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EFFECTS OF PARTICLE SHAPE AND SIZE DISTRIBUTION ON SORPTION AND FLOW PERFORMANCE IN ELECTRICALLY STABILIZED EXPANDED BEDS

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ABSTRACT

Use of the electrically stabilized expanded bed is an approach to the improvement of the performance of processes in beds of solid sorbents. Particle motion in a fluidized bed of nonconducting particles such as molecular sieves is halted when the bed is "frozen" by a strong electric field imposed across the bed. This allows sorption performance to approach that of fixed beds while maintaining the low pressure drops which are characteristic of fluidized beds. Beds with axial and radial electrode geometries function equally well in the sorption of carbon dioxide (CO_2) from nitrogen (N_2). The effects of particle shape and particle size distribution on bed stabilization are presented. Particle shape was varied from spherical to the irregular forms that are characteristic of crushed particles. The sorption performance was not appreciably different with particles having various shapes.

Particles with a size range from 200 to approximately 1200 μm were blended from individual spherical fractions to create mixtures with peak distributions at both ends of this range. To a first approximation, the distribution of particle sizes did not affect sorption performance. These results demonstrate that both irregularly shaped particles and multiple-sized particles can be stabilized in electric fields, and the stabilized beds behave much like fixed beds of the same materials except that the pressure drops across the beds can be significantly less than those for fixed beds. However, the rate of adsorption in the system used for this study is controlled by diffusion within the particles (actually within the micropores of crystals); so the study did not address the effects of electric stabilization on the gas-film resistance.

INTRODUCTION

Fixed and, to a lesser extent, fluidized adsorption beds are used extensively in the chemical process industries for gas and liquid purification and for the separation of mixtures (1,2). Molecular sieves are used in a large fraction of these processes (3). A balance must often be drawn in such processes between high throughput operations and excessive energy losses caused by the pressure drop required to force the fluid through the sorbent particles. Fixed beds of sorbent are usually necessary to minimize axial mixing (dispersion) of the fluid and to maintain sharply defined concentration gradients in the bed. This process allows sharply defined concentration profiles in both the solid and the fluid phases and is usually achieved through confinement of the packed beds by fixed screens. On the other hand, fluidized beds being expanded (as much as 50%) exhibit significantly lower pressure drops; and, during regeneration, they heat more rapidly and uniformly, allowing more rapid regeneration (4).

Several methods have been studied for restraining or freezing particle motion in fluidized beds so that the sorption performance of the bed will approach that of fixed beds, while retaining the low pressure drops associated with the fluidized bed. Johnson and Melcher (5) suggested using an electrically stabilized bed as a fine-particle filter. Magnetic stabilization has been developed by Rosensweig, Siegell, and others (6-9). The use of magnetic stabilization in improving the characteristics of chromatographic columns has been recommended by Siegell et al. (10). Magnetic stabilization requires that the granular medium be magnetic or paramagnetic, while electrical stabilization requires only that the particles be an electrically nonconducting material with a dielectric constant greater than that of the fluid.

A concept having the positive features of both types of beds has been developed and investigated in detail at Oak Ridge National Laboratory (ORNL) and reported in a number of publications (11,12). The electrically stabilized expanded bed (ESEB) was developed to improve fluidized-bed performance without sacrificing the pressure-drop characteristics of the fluidized bed. The ability to switch to normal fluidized-bed operation is retained so that the bed can be fluidized during regeneration. In addition, the concept offers the possibility of developing some interesting moving-bed configurations (11).

Both axial and radial electrode geometries for cylindrical ESEBs were studied at ORNL (see Fig. 1). While stabilization can be achieved with either of the electrode orientations, the nonuniform field gradient inherent with the radial field results in a tendency for dielectric particles to drift toward the central electrode, where the field gradient is a maximum. Furthermore, if particles accumulate any net electrical charge, they will be

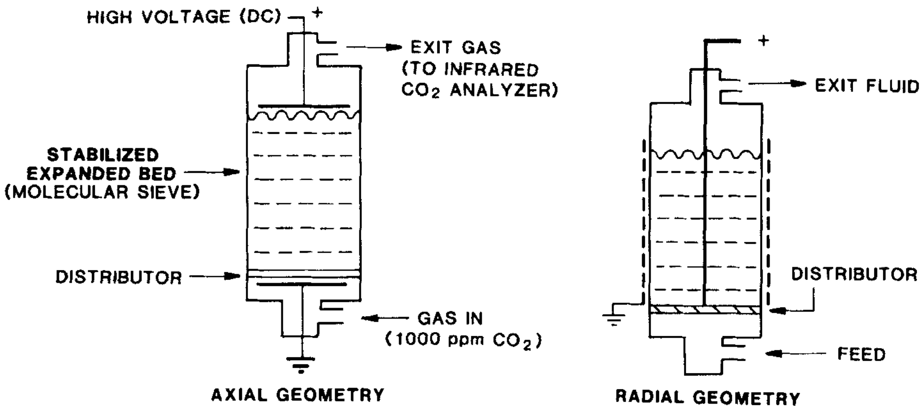

Fig. 1. The ESEB concept shown in axial and radial configurations.

attracted to the electrode of opposite charge when dc fields are used. Either of these phenomena can lead to gas channeling with radial fields, but not with axial fields. The effects of particle charge can be reduced by using ac fields (12).

While most of the work we have reported is with gases and with axial geometry, any bed of fluidized particles in an upflowing stream of nonconductive fluid can be stabilized by the application of a high dc or ac field. For studies with molecular sieve particles in dry nitrogen, a field of about 1 to 3 kV/cm was necessary to stop all particle motion. Using an ac field has two potential advantages. First, it eliminates electrostatic effects, which can be detrimental to bed performance, as noted earlier. Second, ac fields permit the convenient use of alternating currents to measure the degree of electrical polarization.

In our earlier studies, a bed with axial electrodes was used to study the hydrodynamics and sorption properties of the stabilized bed. Spherical Davison 4A molecular sieves in narrow size ranges, from 200 to 1200 μm, were used to sorb CO_2 from a nitrogen stream. The fixed-bed adsorption equation was solved by orthogonal collocation, incorporating a Freundlich isotherm and a lumped-parameter transport resistance. Results indicated that the controlling factor for the process was micropore diffusion. Further, it was found that, with stabilized beds under the conditions studied, pressure-drop improvements of 30 to 50% over conventional fixed-bed operation were achieved without significant loss of mass-transfer efficiency.

This paper focuses on the investigation of two normal features of more practical beds: variable particle shape and nonuniform particle size distribution. In many typical industrial beds of sorbents, the particles tend to be somewhat irregular in shape, raising the possibility that they might not be amenable to stabilization. In addition, nonuniform particle size distributions virtually always exist in industrial systems. Therefore, these conditions were examined and compared with existing results. There is no practical way to test all the types of particle size and shape distributions which could be encountered, but a few representative cases were investigated. Within the size range of the sieves with which we worked (200 to 1200 μm), the stabilization behavior was very similar to that which we had previously reported (12). There appeared to be no visible segregation of distributed systems, and the irregularly shaped particles showed no extraordinary clumping or channeling characteristics. Similarly, the hydrodynamics of these parametric studies was quite similar to that reported previously.

THEORETICAL BACKGROUND

The hydrodynamics of fixed and fluidized beds has been reviewed extensively by Froment and Bischoff (13) and by Harriott and Simoni (14), while the section specifically pertaining to stabilized expanded beds has been explored in earlier publications (11,12). It is also desirable to examine the sorption background as presented by Sherwood, Pigford, and Wilke (2) and in a recent monograph by Ruthven (15) for a comprehensive review of the theory of sorption in packed beds. Our primary interest in this study is the comparison of the behavior of stabilized expanded beds with the two extremes: fixed beds and fluidized beds. Because we are primarily dealing with dilute solutions of CO_2 in an inert medium, isothermal, low-flux transport may be assumed. The convective diffusion equation in a bed for this case is

$$-D_L \frac{\partial^2 c}{\partial z^2} + v \frac{\partial c}{\partial z} + \frac{\partial c}{\partial t} + \left(\frac{1 - \varepsilon}{\varepsilon}\right) \frac{\partial \bar{q}}{\partial t} = 0 \ , \tag{1}$$

where c and $\bar{q}$ are the concentrations in the bulk gas phase and the average concentration in the solid phase, respectively. At any point z in the bed, the value of $\bar{q}$ is given as

$$\bar{q} = \left(\frac{3}{R_p^3}\right) \int_0^{R_p} R^2 q dR \ . \tag{2}$$

The boundary conditions for the adsorption case are given as

$$\begin{cases} t < 0, \ q(R,0,z) = c(0,z) = 0 \\ t \geqslant 0, \ c(t,0) = c_0 \ . \end{cases} \tag{3}$$

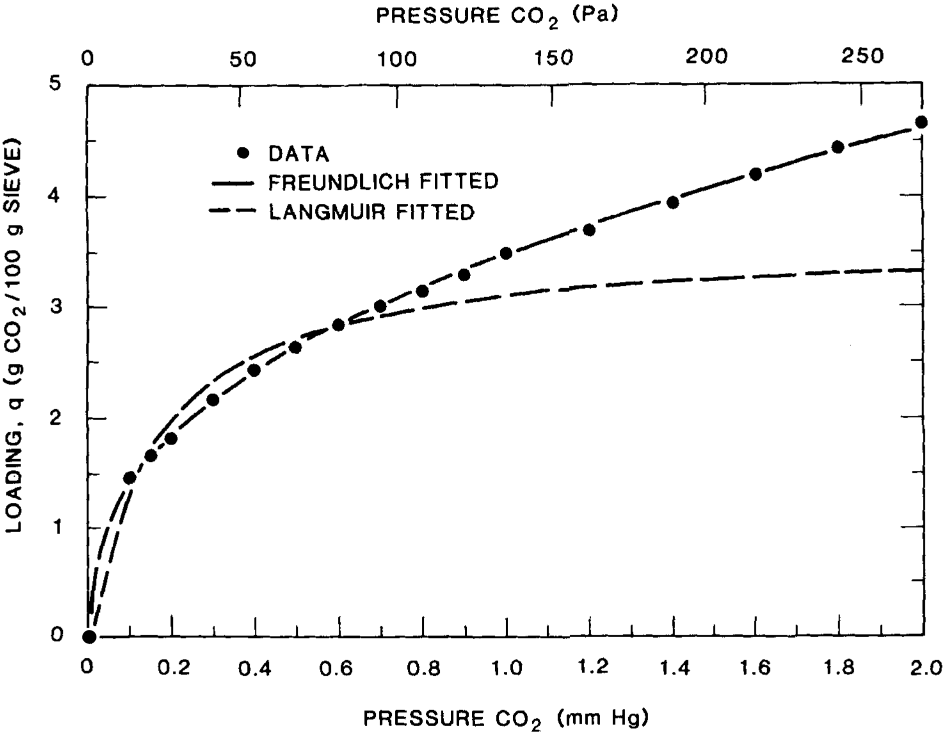

Fig. 2. Modeling sorption equilibrium data for CO_2 on Davison 4A molecular sieves using Freundlich and Langmuir isotherms.

For this experimental study, it is necessary to apply the equilibrium relationship between the gas phase and the solid surface from zero partial pressure to about 1 mm Hg. Analytical solutions have been presented by many workers for a linear isotherm (15); however, the sorption data provided by the Davison Division of W. R. Grace for CO_2 on their 4A molecular sieves indicate significant nonlinearity at 25°C even in this range. As is indicated in Fig. 2, a Langmuir isotherm could not acceptably fit the data. The Freundlich isotherm,

$$q = Kc^{1/n} , \tag{4}$$

gave an excellent correlation with the data over the range studied. For partial pressures up to 2 mm Hg, a K value of 0.0348 g CO_2 per gram of sieve per mm $Hg^{2.5}$ was found, where n is 2.5. The correlation coefficient was 0.999. The adsorption of the "inert" N_2 on Davison 4A molecular sieves has been found to be inconsequential as compared with that of CO_2 (16).

Solutions of the convective diffusion equations for this isotherm have been presented by Tien and Thodos (17) and Kyte (18). Both were numerical studies, and, unfortunately, neither yielded results with sufficiently high n-values. Therefore, it was necessary to solve the convective diffusion equations for packed and stabilized beds using numerical methods. The orthogonal collocation method proposed by Villadsen and Michelsen (19) and more recently used by Howard, Carta, and Byers (20) and by Prazniak and Byers (21) was modified to simulate the adsorption-desorption problem in a stabilized bed. The results of the simulation were used to produce the solutions with which the data were compared. Following Garg and Ruthven (22), the diffusion characteristics of the porous molecular sieve were computed by the following relationship for a case where micropore diffusion controls the adsorption rate:

$$\frac{\partial q}{\partial t} = \frac{1}{r^2} \frac{\partial}{\partial r} \left(r^2 D_c \frac{\partial q}{\partial r} \right) , \tag{5}$$

where D_c is the intracrystalline diffusion coefficient and r is the radial position in a spherical crystal. Yucel and Ruthven (23) found the diffusivity of CO_2 on 4A molecular sieves to be approximately 10^{-8} to 10^{-10} cm^2/s.

More generally, one must account for several dispersive effects in a bed, including axial dispersion, gas-film mass transfer, pore diffusion, and intracrystalline diffusion. While this problem can be solved numerically, it was decided to follow the procedure developed by Glueckauf (24), who found that for systems with linear isotherms a good approximation to the overall rate of mass-transfer resistance could be obtained by adding the individual contributions as follows:

$$\frac{1}{Kk'} = \frac{D_L}{v^2} \left(\frac{1 - \varepsilon}{\varepsilon} \right) + \frac{R_p}{3k_f} + \frac{R_p^2}{15\varepsilon_p D_p} + \frac{r_c^2}{15KD_c} . \tag{6}$$

Raghavan and Ruthven (25) found that, within limits, one could apply this concept directly to nonlinear cases. Therefore, the solution shown in Fig. 3, which was found by the orthogonal collocation methods just described, strictly speaking, describes the effect of a film resistance to mass transfer in a bed where the Freundlich isotherm shown in Fig. 2 describes the equilibrium relationship. As is shown in Eq. (6), we may take a broader

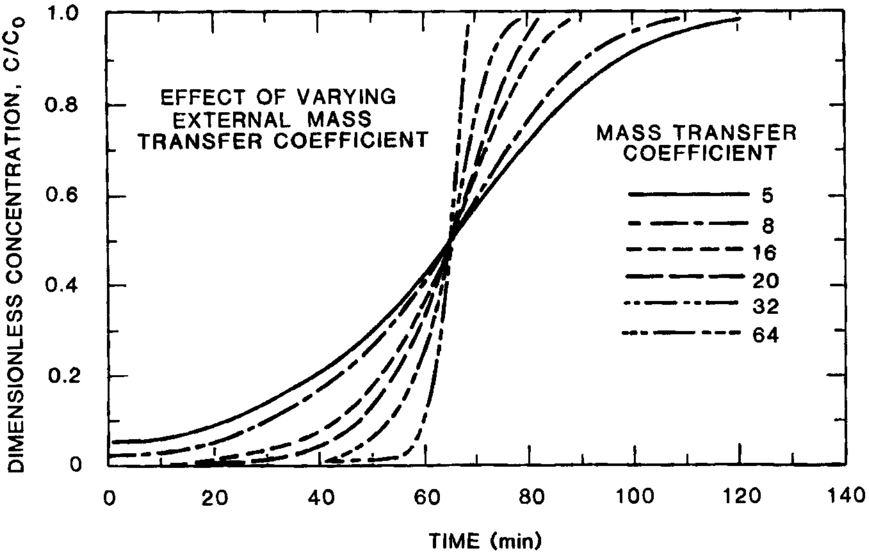

Fig. 3. Theoretical sorption performance of a sorption bed showing the influence of a single mass-transfer coefficient to model the dispersive characteristics.

interpretation by taking an average value of the q-c slope for the isotherm. While results for specific situations would be made more precise, the current approach gives an approximation that can be easily used with acceptable accuracy.

EXPERIMENTAL EQUIPMENT

A schematic diagram of the experimental apparatus used in the current study is given in Fig. 4. The central feature of the apparatus is the stabilized bed, which consists of a cylindrical glass column with a 4-cm inside diameter and an approximate length of 25 cm overall. The beds, which were Davison 4A molecular sieves in various size ranges from 200 to 1200 μm, were about 10 cm high in their unexpanded state. The gas phase entered through a fritted-glass distributor that was the diameter of the column itself. The column was fitted with pressure taps in the top of the column, just above the fritted-glass distributor.

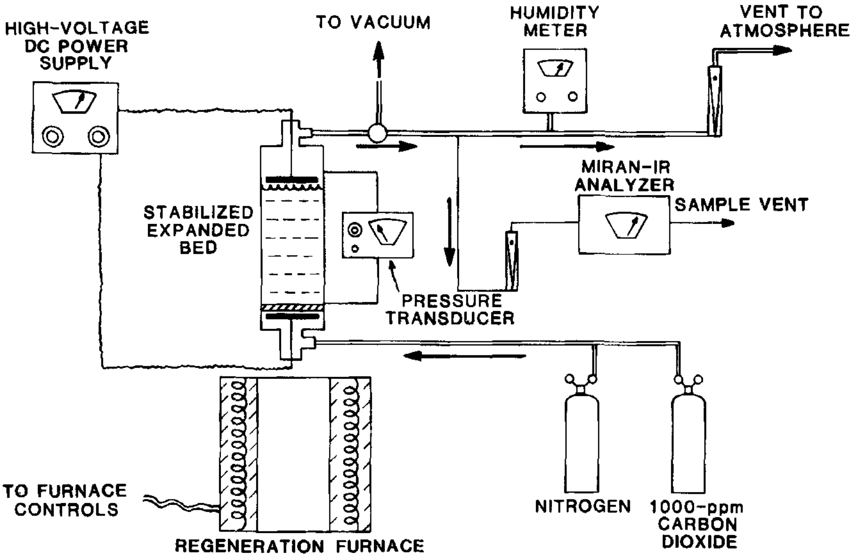

Fig. 4. Schematic diagram of an electrically stabilized expanded-bed (ESEB) system.

The electrodes had different geometries, depending on the configuration of the experiment. An axial configuration is shown in Fig. 1, with the positive electrode attached to a screen above the bed and the ground attached to a second screen below the fritted glass. Sufficient space was left above the bed to allow for expansion, without the positive electrode of the bed being engulfed. The power supply consisted of a Hyposonics 30-kV dc generator equipped with a milliammeter. In the radial configuration, the ground electrode was a wire mesh wrapped around the entire expanded length of the bed outside the glass. The positive electrode was a 3-mm stainless steel rod that extended from the top of the column to the fritted-glass distributor. Although similar results were usually obtained by both approaches, most of the studies were performed with the axial configuration because it was less prone to the difficulties mentioned earlier.

The experiments in this program involved flow of bottled N_2 and N_2 containing 1000 ppm CO_2 through the molecular sieve-filled beds. The exit CO_2 concentration was continuously measured using a Miran 101 infrared analyzer. There was no observed difference in the hydrodynamics or electrical properties of these gases. The moisture content of the gas stream was too low to affect the performance of the bed in all of the experiments (5). Pressure drop was monitored by calibrated pressure transducers, and the flow rate was observed with rotameters. The absolute pressure in the bed was within a few pascals of ambient. Using a system of three-way valves, it was possible to reverse the flow direction in the bed so that fixed-bed runs could be made in downflow while the fluidized experiments were conducted in upflow. Stabilized-bed runs were performed in both directions.

Important auxiliary equipment used in this experiment included a tubular furnace that regenerated the molecular sieves. The regeneration procedure involved isolation of the system followed by evacuation to a moderate vacuum. The furnace was moved into position around the bed, and the system was heated to 400°C. To ensure complete regeneration, the bed was held at this temperature and pressure for at least 4 h.

A typical stabilized-bed experiment was performed in the following manner. A flow of pure nitrogen was established at the rate selected for the experiment. The bed was stabilized by the application of an electric field sufficient to freeze the motion of the bed. For a 10-cm electrode separation, fields between 10 and 20 kV were required to arrest the particle motion. The intensity required to stabilize the bed was a function of the parameters that affected fluidization, such as density, size, and shape (distributions) of the particles in addition to the dielectric properties of the material. When stabilization was complete, the adsorption experiment was begun by introducing feed from the cylinder with the N_2 containing 1000 ppm CO_2. The rate of flow during the run was often different from that used for initial fluidization. During the experiment, the CO_2 exit concentration, pressure drop, flow rate, temperature, and bed humidity were continuously monitored. In most cases, the feed was discontinued when approximately 99% of bed saturation was achieved. Desorption experiments were performed by passing pure nitrogen through the bed and monitoring the concentration of the outlet stream; these experiments are not reported here. The Miran IR analyzer was calibrated during each run with pure N_2 and the feed gas mixture. In most runs where the concentration was not deliberately manipulated, the feed concentration varied less than 1%.

EXPERIMENTAL RESULTS AND DISCUSSION

For each stabilized-bed run reported here, there was a complete series of four curves comparing the extremes of operation of the bed. An example of one of these series is given in Fig. 5. Sorption data for four different conditions are depicted in the graph. A 5.0-L/min gas stream containing 1000 ppm CO_2 in N_2 enters a 69-cm-tall bed of 500- to 600-μm 4A molecular sieves. The outlet concentration traces show internal consistency in that at 500 ppm the curves cross and the time is that predicted by equilibrium

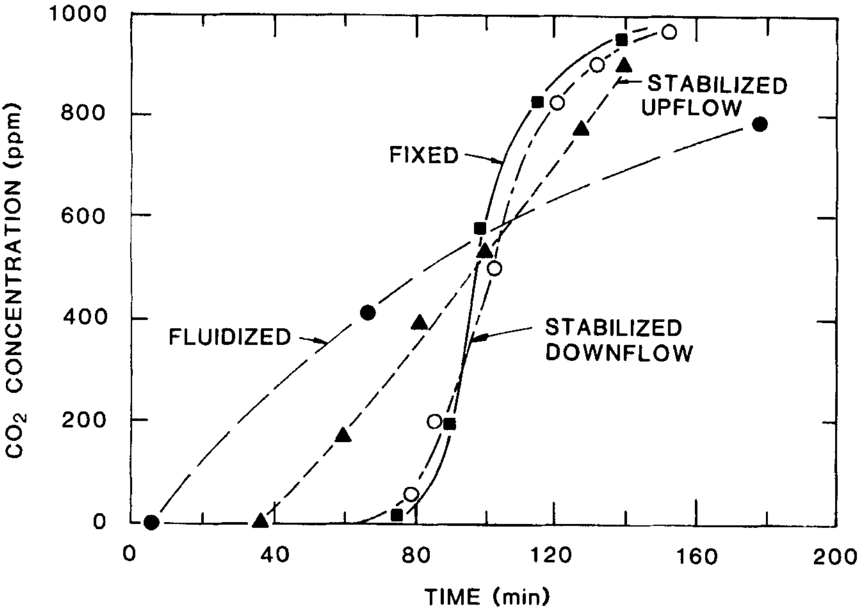

Fig. 5. Sorption of CO_2 in 500- to 600-μm spherical Davison 4A molecular sieves using different configurations in an ESEB.

data. The "fixed" run is in downflow and represents the normal sorption experiment, while the "fluidized" run is at the same feed flow rate in upflow. This results in moderate fluidization of the bed. Our results, which show immediate breakthrough and a long tail, are typical of fluidized-bed operation. If one chooses to stabilize the fluidized bed with an electric field and then performs the adsorption experiment ("stabilized upflow"), a behavior that is intermediate between the fixed- and fluidized-bed operation is observed. This indicates that there is some channeling or bypassing or possibly particle motion. Under different flow and field conditions, the position of the adsorption curve can be closer to either the fixed-bed or the fluidized-bed curves. However, if, subsequent to stabilization, one reverses the flow to the fixed-bed configuration, the "stabilized downflow" run is the result. This sorption curve is not significantly different from the fixed-bed sorption run, but exhibits a significantly lower pressure drop, indicating that stabilization has been retained. The expanded bed has a significantly higher bed void fraction (~10% greater), indicating that axial dispersion must not be a significant factor here.

In beds of small particles, a range of particle sizes is usually present. To learn the effect of particle size distribution on sorption behavior in stabilized downflow, synthetic size distributions were made with spherical particles in the particle range between 200 and 1200 μm. Fines were carefully removed to eliminate spurious results that would occur from having order-of-magnitude smaller particles present. Three distributions were selected. First, a synthetic distribution that had a balance between the numbers of small particles and the large ones was typical of the as-received material. Secondly, a distribution was prepared in which more than 90% of the particles were smaller than 400 μm. Finally, a distribution with only larger particles (800 to 1200 μm) was synthesized. The electric field required for bed stabilization was not affected meaningfully by using wide particle size distribution, and there was no visual evidence of bed segregation. Pressure drops were typically 30 to 50% lower than those observed in fixed-bed operations with the same particles. This is similar to the pressure-drop reduction that we found in previous studies.

Sorption results for the skewed distributions are shown in Fig. 6. Minor differences in the positions of the curves can be attributed to differences in feed-gas concentrations that were not normalized out of these curves. The supply of mixed gases was not as uniform (from cylinder to cylinder) as desired. Normalization, based on feed-gas concentration, was incorporated in the reports of the remaining experiments in this series. The overall result is that, within the accuracy of the experiments, the curves are the same. Since the rate of adsorption of CO_2 by molecular sieves is likely to be controlled by diffusion within the micropores of the sieve crystals, the similarity of the curves is not necessarily a surprising result. However, it shows that neither the particle size nor the particle size distribution affects the ability of electric fields to restrain particle motion and allow expanded beds to have fixed-bed-like adsorption performance with a significantly reduced pressure drop.

It is difficult, in practice, to control the shape of particles that are used in sorption operations. It is desirable that ESEBs be capable of operation with all shapes of particles. As an experimental situation, three different shapes of particles were prepared; these are shown in Fig. 7. All are in the 500- to 600-μm range and are described as spherical, aspherical, and crushed. An as-received batch of Davison 4A nominally spherical sieves was screened, and the 500- to 600-μm particles were selected for use. They were then placed in a particle classifier (11), and the 15% of the particles that were most nearly spherical were selected as the "spherical" fraction and the least spherical 15% were selected as the "aspherical" fraction. Figure 7 shows that there is a significant difference in shape, with both the near-spherical and the aspherical fractions having many broken particles. For the

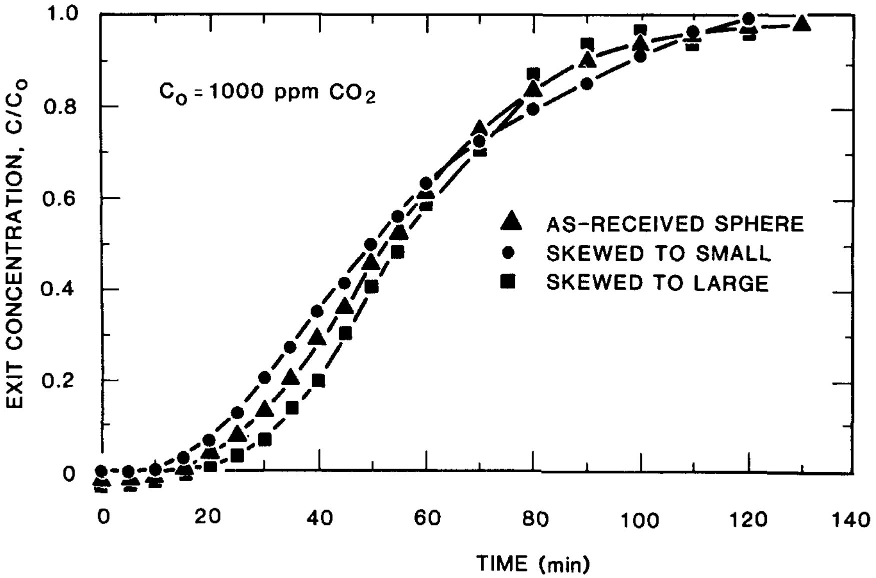

Fig. 6. Effect of size distribution on the sorption characteristics of an ESEB of Davison 4A molecular sieves.

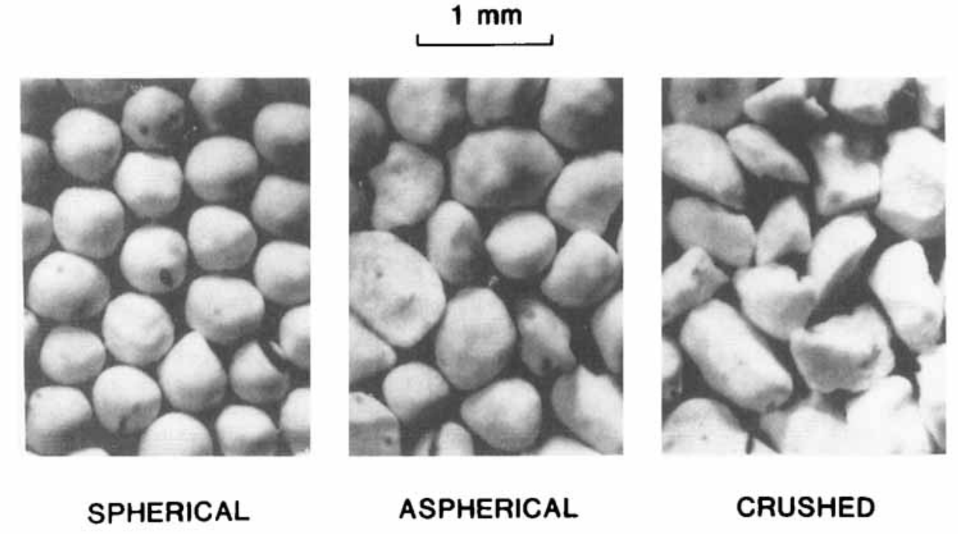

Fig. 7. Photograph of 500- to 600-μm Davison 4A molecular sieves showing the three shapes for which the results are given in Figs. 8 to 10.

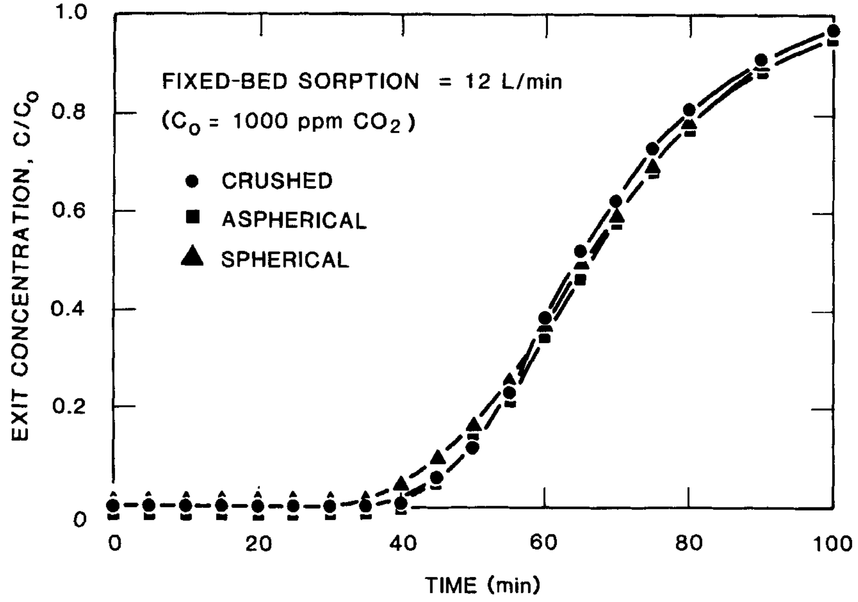

Fig. 8. Effect of particle shape on the sorption performance of a fixed bed of Davison 4A molecular sieves.

crushed case, large particles from the same batch were crushed mechanically, and the resulting mixture was screened to collect the 500- to 600-μm fraction. Beds of identical amounts of material were tested for pressure drops, which showed the expected patterns, based on our previous experiments.

Bed stabilization tests indicated little difference in performance between the spherical and aspherical particles. On the other hand, the fluidized crushed-particle beds required significantly higher electric fields (2.0 vs 1.5 kV/cm) for complete stabilization. These beds also appeared to operate well only over a smaller range of flow rates. Extremes of shape should probably be avoided unless all particles are identical, such as is the case with cylindrical pellets.

Comparison of the sorption performance of the various shapes is shown in Figs. 8 to 10, which are, respectively, fixed, fluidized, and stabilized downflow runs. In all cases, the results are essentially identical. This outcome lends further credence to the concept that the sorption process in all cases is controlled by

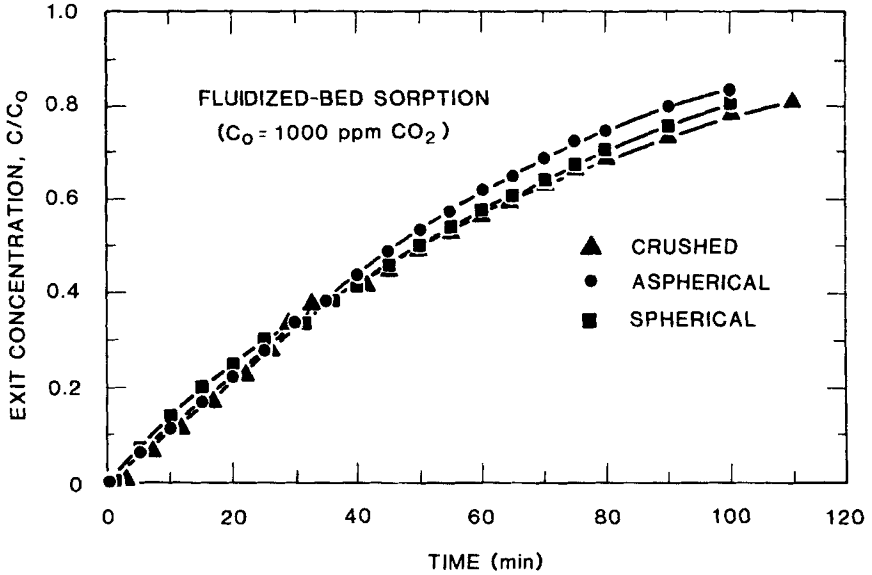

Fig. 9. Effect of particle shape on the sorption performance of a fluidized bed of Davison 4A molecular sieves.

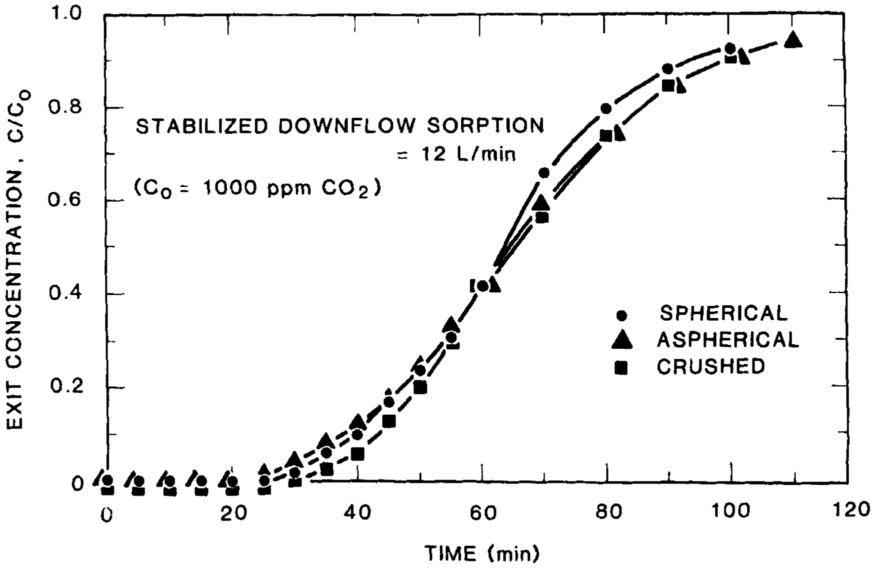

Fig. 10. Effect of particle shape on the sorption performance of a fluidized bed of Davison 4A molecular sieves.

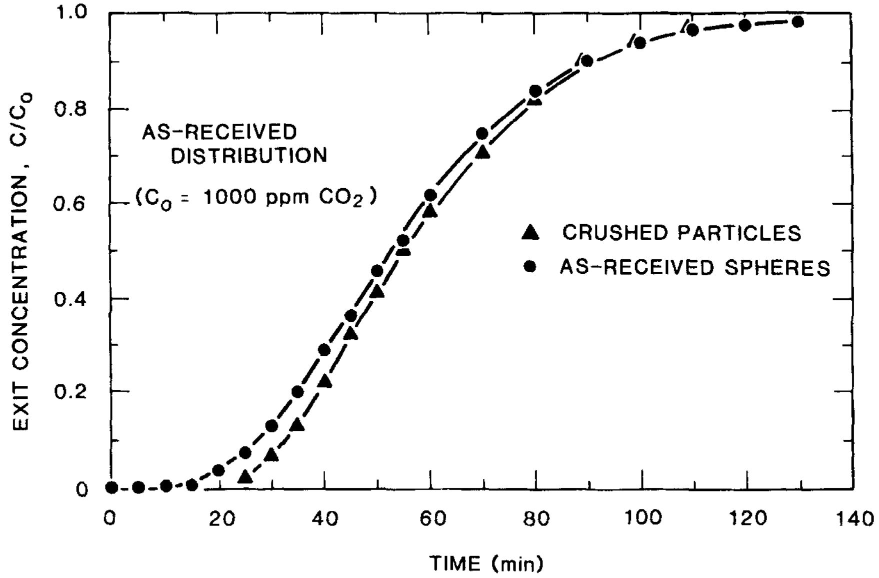

Fig. 11. Sorption performance comparison of two beds with the same size distributions but with different shape characteristics.

diffusion through crystals within the particles. Because of the wide variety of shapes, one would expect to see some differences if macropore diffusion were affecting the process. There appears to be a minor difference in curve shape between the crushed runs and the others, but it is insufficient to ascribe any cause other than run-to-run variation. Given the diffusion coefficients of Yucel and Ruthven (23) for intracrystalline diffusion in Linde 4A molecular sieves, we can show that, for our conditions, micropore diffusion should be controlling. However, considering the differences in manufacturers and the internal variations in their data, such a conclusion could not have been valid without the current experiments.

To assure ourselves that we had micropore control, we prepared a synthetic mixture of crushed particles with the same size distribution as the as-received spherical batch. Comparative experiments were made on these two widely different beds. As shown in Fig. 11, there was no significant difference in the stabilized downflow experiments. The fixed- and fluidized-bed runs yielded the same comparative results. If a factor other than micropore control had any influence on the operation, it would have become evident in these runs. Based on comparison with the theory presented in Fig. 3 and the measured zeolite crystal size of 1.5 μm (16), one would predict a micropore diffusion coefficient of $\sim 2 \times 10^{-8}$ cm^2/s for these sieves. **This value agrees well with** the measurements of Yucel and Ruthven (23).

CONCLUSIONS

It has been shown that an ESEB can be operated by using beds that contain particles with widely varying sizes and shapes. With crushed particles, a higher field is required to "freeze" particle motion. When equal-weight beds were used while varying the shapes of the particles, it was found that, under identical feed concentrations and flow conditions, the sorption curves were virtually the same. Within the accuracy of the data, size distribution had no effect on the sorption performance of the bed. On the basis of the second and third conclusions, it appears that, in this range, the sorption of CO_2 on Davison 4A molecular sieves is micropore controlled.

NOMENCLATURE

c	gas-phase solute concentration, g/L
c_o	initial gas-phase solute concentration, g/L
d_p	average bed particle diameter, cm
D_c	intracrystalline diffusion coefficient, cm^2/s
D_L	dispersion coefficient, cm^2/s
D_p	pore diffusion coefficient, cm^2/s
k'	effective mass transfer coefficient [Eq. (6)], cm/s
k_f	fluid-phase mass transfer coefficient, cm/s
K	equilibrium distribution coefficient
n	exponent in concentration in the Freundlich equation
Pe	Peclet number (d_pv/D)
q	local solid-phase solute concentration, g/g solid
$\bar{q}$	solid-phase average solute concentration, g/g solid
r_c	average crystal radius, cm

R	radial position in a pellet, cm
R_p	radius of a pellet, cm
Re	Reynolds number ($d_p u/v$)
t	time, s
v	interstitial velocity, cm/s
z	bed axial position, cm

Greek

ε	bed voidage fraction
ε_p	pellet voidage fraction

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