

Regenerable adsorbent for removing ammonia evolved from anaerobic reaction of animal urine

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Abstract: The waste gas evolved from biodegradation of animal urine contains ammonia causing environmental concerns. A new and effective method for removing ammonia from such waste gas using reactive adsorption is presented. In the process, activated carbon impregnated with H_2SO_4 ($\text{H}_2\text{SO}_4/\text{C}$) is employed. Ammonia in the waste gas reacts with H_2SO_4 on the adsorbent instantaneously and completely to form $(\text{NH}_4)_2\text{SO}_4$. The $\text{H}_2\text{SO}_4/\text{C}$ adsorbent is high in NH_3 adsorption capacity and regenerable. The NH_3 removal capacity of this regenerable adsorbent is more than 30 times that of the adsorbents used normally in the industry. The spent $\text{H}_2\text{SO}_4/\text{C}$ is regenerated by flowing low-pressure steam through the adsorbent bed to remove the $(\text{NH}_4)_2\text{SO}_4$ from the adsorbent. The regeneration by-product is concentrated $(\text{NH}_4)_2\text{SO}_4$ solution, which is a perfect liquid fertilizer for local use. Re-soaking the activated carbon with H_2SO_4 solution rejuvenates the activity of the adsorbent. Thus the $\text{H}_2\text{SO}_4/\text{C}$ can be reused repeatedly. In the mechanism of this reactive adsorption process, trace of H_2O in the waste gas is a required, which lends itself to treating ammonia gas saturated with moisture from biodegradation of animal urine.

Keyword: ammonia removal; regenerable adsorbent; reactive adsorption; biodegradation of animal urine; activated carbon

Introduction

Disposal of livestock wastes are of great environmental concern. Discharging large quantities of liquid manure to rivers results in serious water pollution of the rivers. In addition, the intense foul odor is intolerable. Ammonia is the culprit for the odors. As a result, the treatment of pig wastewater has been the subject of many studies. Separation of concentrated and diluted pig wastewater for further treatment were discussed by Gorecki *et al.* (1993) and Yang *et al.* (1993). Anaerobic treatment was the most popular method and discussed by Yang and Kuroshima (1995), Hill (1982, 1985), Floyd and Hawkes (1986), Yamamoto (1992) and Batstone *et al.* (1997).

Another frequently used process for swine wastewater treatment is an aerobic process, which is used by Jutear (2004) to treat high-strength wastewater. There are other methods: Thorneby *et al.* (1999) used reverse osmosis to treat liquid effluent from dairy cattle and pig. Kim *et al.* (2004) applied integrated real-time control strategy to remove nitrogen in swine wastewater treatment.

In Taiwan, most of the swine wastewater is treated using a three-stage process, consisting of: solid separation, anaerobic reaction and aerobic reaction processes. Foul smelling ammonia is evolved from the anaerobic process. Therefore it is important and urgent to control the ammonia emission to improve the air quality in Taiwan.

To remove ammonia, the wastewater can be treated by stripping with steam or air. However, it is difficult and costly to remove the ammonia to levels

below the specification of 10 ppm. In addition, the small quantity of ammonia can escape to the air causing environmental concerns. The other method is aerobic treatment. It requires a large wastewater treating facility, which a typical pig farm cannot afford. Clearly, there is a need to develop an inexpensive and simple method to remove ammonia from wastewater in Taiwan.

In production of ammonium sulfate, ammonia is scrubbed with sulfuric acid. In addition, wet scrubbing has been used to remove ammonia in biological wastewater-treatment process (Cooper and Alley, 1990). Wet scrubbing is effective but suffers from the problems of scaling inside the tower, equipment plugging, and corrosion (Cavaseno 1980). Furthermore, it is costly to dispose the resulting dilute scrubbed solution.

Instead of wet scrubbing, a fixed bed of zeolite or $\gamma\text{-Al}_2\text{O}_3$ can be employed to adsorb and remove NH_3 from the gas stream. However, in the process, the NH_4OH reacts gradually with the adsorbent to destroy its structure, resulting in increased pressure drop, which makes the operation itself impossible. Moreover, the great quantity of used adsorbent has to be disposed off by use of costly landfill.

Recently, Hudson *et al.* (1974), Tsutsui and Tanada (1987), Brown (1989), Amos Turk *et al.* (1989), and Nevskaja *et al.* (1999) showed that the adsorption properties of the activated carbon can be improved by impregnating it with acid or base solution. Such modified product is called Impregnated Activated Carbon (IAC). Yan (1997) showed that the adsorption efficiency and capacity of IAC are higher than that of the activated carbon itself. Previously, we

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have disclosed a new method to remove HCl from the acidic gas stream by reactive absorption, using NaOH IAC (NaOH/C) (Lee *et al.*, 2003). The experimental results indicated that the NaOH/C has a much higher HCl removal capacity than alumina, generally used in the refinery to remove HCl from refinery gas streams. Moreover, we also showed that NaOH/C is regenerable without generating waste for disposal.

It is thought that a similar principle can be applied to prepare a regenerable adsorbent, H₂SO₄/C to remove NH₃ from waste gases. It is important to note that the NH₃ containing gas from biodegradation of animal urine is saturated with moisture, which is not amenable to treatment using conventional solid adsorbents. In this study, passing an air stream through a reservoir filled with NH₃ simulated evolution of waste gas from biodegradation of animal urine. The effluent was saturated with moisture and contained the desired level of NH₃. This effluent was sent to an adsorption reactor for the test. The details of the preparation procedure, adsorbent testing and regeneration procedure, and efficacy of the adsorbent are described in this paper.

1 Experimental

1.1 Materials and H₂SO₄/C adsorbents preparation

The carbon-supported acidic adsorbent, H₂SO₄/C was prepared by impregnating the activated carbon with sulfuric acid solution. The same procedure was used to re-impregnate the regenerated activated carbon right in the reactor for reuse in the next adsorption cycle. The activated carbon (GAC 830) was purchased from Norit Americas Inc., Atlanta, Georgia USA and the properties were as follows: particle size, 2 to 5 mm; surface area, 1050 m²/g; pore volume, 0.85 ml/g; iodine No., 75 mg/g; minimum and apparent density, 0.54 g/ml. In preparing the H₂SO₄/C, the activated carbon was packed in an adsorption reactor to form a fixed bed and dried at

120°C by passing through air to remove physically adsorbed water. The dried activated carbon was brought into contact with an equal volume of sulfuric acid solution. Upon soaking for 30 min, the reactor was purged with N₂ for 30 min. The resulting material was noted as H₂SO₄/C. In order to obtain H₂SO₄/C with various levels of H₂SO₄ loadings, the concentration of H₂SO₄ solution was varied from 0 to 16 mol/L. For simplification, the H₂SO₄/C prepared from *x* mol/L H₂SO₄ was noted as HIAC(*x*). The actual sulfuric acid concentration on the H₂SO₄/C was determined by titration, employing 6 mg/L NaOH aqueous solution as the titrant.

1.2 Performance test of the H₂SO₄/C (HIAC) adsorbents

The HIACs were tested in a continuous up flow fixed-bed reactor. The reactor was a polyvinyl ethylene tube with an inside diameter of 2.5 cm and length of 50 cm. In order to demonstrate feasibility of *in-situ* regeneration and rejuvenation of the adsorbent for multiple cycles of operation, HIAC was prepared in the reactor according to the following procedure. For impregnation, sulfuric acid solution of the desired concentration was poured into the reactor packed with 50 g of pre-dried activated carbon for 30 min soaking. The H₂SO₄ impregnated carbon was dried by passing N₂ through the bed for 30 min. The test gas stream was prepared by passing 150 ml/min of dry air through a reservoir filled with NH₃ aqueous solution to pick up NH₃ and moisture. A mass flow controller controlled the total gas flow rate and the NH₃ concentration in the gas stream was controlled by the flow rate of carrier gas as shown in Fig.1. During the test, NH₄OH was continuously added to the reservoir to maintain the pH level at 11.5 using a pH controller. By keeping the pH level of the reservoir constant, the NH₃ concentration in the carrier gas was kept constant. The final NH₃ concentration of 70 µg NH₃/ml air was achieved by breeding in the necessary amount of

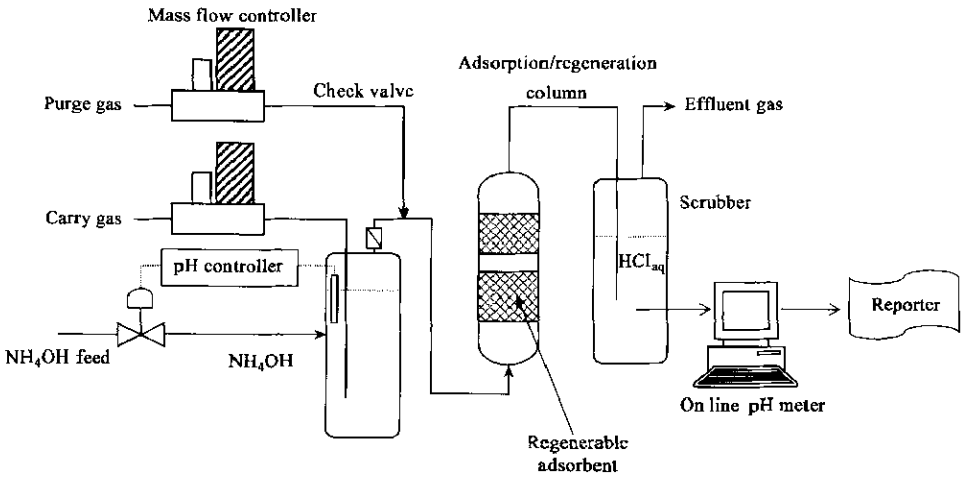


Fig.1 Schematic diagram of the H₂SO₄/C regenerable adsorbent performance tests

purge gas through a mass flow controller.

Typically an ammonia concentration of 70 $\mu\text{g NH}_3/\text{ml}$ air was used in the performance tests. To determine the adsorption capacity of $\text{H}_2\text{SO}_4/\text{C}$ (g NH_3 adsorbed/g of adsorbent), the break through NH_3 in the effluent gas from the adsorption reactor was titrated with HCl solution using an on-line pH meter with a precision of 0.01 pH. A break through curve was constructed by plotting the rate of HCl addition with time. The total amount of NH_3 removal was calculated from the break-through curve and verified by analyzing and characterizing the used $\text{H}_2\text{SO}_4/\text{C}$. For direct comparisons, similar testing procedure was also applied on γ -alumina (A2U in granular form with surface area of 170 m^2/g , purchased from Osaka Yogyo, Japan), alumina bound HY zeolite in cylindrical shape (CBV 780 with surface area of 170 m^2/g , $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio of 80, purchased from PQ Zeolite, USA), and activated carbon itself (GAC 830). However, no regeneration procedure was performed on those adsorbents because their efficacies were so inferior to that of the $\text{H}_2\text{SO}_4/\text{C}$.

1.3 Regeneration of used adsorbent

The used adsorbent can be regenerated by washing away the $(\text{NH}_4)_2\text{SO}_4$ formed on the activated carbon and followed with drying, and rejuvenated by re-soaking with sulfuric solution right in the adsorption reactor. This *in-situ* regeneration and rejuvenation procedure are characteristics of the present process. The apparatus for adsorbent regeneration is shown in Fig.2. In the washing step, low-pressure steam was used to provide the heat and the water for washing, and the process was continued until no sulfate was detected in the effluent water. In stead of low pressure steam, water can also be used to wash off the $(\text{NH}_4)_2\text{SO}_4$ salt to regenerate the activated carbon. After the washing step, the activated carbon support was purged with low-pressure steam (103–105°C) for 2 h to remove excess water, followed with 12 mol/L sulfuric solutions soaking for 30 min. The H_2SO_4 solution soaked carbon was purged with nitrogen for 30 min, before starting the next adsorption cycle. The adsorption and regeneration

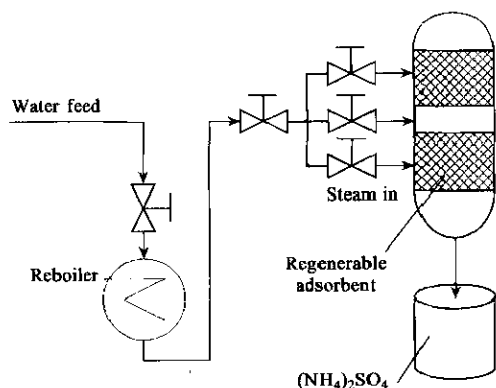


Fig.2 Schematic diagram for the steam regeneration of $\text{H}_2\text{SO}_4/\text{C}$

cycle was repeated for 3 times to assess the re-usability of $\text{H}_2\text{SO}_4/\text{C}$. Surface area loss of the activated carbon support during the regeneration process was determined using Micro-meritics ASAT 2000, USA.

2 Results and discussion

2.1 Performance of adsorbents

A blank test was conducted with an empty adsorption reactor before the performance tests. As shown in Fig.3, the experimental results indicated that the experimental apparatus did not adsorb NH_3 gas, suggesting that the performance test would not be influenced by the adsorption and/or deposition of NH_3 on the reactor and the piping.

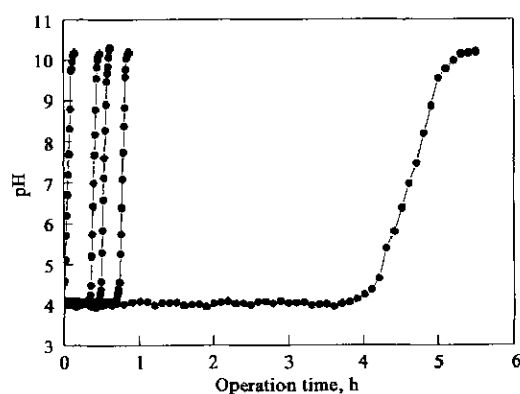
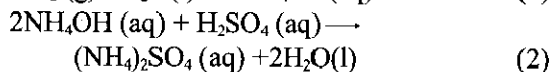


Fig.3 Break-through curve for different adsorbents
From left to right: glass ball, activated carbon, $\gamma\text{-Al}_2\text{O}_3$, HY zeolite, and HIAC(2.1); $\text{H}_2\text{SO}_4/\text{C}$ prepared from 2.1 mol/L H_2SO_4

In comparing the NH_3 breakthrough curves of $\text{H}_2\text{SO}_4/\text{C}$ with those of γ -alumina, HY zeolite, and activated carbon, it is obvious that the NH_3 removal capacity of $\text{H}_2\text{SO}_4/\text{C}$ was much greater; the adsorption capacities are about 0.05 and 0.01 g NH_3/g adsorbent for HIAC (2.1) and HY, respectively. The results of $\text{H}_2\text{SO}_4/\text{C}$ are interesting not only because of its excellent performance, but also the NH_3 removal mechanism as described in the next section. Unlike physical adsorption of NH_3 on activated carbon, zeolite, and γ -alumina, HIAC removes NH_3 in the waste gas by reactive adsorption through acid/base neutralization. Stoichiometric calculation indicated that essentially all the H_2SO_4 on the activated carbon were reacted with NH_3 in the adsorption process. Since NH_3 removal is mostly through acid/base neutralization and all the H_2SO_4 on $\text{H}_2\text{SO}_4/\text{C}$ can be used for the neutralization, we can predict the life cycle of the adsorbent by determining H_2SO_4 concentration in $\text{H}_2\text{SO}_4/\text{C}$.

2.2 Mechanism of NH_3 removal and the role of activated-carbon support

In the reactive adsorption of NH_3 by use of $\text{H}_2\text{SO}_4/\text{C}$, NH_3 reacts with H_2SO_4 according to the following equations:



The mechanism by which the NH_3 is removed from the gas was speculated to consist of four steps in series: (1) NH_3 transport from waste gas stream to the external surface of the activated carbon support; (2) the adsorbed NH_3 diffuse to the pore mouth of the activated carbon; (3) NH_3 molecule diffuses through a gas-liquid interface and dissolves in the aqueous H_2SO_4 layer (Equation (1)); (4) aqueous NH_3 reacts with H_2SO_4 to form $(\text{NH}_4)_2\text{SO}_4$ (Equation (2)) which remains on the adsorbent.

As shown in Fig.3, steep breakthrough curves have been observed for γ -alumina, HY zeolite, and activated carbon samples, suggesting that the adsorption process do not have mass transfer barrier for those three samples. In contrast, a relatively gradual slope on the breakthrough curve has been observed for the $\text{H}_2\text{SO}_4/\text{C}$ sample. These results indicated that transportation of NH_3 from gas stream to water film could present a significant mass transfer resistance. Fortunately, the mass transfer is enhanced by the chemical reaction since the neutralization reaction in the 4th step is irreversible and spontaneous. The activated carbon provides very high surface area to facilitate dispersion of aqueous H_2SO_4 . Hence, the short diffusion path of NH_3 for reaction with H_2SO_4 further minimizes the mass transfer resistance.

In typical physical adsorption process, moisture competes strongly with NH_3 for adsorption, leading to greatly reduced adsorption capacity of the adsorbent. However, in the reactive adsorption of NH_3 by use of $\text{H}_2\text{SO}_4/\text{C}$, moisture is a necessary component in the reaction (Reaction 1). Thus, $\text{H}_2\text{SO}_4/\text{C}$ is most useful for removing NH_3 from gases containing moisture, such as effluent gas from biological degradation of animal urine, which is the main objective of this study.

2.3 Limitation of H_2SO_4 concentration

When NH_3 diffuses through and dissolves in the water film (Equation (2)), the mass transfer rate of NH_3 is increased by the neutralization reaction. Hence, we might expect that mass transfer increase with increasing H_2SO_4 concentration on the carbon (Froment and Bischoff, 1979). However, unlike the normal absorption process, the resistance of mass transfer is also impeded by the $(\text{NH}_4)_2\text{SO}_4$ crystals accumulated in the pore, particularly near the pore mouth. When the carbon is impregnated with too high H_2SO_4 concentration, the large quantity of $(\text{NH}_4)_2\text{SO}_4$ formed in the reaction will not completely be dissolved in water, and will crystallize and deposit in the pore of the activated carbon, particularly near the pore mouth. Pore mouth plugging by the $(\text{NH}_4)_2\text{SO}_4$ buildup inhibits the diffusion of NH_3 into the inner

pores. The H_2SO_4 deep in the pore becomes inaccessible to reaction with NH_3 . The inaccessible H_2SO_4 not only reduces NH_3 adsorption capacity but also causes disposal problems of spent HIAC. Impregnating the carbon with too much water, on the other hand, would decrease NH_3 treatment capacity. Thus, an optimum concentration of H_2SO_4 on activated carbon can be expected.

The breakthrough curves characterizing the effects of H_2SO_4 concentration on the performance of HIAC for NH_3 removal are shown in Fig.4. The time at which a significant concentration of NH_3 breaks through the bed (break point) is linearly proportional to H_2SO_4 concentration up to 11.27 mol/L, then decreases when the concentration is further increased. The NH_3 -removal capacity calculated from the breakthrough curves as function of H_2SO_4 concentration is shown in Fig.5. These results confirmed that NH_3 removal is through a neutralization reaction. Moreover, the adsorption capacity for HIAC (16.0) less than that for HIAC (11.3), confirming that the pore-mouth plugging caused by $(\text{NH}_4)_2\text{SO}_4$ deposition limits the concentration of H_2SO_4 in preparation of $\text{H}_2\text{SO}_4/\text{C}$. The results would suggest that the optimum concentration of H_2SO_4 is about 11.3 mol/L.

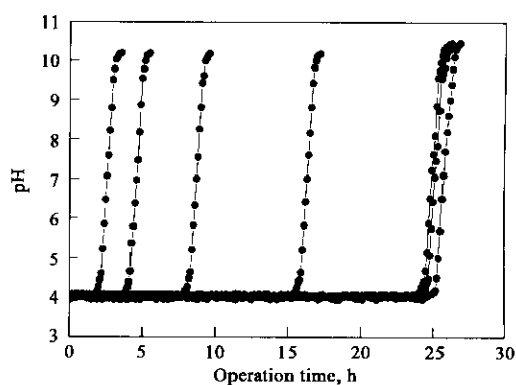


Fig.4 Effects of H_2SO_4 concentration and steam regeneration on the break-through curve of $\text{H}_2\text{SO}_4/\text{C}$

From left to right: HIAC (1.0), HIAC (2.1), HIAC (16.0), HIAC(7.9), HIAC (11.3) after two times regeneration, HIAC (11.3) after one time regeneration, fresh HIAC(11.3)

2.4 Efficiency of regeneration

The used absorbents can be regenerated with low-pressure steam and rejuvenated with H_2SO_4 reimpregnation. The effluent from regeneration is an aqueous solution of $(\text{NH}_4)_2\text{SO}_4$, which is a perfect fertilizer for use right at the farm. As shown in Fig.6, about 800 g of steam was needed to treat 50 g absorbents. The performance tests for the steam regenerated $\text{H}_2\text{SO}_4/\text{C}$ are also shown in Fig.4. The experimental results showed that the adsorption capacity of used $\text{H}_2\text{SO}_4/\text{C}$ was restored, indicating that the $\text{H}_2\text{SO}_4/\text{C}$ can be regenerated and rejuvenated. After the first regeneration, NH_3 removal capacity was decreased by about 7% but remained rather constant

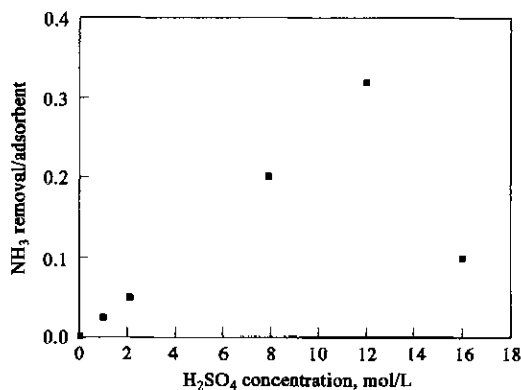


Fig.5 Effects of H_2SO_4 concentration in $\text{H}_2\text{SO}_4/\text{C}$ on the NH_3 removal efficiency

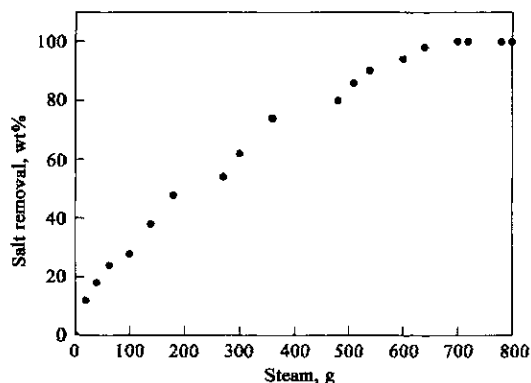


Fig.6 $(\text{NH}_4)_2\text{SO}_4$ removed from used $\text{H}_2\text{SO}_4/\text{C}$ vs. the amount of steam regeneration for the used HIAC(16.0)

in the subsequent regeneration cycles. The slight loss in NH_3 treatment capacity might be caused by the loss of surface area during the steam regeneration. After 3 times of regeneration, the $\text{H}_2\text{SO}_4/\text{C}$ was unloaded and the BET surface area measurement indicated that the surface area of the activated-carbon support was about 90% that of the fresh activated carbon. It is speculated that the surface area losses would be minimal in the subsequent regeneration cycles. As indicated in Fig.7, the decrease in the surface area was caused by the collapse of the pores less than 10×10^{-9} and formation of new pores of about 20×10^{-9} in radius. However, the total pore volume might not change significantly, so that the NH_3 removal capacity should not decrease significantly. Couple the experimental data with the consideration on structural change of the carbon support in regeneration, we concluded that $\text{H}_2\text{SO}_4/\text{C}$ was regenerable. It is noted that, as shown in Figs.3 and 4, the NH_3 removal capacity of the regenerated $\text{H}_2\text{SO}_4/\text{C}$ is about 30 times that of zeolite. The experimental results prove that $\text{H}_2\text{SO}_4/\text{C}$ is effective with high NH_3 removal capacity and the activated carbon support can be reused repeatedly. Hence, $\text{H}_2\text{SO}_4/\text{C}$ is very effective for the treatment of NH_3 containing waste gas, particularly that evolved from biodegradation of animal urine.

3 Conclusions

A new process using reactive adsorption for removing ammonia evolved from biodegradation of animal urine is presented. In the process, HIAC adsorbent is employed and ammonia in the gas reacts with H_2SO_4 in activated-carbon pore instantaneously and completely. The NH_3 removal capacity of $\text{H}_2\text{SO}_4/\text{C}$ adsorbent is more than 30 times that of typical zeolites. An adsorbent with the highest absorption capacity was obtained by use of 11.3 mol/L H_2SO_4 concentration in the preparation. The used adsorbent can be regenerated repeatedly by low pressure steaming without generating waste for disposal. About 5% loss in NH_3 removal capacity of

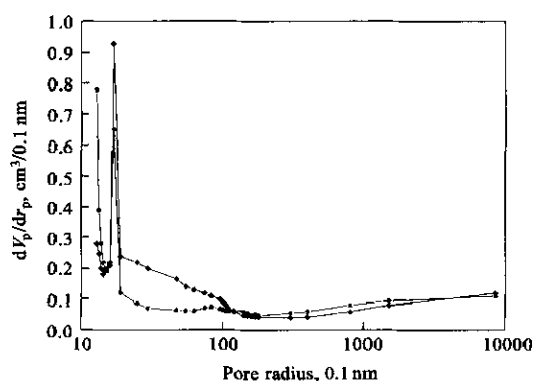


Fig.7 Comparison of pore size distribution for the activated carbon (●) and the activated carbon after 3 times steam regeneration (◆)

$\text{H}_2\text{SO}_4/\text{C}$ in the early cycles of regeneration was due to collapse of the fine pores and, in turn, formation of the larger pores. The process lends itself to removal of NH_3 from the effluent in biodegradation of animal urine, which is normally saturated with moisture.

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