Diffusion of hydrogen sulfide and methyl mercaptan onto microporous alkaline activated carbon

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Abstract

Activated carbon kinetic studies show that both H₂S and CH₃SH yielded pore diffusion coefficients from 10⁻⁶ to 10⁻⁸ cm²/s. Results indicated that pore structures could influence effective diffusivity. Under the same adsorbate concentration, CH₃SH exhibited a greater effective pore diffusion coefficient than H₂S. This may be attributed to the fact that CH₃SH has both polar (–SH) and non-polar (–CH₃) functional groups and dissolves into water easier, thus providing more attraction for the activated carbon surface. In addition, the saturation vapor pressure of CH₃SH is lower than that of H₂S. Therefore, CH₃SH is easier to adsorb onto activated carbon than H₂S. © 2000 Elsevier Science Ltd. All rights reserved.

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1. Introduction

Gaseous odors are emitted from various plants, such as sewage treatment plants and chemical industries (Vigneron et al., 1994). Odors from such plants are being treated by adsorption on activated carbon or impregnated activated carbon, scrubbing and neutralizing by chemical solution, condensation, mashing or catalytic combustion. Adsorption, however, is used most often to remove H₂S and CH₃SH from gas streams (Turk et al., 1989; Koe and Tan, 1990). Therefore, several impregnated activated carbons have been developed for deodorization (Tsutsui and Tanada, 1987; Ikeda et al., 1988; Tsai et al., 1992, 1999).

When removing adsorbate from air mixtures flowing into granular activated carbon, the following sequence

of reaction steps is possible (Jonas, 1978): mass transfer, surface diffusion, intragranular diffusion, physical adsorption, gas desorption, chemical reaction and surface renewal. If the products of a chemical reaction are volatile and poorly adsorbed, they leave the macropore region and enter the fluid stream surrounding the carbon granule where they are swept away from the activated carbon by mass transport.

When Tien (1994) modified the results of Jonas, he noted that diffusion was the predominant mechanism in activated carbon sorption. Recent research on sorption by dry soil grains revealed that diffusion processes govern the rate at which gas-phase species reach the intragranular soil surface. The structural similarity between porous soil grains and activated carbon suggests similar mechanisms in both cases. Several related studies support this conclusion (Garg and Ruthven, 1972; Ruthven and Derrah, 1972; Gray and Do, 1989a,b, 1990).

The purpose of this study was to investigate the physicochemical characteristics of five activated

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carbons. External diffusion, macropore diffusion, and micropore diffusion of H_2S and CH_3SH on the five activated carbons were calculated.

2. Theory

Three solid surface adsorption mechanisms were investigated: external diffusion and internal diffusion (macropore diffusion and micropore diffusion). These mechanisms affected the pore structural distribution of the adsorbent (Yang, 1987).

2.1. External diffusion

External diffusion is the gaseous reactant transfer from the bulk gas stream to the external surface of the solid particle. The mass flux being transferred from the gas stream to solid surface is given by Noll et al. (1992).

$$N_A = \frac{k_f a_p \varepsilon}{\rho_p} (C - C_s), \quad (1)$$

where N_A is the mass flux of the transferred species from gas stream to adsorbent surface, k_f the external film mass transfer coefficient, a_p the external surface area of adsorbent, ε the voids between adsorbents, ρ_p the bulk density of adsorbent, C the concentration of the transferred species in the bulk of gas stream, and C_s the concentration of the transferred species at the adsorbent surface.

For packed bed adsorption, the external film mass transfer coefficient (k_f) was correlated by the Ranz–Marshall equation following Eq. (2) (Noll et al., 1992):

$$\text{Sh} = \frac{2k_f r_p}{D_m} = 2.0 + 0.6 \text{Re}^{0.5} \text{Sc}^{0.33}, \quad (2)$$

where D_m is the molecular diffusivity, r_p the radius of adsorbent, Re the Reynolds number ($\text{Re} = (2r_p V_s \rho_f / \mu)$), Sc the Schmidt number ($\text{Sc} = \mu / (D_m \rho_f)$), Sh the Sherwood number ($\text{Sh} = (2k_f r_p) / D_m$), μ the viscosity of gas stream, V_s the velocity of gas stream, and ρ_f is the density of gas stream.

2.2. Internal diffusion

2.2.1. Macropore diffusion

The importance of heterogeneous catalysis in macropore diffusion has been a popular research subject. There are four kinds of macropore diffusion: (1) molecular diffusion, (2) Knudsen diffusion, (3) Poiseuille flow diffusion, and (4) surface diffusion. The diffusion mechanisms have been shown to affect adsorbent pore structure distribution (Ruthven, 1984).

The mathematical relationships for each macropore diffusion are shown below:

(1) Molecular diffusion:

$$D_p = \frac{D_m}{\tau}, \quad (3)$$

where D_p is the pore diffusivity, D_m the molecular diffusivity and τ is the tortuosity factor.

Fuller et al. derived the molecular diffusivity (D_m) of a two-component system as follows (Bird et al., 1960; Sherwood et al., 1975; Hines and Maddox, 1985; Szekely et al., 1976):

$$D_m = \frac{1.0 \times 10^{-3} \times T^{1.75}}{P \left[(\sum v)_A^{1/3} + (\sum v)_B^{1/3} \right]^2} \left(\frac{1}{M_A} + \frac{1}{M_B} \right)^{1/2}, \quad (4)$$

where P is the pressure of gas stream, T the absolute temperature, $(\sum v)_A$ and $(\sum v)_B$ are the molecular volumes of gases A and B, and M_A and M_B are the gas molecular weights of A and B. Generally, the mean free path of molecules is smaller than the pore diameter of the adsorbent. The transport mechanism is therefore molecular diffusion.

(2) *Knudsen diffusion*: Knudsen showed that under these conditions the diffusivity per unit cross-sectional area of pore is given by

$$D_k = \frac{2}{3} \bar{v} = 9700 r_p \sqrt{\frac{T}{M}} \quad (5)$$

where D_k , r_p , $\bar{v}$, T and M are Knudsen diffusivity, pore radius, mean molecular velocity, absolute temperature and molecular weight, respectively.

(3) *Surface diffusion*: The overall diffusion coefficient is shown as:

$$D = D_k + \left(\frac{1 - \varepsilon_p}{\varepsilon_p} \right) \cdot K D_s, \quad (6)$$

where D is the overall diffusivity, D_k the Knudsen diffusivity, ε_p the adsorbent porosity, K the equilibrium constant, and D_s is the surface diffusivity.

2.2.2. Micropore diffusion

According to Fick's law, one has

$$N = -D \frac{\partial C}{\partial r}, \quad (7)$$

where N is the mass flux of transferred species, D the effective diffusivity of transferred species, C the concentration of transferred species, and r is the distance normal to the external surface.

The micropore diffusion is derived as follows (Noll et al., 1992):

$$\frac{\partial q}{\partial t} = \frac{1}{r^2} \frac{\partial}{\partial r} \left(r^2 D_c \frac{\partial q}{\partial r} \right). \quad (8)$$

Assuming D_c is constant, Eq. (8) can be derived as Eq. (9).

$$\frac{\partial q}{\partial t} = D_c \left(\frac{\partial^2 q}{\partial r^2} + \frac{2}{r} \frac{\partial q}{\partial r} \right). \quad (9)$$

The initial and boundary condition are shown as follows:

$$q(r, 0) = q_0, \quad q(r_c, t) = q_0, \quad \left(\frac{\partial q}{\partial r} \right)_{r=0} = 0,$$

then

$$\frac{\bar{q} - q_0}{q_0 - q_0} = \frac{M_t}{M_\infty} = 1 - \frac{6}{\pi^2} \sum_{n=1}^{\infty} \frac{1}{n^2} \exp \left(- \frac{n^2 \pi^2 D_c t}{r_c^2} \right) \quad (10)$$

and

$$\bar{q} = \frac{3}{r_c^3} \int_0^{r_c} q \cdot r^2 dr, \quad (11)$$

where q is the amount adsorbed (per unit volume of sorbent), $\bar{q}$ the average amount adsorbed in a pellet or particle, M_∞ the total mass uptake at a gas-phase concentration of C_0 and M_θ is the cumulative mass uptake at dimensionless time θ .

3. Experimental

This research selected spent activated carbon (SAC), regenerative activated carbon (RAC), fresh activated carbon (FAC), impregnated-regenerative activated carbon (RAC-N), and impregnated-fresh activated carbon (FAC-N) as the experimental adsorbents. The activated carbons were made from coconut shell.

3.1. Regeneration of spent activated carbon

50 g of SAC (Chinese Carbon, Taiwan) was placed into a vacuum oven (10^{-1} – 10^{-2} mmHg) at room temperature, under nitrogen (99.95%), for 2 h. Next, 10 g of the pretreated SAC was put into an oven at 400°C for 1.5 h. The high purity nitrogen gas was used to cool the oven. The regenerative-spent activated carbon was stored in gas-sealed vials until experimentation.

3.2. Preparation of impregnated activated carbons

50 g of activated carbon (RAC or FAC) was placed into an oven and dried with flowing nitrogen gas (140°C) for 6 h. The pretreated activated carbons were immersed in 500 ml⁻¹ N NaOH solution (Merck, Germany) and stirred for 30 min. The immersed activated carbons were kept in a vacuum oven for 30 min and in a dryer (glass container with silica gel) for 200 min (stationary times)

at room temperature. The immersed activated carbons were then filtered from the impregnation solutions and dried in an oven at 130°C for 60 h. The prepared alkaline activated carbons were stored in gas tight, nitrogen gas-filled containers before use.

3.3. Physical characteristics

The activated carbons were stored in an oven at 105°C and dried for 48 h. The physical characteristics of the activated carbon, including specific surface area, micropore area, total pore volume, micropore volume and pore diameter were measured with $N_2(g)$ adsorption using an ASAP 2000 Micropore Analyzer (Micromeritics, USA) at 77 K using liquid N_2 .

3.4. Amount of alkaline on activated carbon

The quantity of alkaline or NaOH on each activated carbon was determined by extraction and titration processes. The impregnated activated carbons were placed in a vacuum oven (1 – 10^{-1} mm Hg, 105°C) for 24 h. HCl solution (1 N) was then added, and the mixture stored at 25°C for 24 h. To separate the supernatant, the sample was centrifuged at 3000 rpm for ten min. The supernatant was then titrated with 1 N NaOH solution until pH 7.

3.5. Sorption experiment

A simulated blend of cylinder gases was passed through a glass column 20 cm in length and 28 mm in diameter. The bottom of the adsorption column was packed with a layer of 10-cm glass beads. 5–10 g of activated carbon (radius 0.5 mm) was packed in the column for each run. Cylinder gases of H_2S (8000 ppm) and CH_3SH (99.9%) were certified by suppliers (Scott Gas Company, USA). The influent concentrations of H_2S and CH_3SH ranged from 30–200 ppmv in the adsorption system. The flow rate, which ranged from 2.0–10 l/min, was controlled by a mass flow meter (Sierra Series 9000, USA).

H_2S and CH_3SH stream constituents were analyzed by a Gas Chromatograph (HP-6890) equipped with a Pulsed Flame Photometric Detector (PFPD) and chromatographic column (G.S.Q.: 30 m, $\varnothing$: 0.53 mm). Temperature of injector, column, and detector were 180, 150, and 220°C, respectively. Retention times of H_2S and CH_3SH were 4.1 and 5.93 min, respectively. The adsorption capacities of H_2S and CH_3SH on each activated carbon in the gas stream were analyzed by the GC/PFPD and calculated by column adsorption kinetic curves. Quality control was also conducted to ensure experimental data performance.

4. Results and discussion

4.1. Physico-chemical characteristics of activated carbons

Physico-chemical characteristics of the five activated carbons are shown as Table 1. The sequence of BET surface area, micropore area, pore volume and micropore volume were as follows: $\text{FAC} > \text{FAC-N} > \text{RAC} > \text{RAC-N} > \text{SAC}$. The pore diameter distribution was $\text{SAC} > \text{RAC} > \text{RAC-N} > \text{FAC} \approx \text{FAC-N}$. The amount of alkaline equivalent on the activated carbons was $\text{FAC-N} > \text{RAC-N} > \text{FAC} > \text{RAC} > \text{SAC}$. Results indicated that SAC was the least effective adsorbent of the five activated carbons. NaOH impregnation changed their physicochemical characteristics.

4.2. External diffusion

At 298 K, the viscosity of air, H_2S , CH_3SH , air- H_2S gas mixture and air- CH_3SH gas mixture are 184.6, 126.5, 95.8, 170.5 and 143.5 μP , respectively. The Reynolds number, Schmidt number and Sherwood number were 16.1, 1.1, and 8.0 for the H_2S -air mixture system, and 12.0, 2.0 and 8.1 for the CH_3SH -air mixture system. The external film mass transfer H_2S and CH_3SH coefficients were 14.8 and 11.2 cm/s , respectively.

4.3. Macropore diffusion

The diffusion of adsorbates in macropore-molecular diffusion, surface diffusion, and Knudsen diffusion, were considered the diffusion mechanisms. Results of the H_2S and CH_3SH macropore diffusivity into the five activated

carbons (SAC, FAC, RAC, RAC-N, and FAC-N) are shown in Table 2.

4.3.1. Molecular diffusion

According to Eq. (4), molecular diffusivity affects the temperature, pressure, molecular weight and adsorbate characteristics. In this research, the molecular diffusivity of the Air- H_2S and Air- CH_3SH systems were 0.185 and 0.138 cm^2/s , respectively. The molecular weight and volume of H_2S is smaller than CH_3SH , so the molecular diffusivity of H_2S was greater than CH_3SH .

4.3.2. Knudsen diffusion

When the mean free path of the adsorbate is greater than the pore diameter of the activated carbon, the collision of molecules and the pore wall can inhibit molecule transport. The Knudsen diffusivity is a function of the pore radius, temperature and molecular weight (Eq. (5)). Analysis indicated that the Knudsen diffusivity of H_2S was between 4.33×10^{-3} and 5.04×10^{-3} cm^2/s and that of CH_3SH was between 3.51×10^{-3} and 4.24×10^{-3} cm^2/s . The Knudsen diffusivity was directly proportional to the pore radius under identical adsorbate and adsorption temperature conditions. Since CH_3SH molecular weight is greater than H_2S , the Knudsen diffusivity of H_2S was greater than CH_3SH in an adsorption system.

4.3.3. Diffusion of transient region

The diffusivity of the transient region can be calculated as

Table 1
Physico-chemical characteristics of activated carbons

Adsorbents	BET surface area (m^2/g)	Micropore area (m^2/g)	Pore volume (cm^3/g)	Micropore volume (cm^3/g)	Pore diameter ($\text{\AA}$)	Alkaline equivalent (meq/g)
SAC	285	186	0.170	0.088	17.6	0.75
RAC	794	610	0.429	0.282	16.1	1.75
FAC	1282	864	0.623	0.394	14.6	1.88
RAC-N	664	505	0.338	0.238	15.1	2.63
FAC-N	1188	789	0.581	0.335	14.5	3.13

Table 2
Macropore diffusion of H_2S and CH_3SH on activated carbons

Adsorbents	H_2S (cm^2/s)		CH_3SH (cm^2/s)	
	$D_k \times 10^3$	$D \times 10^3$	$D_k \times 10^3$	$D \times 10^3$
SAC	4.33	4.23	3.64	3.55
RAC	4.63	4.52	3.90	3.79
FAC	4.19	4.09	3.52	3.44
RAC-N	5.04	4.91	4.24	4.11
FAC-N	4.18	4.08	3.51	3.43

$$\frac{1}{D} = \frac{1}{D_k} + \frac{1}{D_m} \left[1 - \left(1 + \frac{N_B}{N_A} \right) X_A \right], \quad (13)$$

where D is diffusivity of the transient region, N_A and N_B are the molecular transport flux of A and B , and X_A is the molar fraction of A . Assuming the molecular transport flux of A is equal to that of B ($N_A = N_B$), the equation simplifies to

$$\frac{1}{D} = \frac{1}{D_k} + \frac{1}{D_m}. \quad (14)$$

The diffusivity of H_2S was between 4.08×10^{-3} and $4.91 \times 10^{-3} \text{ cm}^2/\text{s}$ and that of CH_3SH was between 3.43×10^{-3} and $4.11 \times 10^{-3} \text{ cm}^2/\text{s}$ in the transient region.

4.4. Micropore diffusion

Governing model equations with similar boundary and initial conditions have been solved by several investigators. In this research, a numeric method and Fortran least squares program were used to solve the governing equation.

The adsorption diffusivity of H_2S and CH_3SH on the five activated carbons are shown as Table 3.

4.4.1. Effective diffusivity of H_2S adsorbed on activated carbon

The effective diffusivity of H_2S that was adsorbed on activated carbon was between 2.1×10^{-8} and $6.49 \times 10^{-6} \text{ cm}^2/\text{s}$. When the influent concentration of H_2S was increased from 50 to 100 ppmv, the effective diffusivity increased: SAC from 5.35×10^{-6} to $6.49 \times 10^{-6} \text{ cm}^2/\text{s}$, RAC from 1.02×10^{-7} to $1.19 \times 10^{-7} \text{ cm}^2/\text{s}$, FAC from 7.3×10^{-8} to $8.8 \times 10^{-8} \text{ cm}^2/\text{s}$, RAC-N from 2.1×10^{-8} to $2.7 \times 10^{-8} \text{ cm}^2/\text{s}$, and FAC-N from 2.1×10^{-8} to $4.0 \times 10^{-8} \text{ cm}^2/\text{s}$.

4.4.2. Effective diffusivity of CH_3SH adsorbed on activated carbon

When the influent concentration of CH_3SH was increased from 50 to 100 ppm, the effective diffusivity increased: SAC from 5.02×10^{-6} to $5.78 \times 10^{-6} \text{ cm}^2/\text{s}$, RAC from 1.36×10^{-7} to $1.42 \times 10^{-7} \text{ cm}^2/\text{s}$, FAC from

3.21×10^{-7} to $3.95 \times 10^{-7} \text{ cm}^2/\text{s}$, RAC-N from 7.2×10^{-8} to $8.7 \times 10^{-8} \text{ cm}^2/\text{s}$, and FAC-N from 1.03×10^{-7} to $1.27 \times 10^{-7} \text{ cm}^2/\text{s}$.

Results indicated that the larger the pore diameter, the larger the effective diffusivity and the higher the influent concentration, the higher the effective diffusivity.

The effective diffusivity of H_2S and CH_3SH on RAC-N from 30 to 200 ppmv is shown as Fig. 1.

When the influent concentration of H_2S was increased from 30 to 200 ppmv, the effective diffusivity of H_2S adsorbed on RAC-N increased from 1.63×10^{-8} to $3.89 \times 10^{-8} \text{ cm}^2/\text{s}$. When the influent concentration of CH_3SH was increased from 30 to 200 ppmv, the effective diffusivity of CH_3SH adsorbed on RAC-N ranged from 5.31×10^{-8} to $1.03 \times 10^{-7} \text{ cm}^2/\text{s}$.

Given the same influent concentrations of H_2S and CH_3SH , the effective diffusivity of CH_3SH was greater than that of H_2S . This can be attributed to methyl mercaptan having both a polar functional group (S–H) and a non-polar functional group (C–H). Methyl mercaptan tends to be adsorbed more easily on activated carbon than H_2S . Additionally, a high concentration of adsorbate in the micropores of the activated carbon may increase the van der Waal's force (due to condensation). The saturation vapor pressure is 19.807 atm for H_2S and

Fig. 1. Effective diffusivity of H_2S and CH_3SH onto RAC-N. The adsorption temperature was controlled at 25°C.

Table 3
Effective diffusivity of H_2S and CH_3SH on activated carbons

Adsorbents	H_2S (cm^2/s)		CH_3SH (cm^2/s)	
	50 ppmv	100 ppmv	50 ppmv	100 ppmv
SAC	5.35×10^{-6} (0.96) ^a	6.49×10^{-6} (0.97)	5.02×10^{-6} (0.90)	5.78×10^{-6} (0.91)
RAC	1.02×10^{-7} (0.91)	1.19×10^{-7} (0.99)	1.36×10^{-7} (0.92)	1.42×10^{-7} (0.90)
FAC	2.1×10^{-8} (0.92)	8.8×10^{-8} (0.97)	3.21×10^{-7} (0.92)	3.95×10^{-7} (0.92)
RAC-N	2.7×10^{-8} (0.89)	2.7×10^{-8} (0.94)	7.20×10^{-8} (0.91)	8.7×10^{-8} (0.92)
FAC-N	2.1×10^{-8} (0.92)	4.0×10^{-8} (0.94)	1.03×10^{-7} (0.92)	1.27×10^{-7} (0.91)

^a Regression coefficient of adsorption curve (experiments) and fitting curve (diffusion model).

is 1.943 atm for CH₃SH at 25°C (Reid et al., 1988). These values indicate that the CH₃SH is more easily condensed in the micropores of activated carbon. Therefore, the effective diffusivity of CH₃SH is larger than that of H₂S.

5. Conclusions

Influent concentrations of H₂S and CH₃SH were 30 to 200 ppmv, the effective diffusion coefficients were 10⁻⁸ to 10⁻⁶ cm²/s. CH₃SH exhibited an effective pore diffusion coefficient greater than that of H₂S at the same adsorbate concentration. This may be attributed to the fact that CH₃SH has both polar (–SH) and non-polar (–CH₃) functional groups with a strong affinity toward the activated carbon surface. Furthermore, the saturation vapor pressure of CH₃SH (1.943 atm) is lower than that of H₂S (19.807 atm) at 298 K. This indicates that CH₃SH is easier to condense in micropore carbon than H₂S. Results indicate that influent concentration and pore structure can affect the effective diffusivity.

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