μm.

A total of 20 mL of sol-gel-derived γ-Al$_2$O$_3$ granular and 150 mL of metal precursor solution, Cu(NO$_3$)$_2$ 3 H$_2$O (aq), were mixed and stirred at room temperature for 12 hr. After a filtration process, 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 sample was named CuXM/SGAl, where X represents the concentration of precursor solution. The commercial γ-Al$_2$O$_3$ (lot no. B0625036, Strem Chemicals) was also used as the support of adsorbent for comparison purposes. Similar to sol-gel-derived γ-Al$_2$O$_3$, the commercial γ-Al$_2$O$_3$ was crushed and sieved into granules with sizes of 420–840 μm. The sample was named CuXM/ComAl.

The amount of metal loaded on the γ-Al$_2$O$_3$ was varied using different concentrations of metal precursor solutions, and the actual mass of metal loaded on the γ-Al$_2$O$_3$ was determined by inductively coupled plasma-atomic emission spectrometry (ICP-AES, Jarrell Ash).

Characterization of the Adsorbent
The surface area of the adsorbent was determined by the Brunauer-Emmett-Teller (BET) method applied to the adsorption isotherms of nitrogen, whereas the pore volume and the pore size distribution were determined by the Barret-Joyner-Halenda (BJH) method applied to the desorption isotherms of nitrogen. Nitrogen adsorption/desorption isotherms were obtained by using an ASAP 2020 apparatus (Micromeritics Instrument Corporation).

The X-ray powder diffractometer (XRPD; model XRD-6000, Shimadzu) was used to examine the crystalline structure of the adsorbents. The X-ray source used Cu as its target, and its working voltage and current were set at 40 kV and 30 mA, respectively. The scanning speed was 1 °/min in the 2θ range of 15–90°.

The chemical states of compounds on the adsorbent surface were measured by electron spectroscopy for chemical analysis (ESCA; model ESCA Lab 250, Thermo Electron) before and after adsorption. The X-ray source used was Al Kα. The core-level binding energy of C 1s for carbon at 284.6 eV was used as an internal reference for calibration.

PH$_3$ Adsorption
The PH$_3$ adsorption experiments were carried out in a chamber that controls pressure to be slightly below atmospheric pressure to avoid PH$_3$ toxic gas escaping from the adsorption column. The adsorption temperature was at an ambient temperature of 25 ± 1 °C. The adsorption column was stainless steel with an inner diameter of 2.25 cm and length of 11.7 cm. A total of 15 mL of adsorbents were packed into the column during each test.

The PH$_3$ concentration of 1% (v/v) in nitrogen was used in this study, which is a typical peak value measured in a semiconductor factory. PH$_3$ gas with a flow rate of 0.235 L/min was used to meet the same linear velocity of 1 cm/sec in a typical industrial adsorber for PH$_3$ removal. Fourier transform infrared (FTIR) spectrometry was used to continuously monitor the concentration of PH$_3$. Before the adsorption test, part of the gas flow was directed to the FTIR spectrometer for measuring the inlet PH$_3$ concentration. After that, the whole flow was directed to the adsorber to start the adsorption test. FTIR spectrometry then continuously measured the PH$_3$ concentration at the outlet of the adsorber. This may cause a slight time lag in the detection of the PH$_3$ concentration at the beginning of the test.
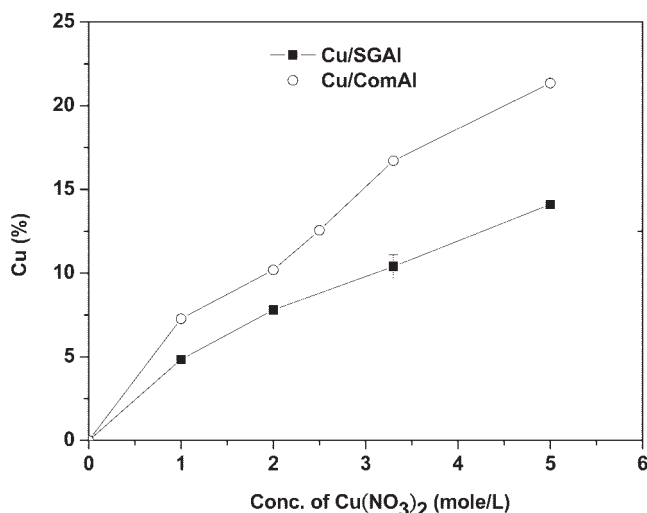
Figure 1. Cu-loaded content (wt %) on ComAl and SGAl adsorbents as a function of $Cu(NO_3)_2$ precursor concentrations.

The RE was defined by the following equation:

$$RE = 100\% \times \left[1 - \frac{Cout}{Cin} \right] \qquad (1)$$

where *Cin* is the influent PH_3 volumetric concentration and *Cout* is the effluent PH_3 volumetric concentration.

The effective adsorption capacity was determined by the cumulated amount of adsorbed PH_3 during the period of over 99% PH_3 RE. That is, the breakthrough point of the adsorbent was set at PH_3 effluent gas concentration of 100 parts per million by volume (ppmv) (0.01% v/v).

Purge and Regeneration

Because PH_3 is a highly toxic compound, the residual PH_3 remaining in the system as gas-phase molecules could result in human health problems. Therefore, after the completion of the adsorption test, the whole system was purged by a nitrogen gas flow to remove the residual PH_3 gas, and then by an airflow to decrease the toxicity of the adsorbed phosphorous species.

It is expected that the mechanism of reaction between PH_3 and Cu compounds should be similar to that between AsH_3 and Cu compounds, in which the active specie, Cu^{2+}, would be reduced to Cu^0 during the PH_3 adsorption.[17] Hence, after adsorption, external air with a flow rate of 2.35 L/min was passed through the adsorption column for 60 min to oxidize the Cu^0. At the same time, the phosphorous species that adsorbed on the adsorbent surface could also be oxidized and then form particulate species, which could be purged out by the same airstream. Thus, the air purging process completed the regenerating procedure of the adsorbent.

RESULTS AND DISCUSSION
Characterization of Cu/γ-Al₂O₃ Adsorbents

This study used Cu as the active metal species for adsorbing PH_3 because it was shown in the authors' prior study[6] that Cu is superior to Zn or Mn under the same preparation conditions. Copper nitrate ($Cu(NO_3)_2$) solutions of

various concentrations ranging from 1 to 5 M were used to obtain Cu/Al_2O_3 adsorbents of various metal loading amounts. The relationships between the precursor concentration of $Cu(NO_3)_2$ 3 H_2O and the actual loading amounts of Cu metal for sol-gel-derived γ-Al_2O_3(Cu/SGAl) and commercial γ-Al_2O_3 (Cu/ComAl) adsorbents are shown in Figure 1. The figure indicates that as the $Cu(NO_3)_2$ 3 H_2O precursor concentrations are increased, the actual Cu content loaded on both types of Al_2O_3 increases in the same manner. However, the amount of Cu loaded on the commercial γ-Al_2O_3 adsorbent is higher than that on the sol-gel-derived γ-Al_2O_3 adsorbent under the same precursor concentration. The maximum Cu loadings on sol-gel-derived γ-Al_2O_3 and commercial γ-Al_2O_3 are 14% (w/w) and 21% (w/w), respectively, with a $Cu(NO_3)_2$ 3 H_2O precursor concentration of 5 M. This may be due to a relatively higher pore volume of commercial γ-Al_2O_3, which provides larger space for the $Cu(NO_3)_2$ precursor solution to be loaded on.

The BET surface area and pore volume of adsorbents are listed in Table 1. One can see that the BET specific surface area and the pore volume for Cu/SGAl and Cu/ComAl adsorbents are decreased as the Cu loading is increased. In addition, the Cu/SGAl adsorbents have relatively higher specific surface areas than those of the Cu/ComAl adsorbents. This can be explained by the pore size distributions of adsorbents, which are shown in Figure 2. The Cu/SGAl adsorbents have smaller pore sizes and narrower pore size distributions than the Cu/ComAl adsorbents. The average pore diameter of Cu/SGAl is increased from 36.1 to 47.6 Å when the Cu loading is increased from 0 to 5 M (14 wt %), and the average pore diameter of Cu/ComAl is increased from 62.6 to 65.6 Å when the Cu loading is increased from 0 to 5 M (21 wt %). This indicates that the relatively uniform and smaller pore size of Cu/SGAl adsorbents as compared with that of the Cu/ComAl adsorbents explains the higher BET surface area of Cu/SGAl adsorbents shown in Table 1.

The XRPD patterns of adsorbents are shown in Figure 3. It is observed that the XRPD patterns of Cu/ComAl adsorbents show observable peaks of CuO crystallite (at 2θ of 35.6 and 38.8°) for Cu loading amounts more than 10% (Cu2M/ComAl). However, the Cu/SGAl adsorbent does not show apparent peaks even if the actual Cu loading is as high as 14% (see B, C, and D pattern in Figure 3 for Cu of 4.8, 10, and 14%). Some studies[10,18,19] found that if the active species is coated on the surface in monolayer or submonolayer form, the active species phase will not be

Table 1. Pore properties of the PH_3 adsorbents.

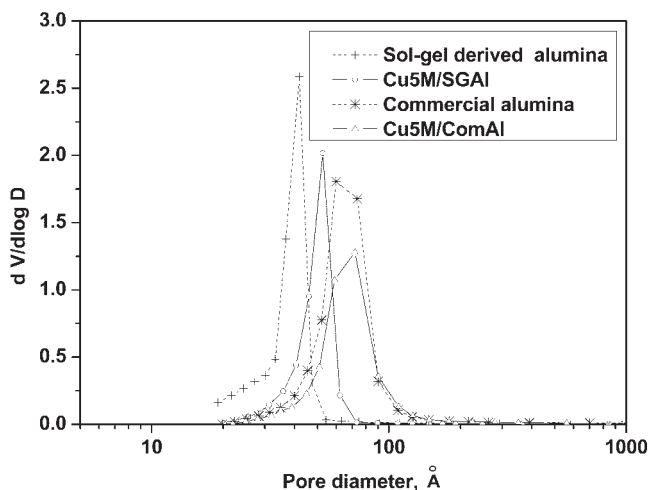| Sample Name	BET Surface Area (m²/g)	Pore Volume (cm³/g)	Average Pore Diameter (Å)
Sol-gel-derived γ-Al₂O₃	296	0.327	36.1
Cu1M/SGAl	196	0.328	44.3
Cu3.3M/SGAl	197	0.296	43.4
Cu5M/SGAl	165	0.275	47.6
Commercial γ-Al₂O₃	209	0.452	62.6
Cu1M/ComAl	173	0.382	64.3
Cu3.3M/ComAl	160	0.364	65.2
Cu5M/ComAl	137	0.324	65.6

Figure 2. Pore size distribution of adsorbents.

detected by XRD. Hence the result of X-ray analysis indicates that CuO might be loaded on sol-gel-derived γ-Al$_2$O$_3$ with well-dispersed monolayer or submonolayer form as compared with the larger crystal form of CuO loaded on the commercial γ-Al$_2$O$_3$.

PH$_3$ Adsorption

The PH$_3$ RE and adsorption capacity were tested for the sol-gel-derived γ-Al$_2$O$_3$ and commercial γ-Al$_2$O$_3$ before metal loading. The results are shown in Figure 4. Without the presence of Cu, commercial and sol-gel-derived γ-Al$_2$O$_3$ could not reach the goal of over 99% PH$_3$ removal and were completely exhausted after a few minutes. On the other hand, the PH$_3$ REs of Cu loaded on γ-Al$_2$O$_3$ adsorbents can be over 99% and they lasted for 27 and 47 min, respectively, for Cu loaded on commercial γ-Al$_2$O$_3$ and sol-gel-derived γ-Al$_2$O$_3$. Hence one can say that blank tests of both γ-Al$_2$O$_3$ materials are not effective in PH$_3$ adsorption and thus Cu must be incorporated with γ-Al$_2$O$_3$ to provide active sites for effectively adsorbing PH$_3$.
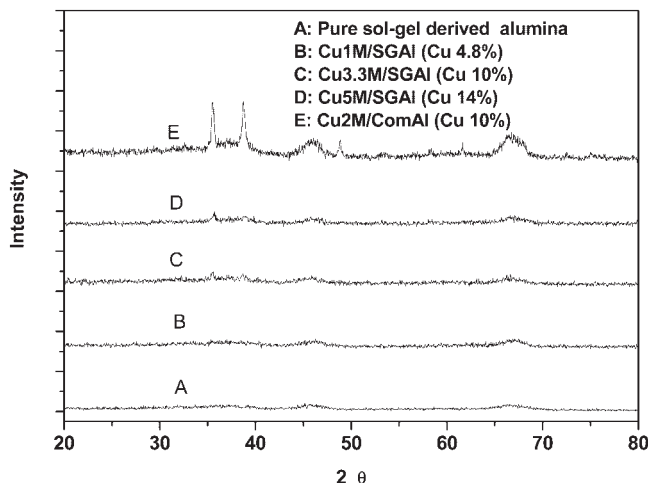

Figure 4. PH$_3$ REs of pure γ-Al$_2$O$_3$ (SGAL, ComAl) and Cu loaded on γ-Al$_2$O$_3$ adsorbents.

The effective adsorption capacities for achieving over 99% PH$_3$ removals with Cu/SGAl and Cu/ComAl adsorbents are shown in Figure 5 as a function of actual Cu-loaded mass concentration. The adsorption capacity of Cu/SGAl adsorbent increases from 0 to 18 mg PH$_3$ per gram adsorbent (mg-PH$_3$/g-ads) as the Cu concentration is increased from 0 to 14% (w/w). Thus the loaded Cu metal amount has a significant effect on the adsorption capacity. Different from Cu/SGAl adsorbents, the adsorption capacity of Cu/ComAl adsorbent increases from 0 to 8.1 mg-PH$_3$/g-ads as Cu loading is increased from 0 to 7.2% (w/w) and then stays at approximately 8.1–8.6 mg-PH$_3$/g-ads even if the Cu-loaded concentration is increased from 7.2 to 16.7% (w/w). And a further increase in the Cu-loaded concentration to 21.4% only leads to a decrease in the PH$_3$ adsorption capacity to 3.4 mg-PH$_3$/g-ads. The maximum adsorption capacity of Cu/SGAl adsorbents, 18 mg-PH$_3$/g-ads, is much higher than that of the Cu/ComAl adsorbents, 8.6 mg-PH$_3$/g-ads.

From the above results, it is possible to further improve the adsorption capacity of adsorbent by increasing
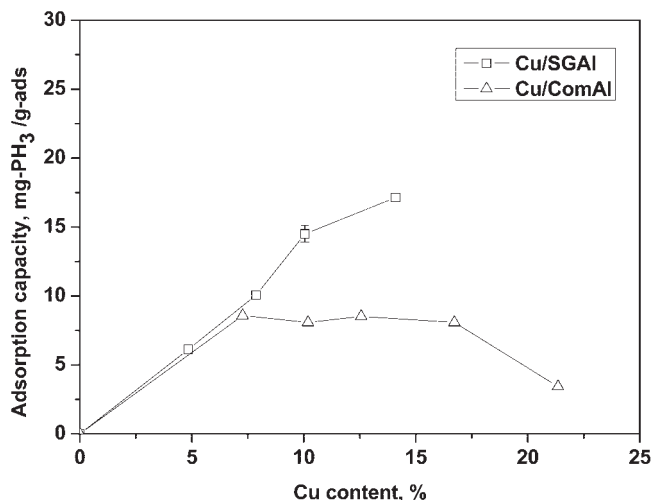

Figure 3. X-ray diffraction patterns of adsorbents.

Figure 5. The PH$_3$ adsorption capacity of Cu/SGAl and Cu/ComAl adsorbents as a function of Cu-loaded content (wt %)
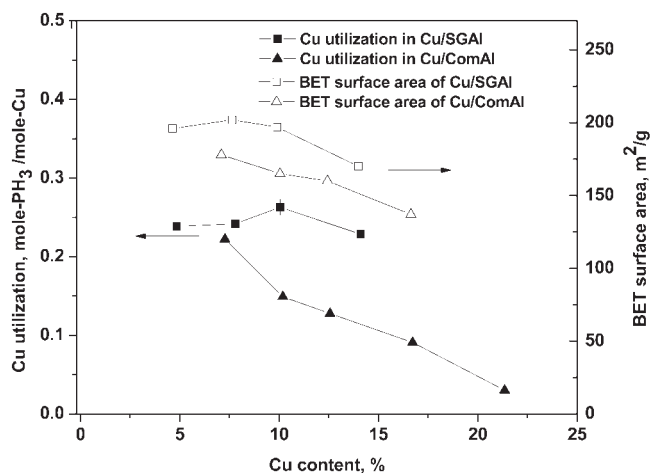
Figure 6. Cu utilization (mole-PH$_3$/mole-Cu) as functions of sorbent specific surface area and Cu-loaded content (wt %).

the Cu loading of Cu/SGAl. However, the maximum concentration of Cu(NO$_3$)$_{2(aq)}$ used in this study (5 M) was close to the upper limit of its solubility in water at room temperature (~5.27 M); a further increase of Cu loading must be achieved by multiple impregnating or under a heating process.

The Utilization of Active Metal Oxides in Adsorbent

For a better understanding of PH$_3$ adsorption behaviors, the relationships between Cu utilization (mole-PH$_3$/mole-Cu), BET specific surface area, and the Cu loading amounts of the Cu/SGAl and Cu/ComAl adsorbents are shown in Figure 6. The figure reveals that the utilization of Cu using the Cu/SGAl adsorbent remains almost the same at approximately 0.24 mole-PH$_3$/mole-Cu when the Cu loading is increased from 4.8 to 14%. The BET surface area is approximately 200 m^2/g for a Cu loading of less than 10% (w/w); it then decreases slightly as Cu loading is increased to 14%. Thus, the decrease in the surface area does not have a significant effect on the Cu utilization rate. Different from the Cu/SGAl adsorbents, the utilization of Cu in Cu/ComAl adsorbent decreases apparently as the Cu loading increases. This corresponds to the decrease of BET surface area as the amount of Cu loading increases. Thus a further increase in the Cu loading amount only results in the decrease in the Cu utilization rate; it cannot help to increase the adsorption capacity.

These results indicate that the Cu utilization rate of Cu/SGAl adsorbent could be kept at a high value even when it was loaded with a high amount of Cu. This may be explained by the XRPD data in which cupric oxide (CuO) was loaded on sol-gel-derived γ-Al$_2$O$_3$ with well-dispersed form. The high BET surface area and narrower and smaller pore size distribution of sol-gel-derived γ-Al$_2$O$_3$ could lead to monolayer or submonolayer distribution of CuO. On the contrary, CuO exists with a larger crystal form although the Cu loading is only 10 wt % in Cu/ComAl adsorbent. The larger CuO crystal has the lower activity to reduce itself and to oxidize the adsorbed PH$_3$ gas. This phenomenon is similar to the observation of SO$_2$ adsorption or CO oxidation by Cu/γ-Al$_2$O$_3$ adsorbent/

catalyst.[10,20] The larger amount of Cu precursor solution loaded on the large pore of commercial γ-Al$_2$O$_3$ leads to the aggregation of CuO particles that become larger crystals and thus decrease the activity of adsorbent.

Except Cu loading amount, the preparing process for adsorbent also influenced the dispersion of active species. Proper stirring time and temperature could cause the Cu precursor to be uniformly coated on the support, which will lead to well-dispersed CuO. In addition, some research showed that mixing the Cu precursor with γ-AlOOH can enlarge the pore volume and BET surface area of the adsorbent,[21] which could be an interesting goal for future study.

Regeneration and Surface Analysis of Adsorbents

The regeneration test was conducted on the Cu5M/SGAl adsorbent for PH$_3$ removal after air purging of the exhausted adsorbent for 60 min. The PH$_3$ REs of Cu5M/SGAl for five regeneration cycles are shown in Figure 7a, which reveals that although the adsorbent deteriorates as regeneration time increases, PH$_3$ REs of up to 99% are still achievable for the regenerated adsorbent. The cumulative adsorption capacity and the adsorption index with respect to the regeneration cycles are shown in Figure 7b.
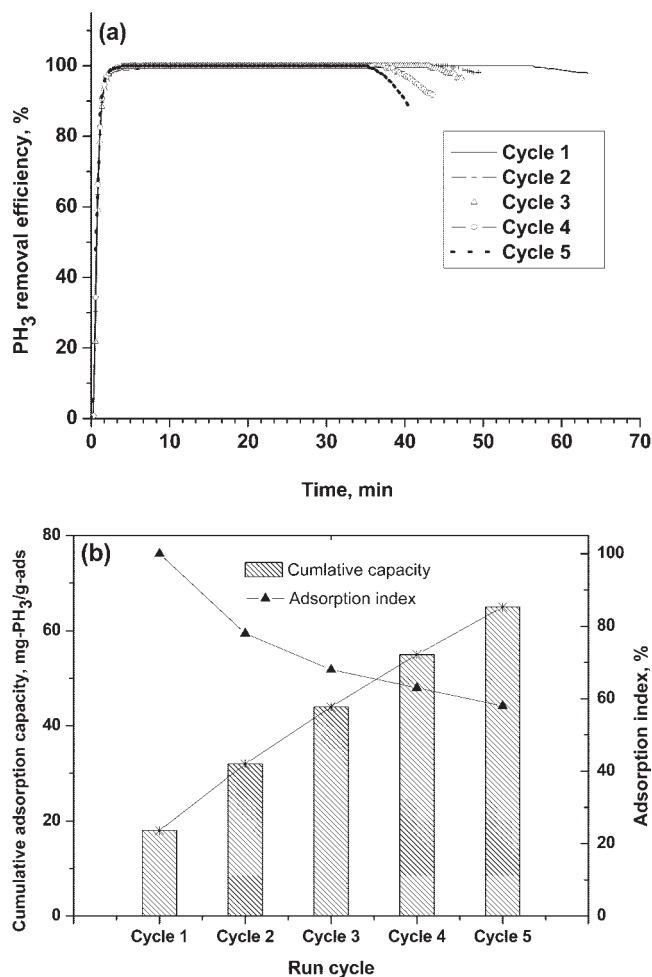

Figure 7. (a) PH$_3$ REs of Cu5M/SGAl at different regeneration cycles. (b) Cumulative adsorption capacity and adsorption index over five cyclic adsorption tests.
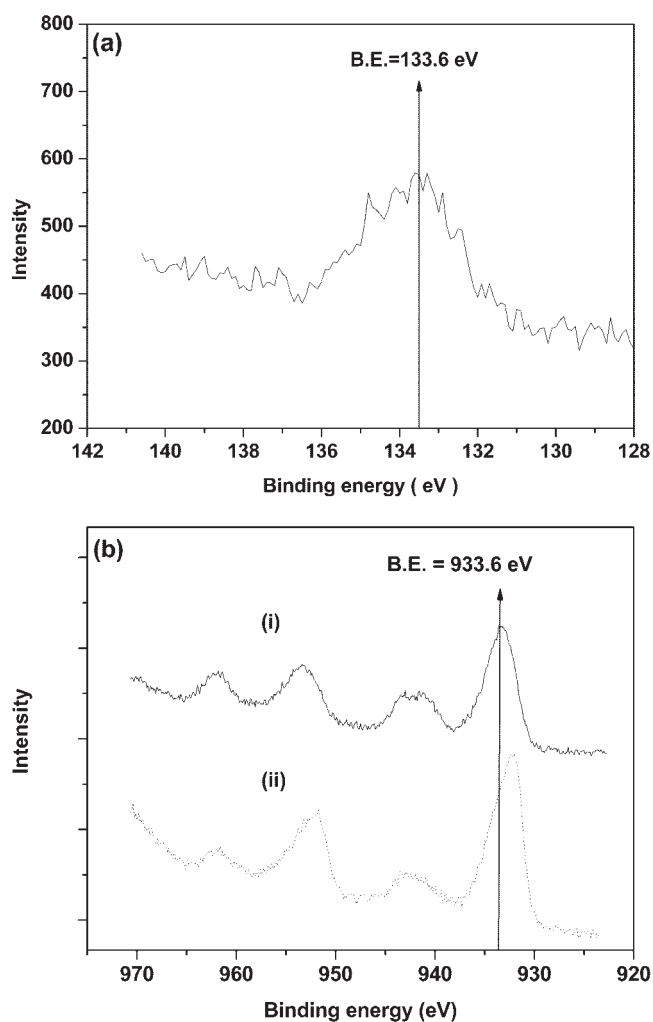
Figure 8. (a) ESCA spectra of Cu5M/SGAl (14% Cu) adsorbent for phosphorus 2p orbital after PH_3 adsorption and air purging. (b) ESCA spectra of Cu5M/SGAl adsorbent for Cu 2p: (i) before PH_3 adsorption and (ii) after PH_3 adsorption and air purging.

The adsorption index (%) was calculated based on the ratio of adsorption capacity of the regenerated adsorbent to the fresh one. Although the adsorption capacity at the second cycle decreases to approximately 80% of the fresh sample, the cumulative adsorption capacity reaches 32 mg-PH_3/g-ads, which is similar to the authors' prior result[7] of 30 mg/g-ads using the relatively expensive Cu/zeolite for adsorbing PH_3. The sum of the adsorption capacity for five cycles is 65 mg-PH_3/g-ads, which is approximately double that of using Cu/zeolite. After the fifth cycle, 58% of the capacity remained as compared with the fresh adsorbent. The adsorption capacity could possibly be increased if the regeneration cycle continues.

For a better understanding of the behavior of the regeneration, surface analysis was conducted before and after the adsorption test. The chemical states of the elements on the Cu5M/SGAl adsorbent before and after adsorption were examined by ESCA. Figure 8a shows ESCA spectra of core-level binding energy in the phosphorus 2p orbital after adsorption/oxidation. The major phosphorus 2p peak centered at 133.6 eV can be assigned to phosphate or phosphite.[4] Thus the reaction product of PH_3

should possibly remain in the form of particles, so if the purged air system is properly designed to blow out the particles and prevent the adsorbent from plugging problems, it may be possible that the adsorption capacity of regenerated adsorbent can be further increased.

The ESCA spectra of core-level binding energy in Cu 2p orbital before adsorption and after adsorption/oxidation are shown in Figure 8b. Before adsorption, the Cu $2p_{3/2}$ peak centered at 933.6 eV can be assigned to CuO.[22,23] After adsorption and air purging (regeneration), the peak shifted to the lower binding energy. This indicates that the Cu exists with a chemical state other than the CuO form. Another possible chemical state of Cu in the regenerated adsorbent is cuprous oxide (Cu_2O) or Cu^0, for which the binding energy of Cu $2P_{3/2}$ is approximately 932.6 eV.[22] This can be explained from the reactions during the PH_3 adsorption and the following regeneration of air purging and oxidation. The reaction during the PH_3 adsorbed on Cu/Al_2O_3 can be inferred to be similar to the reaction between AsH_3 and Cu-Cr/AC adsorbent,[17] at which CuO was reduced to Cu^0 whereas PH_3 was oxidized to less valence of phosphorus. The dominant reaction is proposed as the following equation:

$$2\ PH_3 + 3\ CuO \rightarrow 2\ P + 3\ Cu + 3\ H_2O \qquad (2)$$
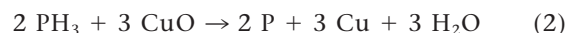
After the air purging, the dominant reactions are proposed as follows:

$$4\ P + 3\ O_2 \rightarrow 2\ P_2O_3 \qquad (3)$$

or

$$4\ P + 5\ O_2 \rightarrow 2\ P_2O_5 \qquad (4)$$

$$2\ Cu + O_2 \rightarrow 2\ CuO \qquad (5)$$

As seen from reaction 2, the stoichiometric reaction for PH_3 and CuO is 2/3, which is higher than the Cu utilization of Cu/SGAl (0.25 mole PH_3/mole Cu) demonstrated previously in Figure 6. This might be because the CuO particles buried in the deep pores can hardly contact the PH_3, the reaction products of phosphate particles blocked the pores, or a small part of CuO did not disperse well on the support. Therefore, after regeneration, the unused CuO in previous adsorption still could not be used. In the regeneration process, Cu^0 was mostly oxidized back to CuO by the oxygen existing in the purging air or by the active oxygen formed on the surface. However, some Cu^0 could not be oxidized or it was oxidized to Cu_2O instead of CuO. This leads to degradation of the chemical adsorbent.

Thus the formation of phosphate on the surface of adsorbent and the transformation of the CuO into another Cu status such as Cu_2O in the CuO/SGAl adsorbent are responsible for the decrease in adsorption capacity for regenerated adsorbent.

CONCLUSIONS

This study demonstrated that the PH_3 adsorption efficiency of greater than 99% using Cu loaded on γ-Al_2O_3 adsorbents was achievable with a PH_3 inlet concentration of 10,000 ppmv. The adsorption capacity of Cu-loaded sol-gel-derived γ-Al_2O_3 adsorbent is much higher than that of Cu-loaded commercial γ-Al_2O_3 because of the high BET surface area and uniform mesopore structure of sol-gel-derived γ-Al_2O_3, which leads to well-dispersed CuO active species as confirmed by XRD patterns. The maximum adsorption capacity is 18 mg-PH_3/g-ads for Cu/SGAl adsorbent and only 8.6 mg-PH_3/g-ads for Cu/ComAl adsorbent. The results also showed that the Cu/SGAl adsorbent can be regenerated by simply purging the air into the adsorption bed. The particulate phosphate formed as the possible reaction product and the decreasing amount of CuO are responsible for the decrease of adsorption capacity for the regenerated cycles. However, the cumulative adsorption capacity for a total of five regenerated cycles is approximately 65 mg-PH_3/g-ads, which is double that reported in the literature. The results indicate that the simple preparation method of Cu loaded on sol-gel-derived γ-Al_2O_3 has a high potential to reduce the hazardous waste produced from hydride air toxic removals. Further study is needed toward the modification of the adsorbent and purging gas conditions for enhancing the regeneration cycles.

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