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HYDROGEN SULFIDE SEPARATION FROM GAS STREAMS USING SALT HYDRATE CHEMICAL ABSORBENTS AND IMMOBILIZED LIQUID MEMBRANES

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ABSTRACT

The salt hydrate tetramethylammonium fluoride tetrahydrate, $[(CH_3)_4N]F{\cdot}4H_2O$, in the liquid state reversibly absorbs large quantities of hydrogen sulfide, for example 0.30 mol H_2S per mole of salt at 50°C and 100 kPa. Gas absorption likely occurs by deprotonation of H_2S to form bisulfide, HS^-, and bifluoride, HF_2^-. The equilibrium constant for the reaction of H_2S with $[(CH_3)_4N]F{\cdot}4H_2O$ is $1.4 \times 10^{-2}\,kPa^{-1}$ at 50°C as determined from absorption/desorption data. The heat of H_2S absorption was surprisingly low, $-0.78\,kcal\,mol^{-1}$. A second salt hydrate, tetraethylammonium acetate tetrahydrate, $[(C_2H_5)_4N]CH_3CO_2{\cdot}4H_2O$, also reversibly absorbs H_2S but with a lower affinity. However, at higher pressures, its H_2S capacity increases more than does that of $[(CH_3)_4N]F{\cdot}4H_2O$ making it more suitable for use as a pressure swing absorbent. Absorption/desorption data are consistent with reaction of one mole of $[(C_2H_5)_4N]CH_3CO_2{\cdot}4H_2O$ for each mole of H_2S and an equilibrium constant of $6.4 \times 10^{-4}\,kPa^{-1}$ mol H_2S per mole of salt. Membranes

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consisting of liquid $[(CH_3)_4N]F\cdot 4H_2O$ immobilized in a microporous support exhibit selective permeation of H_2S from CH_4 and CO_2. Permeabilities of H_2S increased with decreasing feed pressure, consistent with facilitated transport of H_2S. The H_2S/CH_4 selectivities ranged from 140 to 34 and decreased with increasing feed pressure while H_2S/CO_2 selectivities were 8–6. The presence of H_2S in the feed tends to suppress permeation of CO_2, implying that both gases compete for the same carrier species.

INTRODUCTION

The separation of acid gases, principally CO_2 and H_2S, from H_2, CO, and CH_4 containing streams and air is of considerable industrial importance and incurs substantial processing costs (1,2). Our previous efforts in this area have been directed primarily toward the removal of CO_2 from gas mixtures using salt hydrate based absorbents and membranes (3–7). Salt hydrates which reversibly absorb large amounts of CO_2 have relatively basic anions, fluoride or carboxylate, coupled with monovalent cations, and fewer than about six moles of bound water per mole of cation. For example, tetramethylammonium fluoride tetrahydrate, $[(CH_3)_4N]F\cdot 4H_2O$, at 50°C and 100 kPa absorbs 0.28 mol CO_2per mole of salt (6). Salt hydrates such as $[(CH_3)_4N]F\cdot 4H_2O$ proved useful in the development of immobilized liquid membranes which selectively permeate CO_2 from H_2 and CH_4-containing gas mixtures (3–5).

Perhaps more important and certainly more challenging are separations involving hydrogen sulfide, particularly where selectivity vs. CO_2 is desired. Typical applications are the removal of H_2S from various refinery process streams and in its separation from wellhead natural gas (1,2). Hydrogen sulfide removal is most often accomplished by the use of chemical absorbents, particularly alkanolamines. Although alkanolamines are effective for H_2S removal, rather substantial heat energy is required to regenerate the gas-free absorbent. Our interest in the development of more energy efficient and selective alternatives for H_2S removal led to an evaluation of salt hydrates.

RESULTS AND DISCUSSION

Hydrogen Sulfide Absorbents

Molten tetramethylammonium fluoride tetrahydrate, $[(CH_3)_4N]F\cdot 4H_2O$, was found to absorb relatively large amounts of H_2S. For example, at 100 kPa and

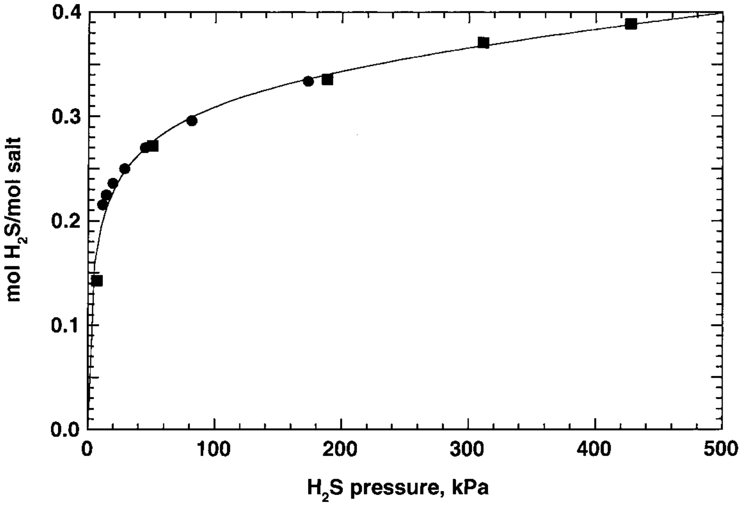

Figure 1. The H_2S absorption/desorption isotherm of $[(CH_3)_4N]F{\cdot}4H_2O$ at 50°C; ■, absorption; ●, desorption.

50°C, 0.30 mol H_2S per mole of salt were taken up. This corresponds to an H_2S concentration of about 2.0 *M* as compared to 0.06 *M* H_2S in water at the same temperature and pressure (8)*. The H_2S absorption/desorption isotherm of liquid $[(CH_3)_4N]F{\cdot}4H_2O$ was determined at 50°C (Fig. 1). Absorption and desorption data points fall along the same smooth curve implying that absorption is fully reversible. The nonlinear dependence of absorption capacity on pressure and the magnitude of gas absorbed strongly imply that H_2S undergoes a reversible chemical reaction with the salt hydrate. The H_2S and CO_2 isotherms of $[(CH_3)_4N]F{\cdot}4H_2O$ are comparable in shape as illustrated by Fig. 2. However, hydrogen sulfide capacities are generally slightly greater than those for CO_2; for example, 0.31 mol H_2S per mole of salt and 0.28 mol CO_2per mole of salt at 100 kPa.

To simplify analytical procedures, a F^--containing salt hydrate which exists as a liquid at room temperature was used to characterize the H_2S absorption products. The salt used was benzyltrimethylammonium fluoride containing 3.7 mol water per mole of salt (9). For analysis, a sample containing 0.42 mol H_2S per mole of salt at 149.4 kPa was prepared. The ^{19}F NMR spectrum of the liquid containing absorbed H_2S indicated the presence of bifluoride, HF_2^- (9), and

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*The concentration of H_2S was calculated by assuming that the volume of the melt was unchanged upon H_2S absorption and that the density of solid and liquid $[(CH_3)_4N]F{\cdot}4H_2O$ are the same, 1.126 g/mL (see Ref. 10).

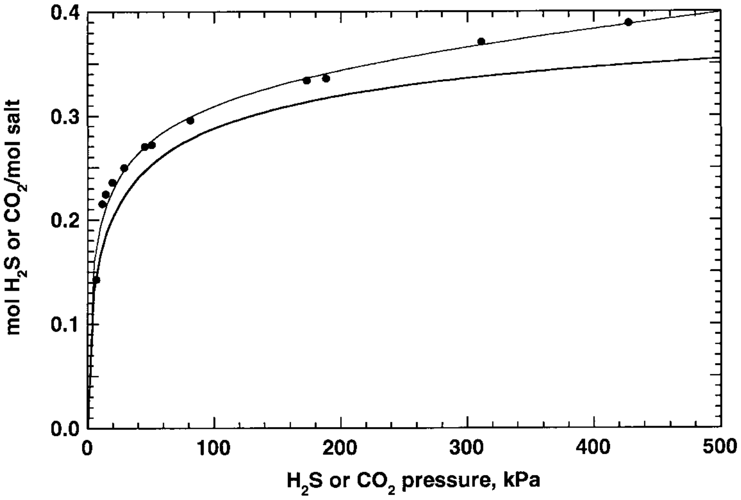

Figure 2. A comparison of the H_2S (■) and CO_2 (curve only) absorption/desorption isotherms of $[(CH_3)_4N]F{\cdot}4H_2O$ at 50°C. For clarity, CO_2 data points are omitted.

implies that H_2S absorption occurs by an acid–base reaction (Eq. 1) forming bisulfide, HS^-. Absorption of CO_2 occurs via an analogous pathway (Eq. 2) yielding bicarbonate and HF_2^- (6).

$$2F^-{\cdot}nH_2O + H_2S = HS^- + HF_2^-{\cdot}2nH_2O \tag{1}$$

$$2F^-{\cdot}nH_2O + CO_2 = HCO_3^- + HF_2^-{\cdot}(2n-1)H_2O \tag{2}$$

This reactivity of $[(CH_3)_4N]F{\cdot}4H_2O$ is a consequence of the strong hydrogen bonds between fluoride ions and water molecules in this limited water environment. The resulting, partially deprotonated water exhibits an enhanced Bronsted and Lewis basicity. It is this enhanced basicity which results in deprotonation of H_2S to HS^- and hydration of CO_2 to HCO_3^-. In water or dilute fluoride salt solutions, however, the much more limited absorption of H_2S and CO_2 is largely the consequence of their purely physical solubility in these media.

The H_2S absorption/desorption isotherm of $[(CH_3)_4N]F{\cdot}4H_2O$ (Fig. 1) is consistent with the stoichiometry of Eq. (1). Capacity–pressure data were adequately fitted to Eq. (3) which consists of a Henry's law term representing the H_2S physical solubility and a second term representing the H_2S chemical capacity of the salt hydrate.

$$\text{capacity} = kP + [K^{1/2}P^{1/2}/(1 + bK^{1/2}P^{1/2})] \qquad (3)$$

In Eq. (3), k is a Henry's law constant, K is the equilibrium constant for Eq. (1), and b is the reciprocal of the chemical capacity at infinite H_2S pressure. If Eq. (1) is strictly obeyed, the value of b is 2 or the maximum capacity is 0.5 mol gas per mole of salt. As described previously for absorption of CO_2, (6) allowing some perturbation of this maximum capacity the following best-fit values were obtained along with estimated errors: k, $1.0 \pm 0.7 \times 10^{-4}\,\text{kPa}^{-1}$·mol H_2S per mole of salt; K, $1.4 \pm 0.4 \times 10^{-2}\,\text{kPa}^{-1}$; b, 2.5 ± 0.2 or a maximum chemical capacity of 0.40 mol H_2S per mole of salt. The calculated physical solubility of 0.069 M at 100 kPa and 50°C is comparable to the H_2S solubility in water, 0.06 M, under the same conditions. The curve in Fig. 1 was calculated based on these parameters and, considering the relatively large estimated errors, the fit to the experimental data is quite satisfactory. As expected from the isotherm comparison in Fig. 2, the equilibrium constant for CO_2 absorption (6), $K_{eq} = 7.2 \times 10^{-3}\,\text{kPa}^{-1}$, is lower than the above for H_2S.

The H_2S heat of absorption for $[(CH_3)_4N]F{\cdot}4H_2O$ was surprising low, $-0.78\,\text{kcal mol}^{-1}$. It is believed that this low heat is a consequence of the relatively strong fluoride–water hydrogen bonds in $[(CH_3)_4N]F{\cdot}4H_2O$ (10). Reaction with H_2S requires some disruption of this hydrogen bonded network and, hence, the net heat of reaction can be viewed as the sum of the exothermic reaction of H_2S with the salt hydrate and the endothermic breaking of $F^-{-}H_2O$ hydrogen bonds. The widely used acid gas absorbents, alkanolamines, have much larger heats for H_2S absorption. For the primary, secondary, and tertiary amines of mono, di, and triethanolamine, the heats are 11.6, 9.6, and 8.1 kcal per mole of H_2S, respectively (2). Thus, regeneration to the gas free salt hydrate absorbent is expected to be more energy efficient than the regeneration of a typical alkanolamine. It is more likely, however, that $[(CH_3)_4N]F{\cdot}4H_2O$ would be better suited to use as a pressure-swing absorbent, for which the low heat is also an advantage. Absorption and desorption pressures of 400 and 50 kPa, respectively, would permit a working capacity of 0.12 mol H_2S per mole of salt hydrate for H_2S separation.

Another salt hydrate, one which is also more suitable for use as a pressure swing absorbent, is tetraethylammonium acetate tetrahydrate, $[(C_2H_5)_4N]CH_3CO_2{\cdot}4H_2O$. Its absorption/desorption isotherm (Fig. 3) shows that it has a lower H_2S affinity than $[(CH_3)_4N]F{\cdot}4H_2O$ but its absorption capacity at higher pressures surprisingly continues to increase while that of $[(CH_3)_4N]F{\cdot}4H_2O$ is nearly constant. The result is that $[(C_2H_5)_4N]CH_3CO_2{\cdot}4H_2O$ has a greater H_2S working capacity. Thus, for an absorption pressure of 400 kPa and desorption pressure of 50 kPa, the working capacity is 0.19 mol H_2S per mole of salt hydrate.

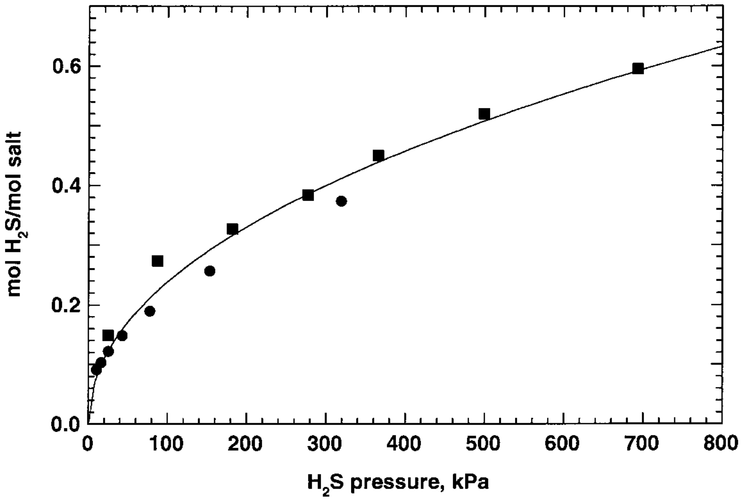

Figure 3. The H_2S absorption/desorption isotherm of $[(C_2H_5)_4N]CH_3CO_2{\cdot}4H_2O$ at 50°C; ■, absorption; ●, desorption.

If the desorption pressure is raised to 100 kPa, the working capacity is still greater than that of $[(CH_3)_4N]F{\cdot}4H_2O$, 0.16 mol H_2S per mole of salt hydrate. At a higher absorption pressure, 600 kPa, along with desorption at 50 kPa, the working capacity is substantially greater, 0.27 mol H_2S per mole of salt hydrate.

Capacity–pressure data for $[(C_2H_5)_4N]CH_3CO_2{\cdot}4H_2O$ could not be adequately described by Eq. (3). Although a reasonable fit of the data was obtained, the H_2S physical solubility was unreasonably large, 0.18 *M* at 100 kPa and 50°C. Therefore, capacity–pressure data were fitted using the 1:1 reaction stoichiometry of Eq. (4).

$$CH_3CO_2^- {\cdot} nH_2O + H_2S = HS^- + CH_3CO_2H{\cdot}nH_2O \qquad (4)$$

Again, data were fitted to a two term expression, a Henry's law term and a chemical capacity term. The following best fit values and estimated errors were obtained: k, $1.6 \pm 0.5 \times 10^{-4}\,kPa^{-1}$ mol H_2S per mole of salt; K, $6.4 \pm 0.8 \times 10^{-4}\,kPa^{-1}$ mol H_2S per mole of salt. The calculated physical solubility is now reasonable, 0.067 *M* at 50°C and 100 kPa.

Absorption of CO_2 by $[(C_2H_5)_4N]CH_3CO_2{\cdot}4H_2O$ was also best described by the 1:1 reaction stoichiometry of Eq. (5) with an equilibrium constant of $2.76 \times 10^{-4}\,kPa^{-1}$ mol CO_2 per mole of salt (6). A comparison of the absorption/desorption isotherms for H_2S and CO_2 of

$$CH_3CO_2^- \cdot nH_2O + CO_2 = HCO_3^- + CH_3CO_2H \cdot (n-1)H_2O \qquad (5)$$

$[(C_2H_5)_4N]CH_3CO_2 \cdot 4H_2O$ (Fig. 4) indicates that the salt has a higher affinity for H_2S in agreement with the above equilibrium constant values.

A unique property of $[(C_2H_5)_4N]CH_3CO_2 \cdot 4H_2O$ is the unusual temperature dependence of its H_2S absorption capacity. As previously reported, (11) cooling $[(C_2H_5)_4N]CH_3CO_2 \cdot 4H_2O$ containing 0.298 mol H_2S per mole of salt hydrate from 50 to 26°C resulted in solidification of the absorbent and near quantitative desorption of H_2S. This modest temperature change can be used as an "on/off" switch for gas absorption, and an alternative method for the removal of H_2S from gas streams.

Facilitated Transport Membranes

Salt hydrates can also be used as the reactive material in facilitated transport membranes as previously reported for separations of CO_2 from methane and hydrogen (3,4). Membranes consisting of liquid $[(CH_3)_4N]F \cdot 4H_2O$ immobilized in the microporous polypropylene support, Celgard 3401® (Celgard Inc., Charlotte, NC, were evaluated for the separation of H_2S from CH_4 and CO_2

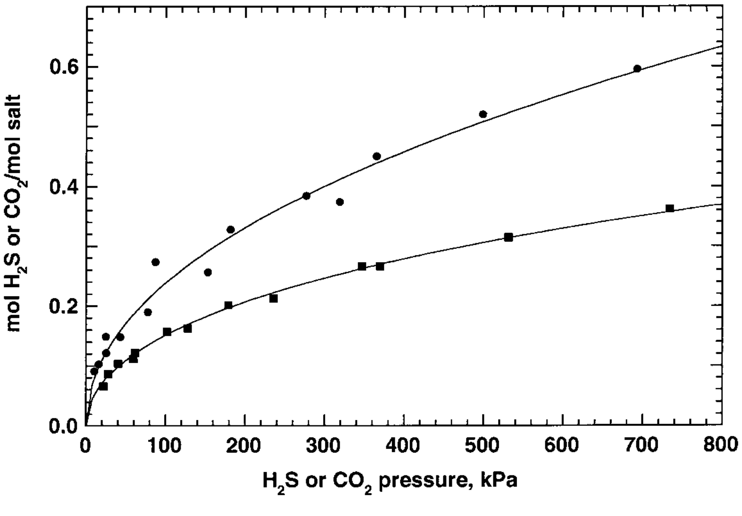

Figure 4. A comparison of the H_2S (●) and CO_2 (■) absorption/desorption isotherms of $[(C_2H_5)_4N]CH_3CO_2 \cdot 4H_2O$ at 50°C.

at 50°C. Permeation testing was performed using humidified feed and sweep gas streams of about equal concentrations of H_2S and CO_2 in CH_4. The dew point of the feed and sweep gases were the same, 10°C, or a relative humidity of approximately 10%. As shown in Table 1, the permeability of CH_4 was largely independent of its feed partial pressure, consistent with its transport by a solution–diffusion mechanism. The permeability of H_2S and CO_2, however, were dependent on their feed partial pressures. Figure 5 clearly shows that decreasing the partial pressure of either gas results in increased permeabilities. Such a pressure dependence is consistent with permeation by a facilitated transport mechanism (12) involving the reversible reaction of the gas and salt hydrate. The H_2S/CH_4 selectivity ranged from 140 to 34 and decreased with increasing feed pressure. The H_2S/CO_2 selectivity was greater than that generally observed for conventional polymeric membranes, 4, (13–16) and ranged from about 8 to 6.

Facilitated transport of H_2S involves reaction of the gas with $[(CH_3)_4N]F\cdot 4H_2O$ as represented by Eq. (1). At the feed interface where the H_2S pressure is highest, Eq. (1) proceeds in the forward direction and H_2S is deprotonated to form bisulfide and bifluoride ions. These ions diffuse across the membrane to the low pressure permeate side of the membrane where Eq. (1) proceeds in the reverse direction and H_2S is liberated into the gas phase. That, in addition to HF_2^- ions, deprotonation of H_2S results in HF is unlikely based on the experimental results. Hydrogen fluoride is sufficiently volatile that if formed it would be removed from the membrane in the sweep stream. This would result in a change

Table 1. Permselective Properties of a $[(CH_3)_4N]F\cdot 4H_2O$/Celgard® Membrane at 50°C

	P_o (Barrers)[a]			Selectivity	
Feed Pressure (kPa)	H_2S	CO_2	CH_4	H_2S/CO_2	H_2S/CH_4
115.8	813	109	5.8	7.5	140
180.5	443	54	4.3	8.2	102
268.7	282	35	7.4	8.0	38
333.5	227	32	6.7	7.2	34
469.3	192	30	5.7	6.4	34

Conditions: Feed—5.13% H_2S, 5.10% CO_2 in CH_4, 10.0 sccm; sweep-helium, 10.0 sccm; feed and sweep gases passed through water bubbler at 10°C; 9.95% relative humidity.

[a] 1 Barrer = 10^{-5} sccm cm/cm^2 sec cmHg.

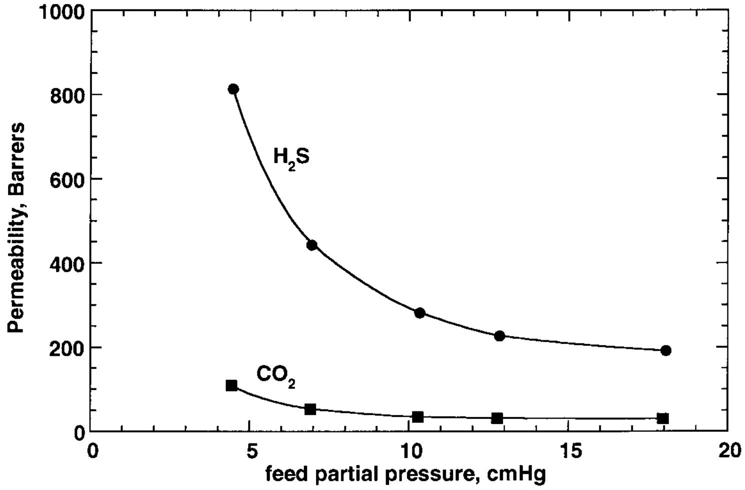

Figure 5. Permeabilities of a $[(CH_3)_4N]F{\cdot}4H_2O$ in Celgard 3401® membrane at 50°C as a function of feed partial pressures.

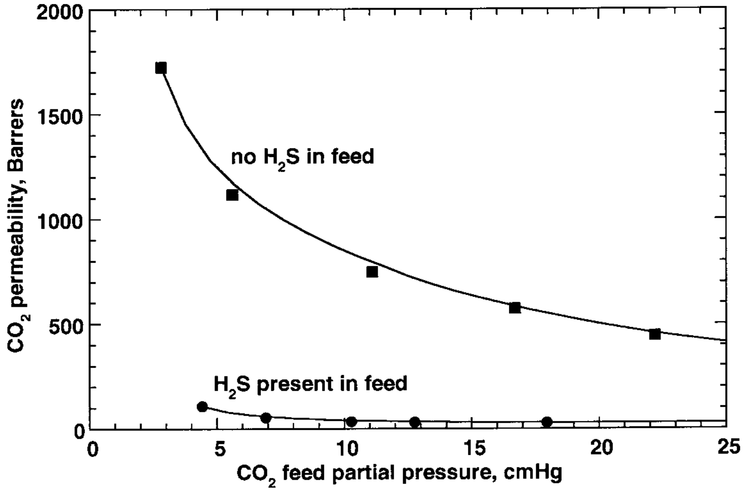

Figure 6. A comparison of the CO_2 permeability for $[(CH_3)_4N]F{\cdot}4H_2O$ in Celgard 3401® membranes in the presence and absence of H_2S in the feed gas.

in the membrane permselectivity properties with time and no such change was observed.

As has been noted previously for fluoride-containing polyelectrolyte membranes, (9) the presence of H_2S in the feed tends to suppress permeation of CO_2. Figure 6 shows that the CO_2 permeability in the absence of H_2S is 10–20-fold greater than that in its presence implying that both gases permeate by a common facilitated transport pathway and compete for the same carrier species, (12,16) hydrated fluoride ions. Considering that the equilibrium constants for reaction of $[(CH_3)_4N]F{\cdot}4H_2O$ with H_2S and CO_2 are of comparable magnitude, that H_2S competes more effectively for the carrier species cannot be ascribed only to reaction thermodynamics. Rather, the preference for permeation of H_2S over CO_2 is likely due to reaction kinetics. Permeation of CO_2 requires a relatively slow hydration step (CO_2 to HCO_3^-) compared with the fast deprotonation of H_2S to HS^-.

CONCLUSIONS

Tetramethylammonium fluoride tetrahydrate reversibly absorbs relatively large amounts of H_2S; for example, 0.30 mol H_2S per mole of salt at 100 kPa and 50°C. Absorption of H_2S occurs by an acid–base reaction resulting in its deprotonation to form bisulfide, HS^-, and bifluoride, HF_2^-, with an equilibrium constant (K for Eq. (1)) of $1.4 \times 10^{-2}\,\text{kPa}^{-1}$ at 50°C. Another salt hydrate, tetraethylammonium acetate tetrahydrate, also absorbs H_2S reversibly. Although it has a lower affinity for H_2S, its isotherm is more "linear" and thus more suitable for use as a pressure swing absorbent for the recovery of H_2S from process gas streams. Absorption occurs by a one-to-one reaction stoichiometry with an equilibrium constant of $6.4 \times 10^{-4}\,\text{kPa}^{-1}{\cdot}\text{mol}$ H_2S per mole of salt. Membranes consisting of liquid $[(CH_3)_4N]F{\cdot}4H_2O$ immobilized in Celgard 3401® selectively permeate H_2S from gas mixtures by a facilitated transport mechanism. The H_2S/CH_4 selectivities ranged from 140 to 34 and decreased with increasing feed pressure. The presence of H_2S in the membrane feed tends to suppress permeation of CO_2, implying that the two gases compete for a common carrier species.

EXPERIMENTAL

Materials

The salts $[(CH_3)_4N]F{\cdot}4H_2O$ and $[(C_2H_5)_4N]CH_3CO_2{\cdot}4H_2O$ were obtained from Aldrich Chemical Company, Milwaukee, WI and were used without further purification.

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Methods

The H_2S absorption/desorption isotherms were determined using standard manometric techniques similar to those previously described for CO_2 (6). A known quantity of the tetramethylammonium fluoride was added to a stainless steel reaction vessel and attached to a manifold of known volume. The reaction vessel was maintained at a constant temperature and the contents were stirred. In-line pressure transducers were used to determine pressures. The volume of the system available to gas was determined by expansion of helium from the manifold at known pressure into the reaction vessel. The water vapor pressure of the salt hydrate was determined by exposing the melt to a static vacuum and allowing the pressure to reach equilibrium. It was assumed that the vapor pressure remained constant throughout the experiment and subsequent H_2S pressures were corrected accordingly. Heats of absorption were obtained as described previously (6).

Membranes consisting of liquid salt hydrates supported in Celgard 3401® were prepared as described previously (3). The methods and apparatus used to determine permselective properties have been described in detail elsewhere (3). A pre-mixed feed gas, 10 sccm, was passed over one surface of the membrane while a stream of helium, 10 sccm, was passed over the other surface. Gas streams were humidified by passage through a series of water bubblers maintained at 10°C. The composition of the permeate stream was analyzed at regular intervals using gas chromatography (GC). Membrane area was 3.77 cm^2 and membrane thickness was 2.5×10^{-3} cm, the thickness of the Celgard 3401® support. Permeabilities were corrected for the porosity and tortuosity of the support, 0.50 and 1.25, respectively.

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