The order of magnitude of the dispersed phase mixing rate is ten volumes interacting per volume of dispersed phase per minute at power inputs of the order 10 hp./1,000 gal. The theoretical analysis in Part I leads to the conclusions that these values of the mixing rates can have significant effects on chemical reactions in stirred vessels, and that such rates are not closely equivalent to either of the two limiting conditions, zero or complete dispersed phase mixing. The value of these results is that some estimate of dispersed phase mixing, and its effect on chemical reactions can now be made in reactor designs, a possiblity that has not existed heretofore.

NOTATION

a = interfacial area per unit volume
c = dye concentration in a drop
$\bar{c}$ = mean of $p(c)$
$\bar{C}$ = average concentration in light path
D = impeller diameter
d = drop diameter
g_c = conversion factor
h = length of light path
$I(I_0)$ = light transmission (zero drop absorptivity)
$\bar{I}$ = average light transmission
$\bar{I}_1$ = initial average light transmission before dye added
$\bar{I}_2$ = final average light transmission after dye dispersed
K = power coefficient of an impeller
N = impeller rotational speed
n = number of drops
p = probability density distribution or frequency function
$p(h,\bar{C})$ = joint probability density distribution of path length h and $\bar{C}$
$p(\bar{C}|h)$ = conditional probability density distribution of $\bar{C}$ when h is specified
P/V = power input per unit volume
q = number of light paths
r = number of drops in the beam

Greek Letters

α = light absorptivity of dispersed phase prior to dye injection
β = extinction coefficient of the dye
γ_0, γ_1 = constants
θ = normalized light transmission
μ_2 = central variance of $p(c)$
ν = kinematic viscosity
ρ = liquid density
ϕ = volume fraction of dispersed phase
ω_i = dispersed phase mixing, vol./vol., of dispersed phase/min.
ω_r = residence frequency

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Drying of Air by Fixed Bed Adsorption with Molecular Sieves

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Fixed bed air drying is representative of many operations including ion exchange and the more general operation of adsorption. All these processes are characterized by the breakthrough phenomenon in which the stream leaving the bed remains at a low concentration for a considerable period and then rises to the value of the stream entering the bed. This rise when plotted against time or cumulative material passed through the bed results in an S-shaped curve and relatively defines the mass transfer zone (MTZ). The mass transfer zone has been precisely defined (*14*) as that part of the bed or breakthrough curve in which the water concentration change from C_B to C_E is occurring, (C_B and C_E are arbitrarily chosen as 0.05 C_0 and 0.95 C_0 respectively). A typical breakthrough curve is shown in Figure 1.

Adsorption has been studied extensively, but the manufacture of new adsorbents and recognition of new applications have greatly increased the necessity of finding simple and dependable design procedures. Typical of the new adsorbents are the molecular sieves which are unique in their separating properties. The selectivity of these adsorbents and their high affinity for water make them ideal for removing moisture from air.

One simple design procedure for fixed bed adsorbers is the mass transfer zone (MTZ) method described by Treybal (*14*) in which an adsorption zone of constant length and shape independent of fixed bed height is used. It was the purpose of this work to obtain MTZ data for molecular sieves and to further develop the MTZ design method by investigating the effect of inlet air moisture content on the mass transfer zone.

EXPERIMENTAL

All runs were made with a 0.628 in. I.D. fixed bed drier (adsorber). The ranges for the independent variables investigated were as follows: air stream flow rate, 468 to 1,131 lb. dry air/(hr.)(sq. ft.); bed temperature, 65° to 90°F; inlet air water concentration, 0.00571 to 0.01870 lb. water/lb. dry air; adsorbent particle size, 0.065 to 0.093 in.; fixed bed height, 1.156

to 2.158 ft.; initial water content of molecular sieves, zero; and regeneration time and temperature, 24 hr. at 600°F. with a continuous dry air purge.

No attempt was made to control bed pressure; hence pressure was exerted sufficient to overcome bed friction and line and instrument pressure drops. The average bed pressure drop at the optimum flow rate of 914 lb. dry air/(hr.) (sq. ft.) and the maximum fixed bed height was 65 mm. Hg.

Figure 2 represents a flow sheet of the experimental apparatus. Compressed air entered a purifying filter of silica gel, activated carbon, and glass wool, and then passed through two water saturators. These saturators, 28 in. high, were half full of water and loosely packed throughout with stainless-steel wool to cause bubble dispersal within and de-entrainment above the liquid. The degree of saturation was controlled by varying the pressure in the saturators. The air then passed through a final entrainment separator to a needle valve which was used to control the flow rate.

The moist air passed through a heating coil before entering the top of the vertical fixed bed drier. The fixed bed was made of ½-in. brass pipe 12, 18, or 24 in. in height as required for the run. The adsorbent was supported on a wire screen at the bottom of the pipe. Thermocouple and pressure taps were provided above and below the bed. Constant temperature conditions were maintained by submerging the saturators, heating coil, and fixed bed in a large constant temperature bath.

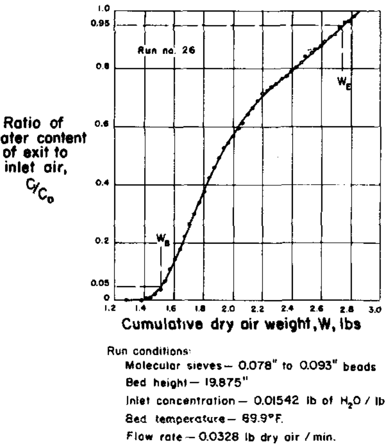


Fig. 1. Representative breakthrough curve for air-water-molecular sieves (type 4A) system.

The water content of the inlet and exit air streams was measured with a dew-point potentiometer. Its construction and operation has been described by Stack (*13*), Frank (*5*), and Eagleton (*3*). Eagleton (*3*) gives details on the accuracy of results obtained with this instrument. Wet and dry bulb thermometers were installed in the dew-point instrument exit air line to give a continuous check on the dew-point readings.

At the beginning of a test run the moist air bypassed the drier until the flow rate and humidity were constant for 10 min. The air was then passed downward through the fixed bed, and at frequent intervals the effluent dew-point, bed exit air temperature, and the wet and dry bulb temperature of the air leaving the dew-point instrument were recorded.

Additional data recorded during a run were saturator pressure, air stream flow rate, bed pressure drop, flowmeter or room temperature, bath temperature, and barometric pressure. When the effluent dew point reached and remained at the inlet value for a short period, the breakthrough part of the run was completed. The run was continued for an hour to insure that equilibrium conditions were reached.

The molecular sieve adsorbent type 4A was screened to obtain the —8+9 and —9+10 Tyler mesh fractions. Runs through no. 31 were made with the —8+9 fraction, nos. 32 to 35 with mixtures of equal weights of the above two fractions, and runs above no. 35 with the —9+10 fraction. The adsorbent was regenerated and weighed immediately before use.

DESIGN EQUATIONS

Much of the previous work with adsorption has dealt with breakthrough curves. The point of breakthrough depends upon three factors: initial conditions, the equilibrium curve, and adsorption rate. The shape of the breakthrough curve is a characteristic of the adsorbent and sometimes depends on the height of the fixed bed.

A given bed of adsorbent has a fixed capacity for the adsorbate (water) which is a function of the concentration in the inlet air stream. The capacity of the adsorbent at other adsorbate concentrations determines the shape of the equilibrium curve (isotherm).

Most methods proposed for interpreting fixed bed data have been too cumbersome for design purposes. A recent simplified solution results from elimination of the variable time. This was accomplished by assuming that the zone in which almost all the adsorption is taking place remains constant in length ($W_E - W_B$) and is independent of fixed bed height.

deVault (*15*) first showed that development of the MTZ as it moves down the bed depends on the shape of the isotherm involved. This was discussed and verified by Glueckauf (*6*) and Barrow et al. (*2*). If gas and solid equilibrium is assumed at all points, and if the equilibrium isotherm is concave to the gas concentration axis, Glueckauf (*6*) showed that there existed a self-sharpening tendency; that is the MTZ length tended to become shorter and shorter as it moved down the bed. Conversely for an isotherm convex to the same axis he showed that the MTZ length tended to become longer and longer as the MTZ moves down the bed; that is a self-diffusing tendency existed.

The effect of nonequilibrium conditions, caused by diffusional resistances, was found to also increase the MTZ length as it moved down the bed. The assumption developed from these principles was that with a self-sharpening type of isotherm the tendency of the isotherm to shorten the MTZ length is counterbalanced by the tendencies of the diffusional resistances to increase the MTZ length. This steady state condition is assumed to be reached a short distance from the entrance to the bed and to continue unchanged, making the MTZ length constant from that point on.

Treybal (*14*), using the ion exchange development of Michaels (*10*) and the fixed bed adsorption data of Eagleton (*3*), developed a simplified MTZ approach to fixed bed adsorber design. The MTZ height equations developed for a given fixed bed condition are

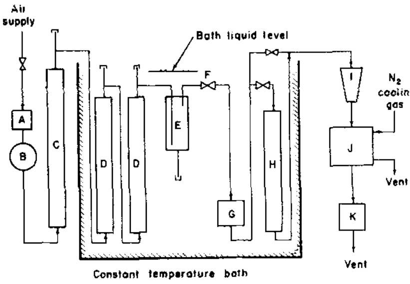


Fig. 2. Flow sheet of experimental apparatus. A—standard air filter, B—pressure regulator, C—packed bed air filter, D—water saturator, E—entrainment separator, F—needle valve, G—heating coil, H—fixed bed drier, I—variable area flowmeter, J—dew-point potentiometer, K—wet and dry bulb psychrometric unit.

$$Z_{MTZ} = Z\left[\frac{t_E - t_B}{t_E - (1-f)(t_E - t_B)}\right] \tag{1}$$

or

$$Z_{MTZ} = Z\left[\frac{W_E - W_B}{W_E - (1-f)(W_E - W_B)}\right] \tag{2}$$

When one uses material balances around the MTZ and the entire fixed bed, the percent of equilibrium saturation at the breakthrough point for the entire fixed bed is obtained and is given by

$$\text{Average \% saturation in bed} = 100\% \left[\frac{Z - (f)(Z_{MTZ})}{Z}\right] \tag{3}$$

The limitations listed by Michaels (*10*) on the use of this method are that the adsorption be from dilute feed mixtures, the isotherm be self-sharpening, the MTZ lengths be constant and independent of bed height, and the height of the adsorbent bed be large relative to the height of the MTZ. Implied in the above are the following assumptions: constant inlet air moisture content; constant inlet air flow rate; initially zero water content for the adsorbent; isothermal fixed bed operation; no concentration, pressure, or temperature gradients across the fixed bed; and no longitudinal diffusion in the air stream.

The mechanism for water adsorption on molecular sieves is important in developing a method for checking the applicability of the above assumptions and for determining the cumulative dry air weight values of W_E and W_B. A combined external and internal diffusion model developed by Eagleton and Bliss (*4*) interprets fixed bed data in terms of mass transfer coefficients by using a gas film and solid shell resistance concept. The linear not through the origin equilibrium relationship used in their work closely approximates the isotherm for molecular sieves as Figure 3 shows.

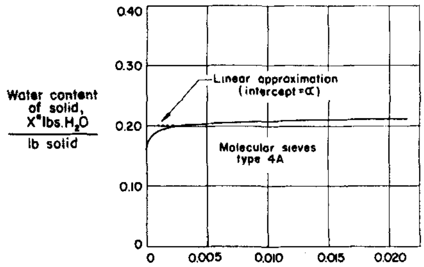


Fig. 3. Equilibrium isotherm for water adsorption on type 4A molecular sieves at 90°F.

The equations developed by Eagleton and Bliss (*4*) for both gas film and solid shell resistances contributing are for $C < C_D$

$$\ln \frac{C_D/C_o}{C/C_o} = -\left[\frac{C_o k_g a}{X_o^* G}\right] W + \frac{k_g a W_x}{G} + 2 - \frac{C}{C_D} \quad (4)$$

for $C > C_D$

$$\ln\left(\frac{1-C_D/C_o}{1-C/C_o}\right) = -\left[\frac{C_D/C_o}{1-C_D/C_o}\right] \left[\left(\frac{C_o k_g a}{X_o^* G}\right) W - \frac{k_g a W_x}{G} - 2 + \frac{C_o}{C_D}\right] \quad (5)$$

For the case of solid shell resistance controlling the equation is

$$-\ln\left(1-\frac{C}{C_o}\right) = \left[\frac{\alpha K_s a}{X^* G}\right] W - \frac{\alpha K_s a W_x}{C_o G} + 1 \quad (6)$$

The linear equilibrium approximation for molecular sieves at 90°F. is given by (*9*)

$$X^* = \alpha + \left(\frac{X_o^* - \alpha}{C_o}\right) C^* \quad (7)$$

The breakthrough curves calculated with these combined external and internal diffusion models are shown in Figure 4. The applicable mass transfer coefficients are given in the next section.

A modification of Acrivos and Vermeulen's empirical pore diffusion model (*1*) was also found to be applicable and is shown in Figure 4. The equation used is

$$\frac{C}{C_o} = 4.63 \times 10^3 \left[\left(\frac{k_s a\, \rho_{air}\, C_o}{G\, X_o^* \rho_b}\right) W - \frac{k_s a\, \rho_{air}\, v}{G} + 1.15 \times 10^{-4}\right] - 5.45 \times 10^6 \left[\left(\frac{k_s a\, \rho_{air}\, C_o}{G\, X_o^*\, \rho_b}\right) W - \frac{k_s a\, \rho_{air}\, v}{G} + 1.15 \times 10^{-4}\right]^2 \quad (8)$$

The combined external and internal diffusion models of Eagelton and Bliss (*4*) reflect the nonconstant character of the separation factor (r) for molecular sieves. Acrivos and Vermeulen's (*1*) pore diffusion model was developed for an irreversible equilibrium ($r = 0$). The molecular sieves separation factor (r) for the 90°F. equilibrium isotherm shown in Figure 3 varies from 0.12 to zero, when calculated from the following equation (*16*):

$$r = \frac{C^*/C_o \left(\dfrac{X_o^*}{X^*} - 1\right)}{(1 - C^*/C_o)} \quad (9)$$

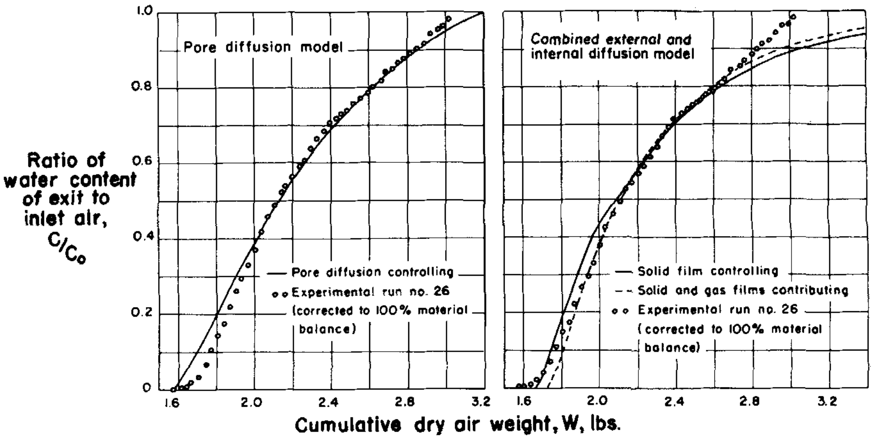
Most of the mechanism models, such as Michaels (*10*) for gas film resistance or Glueckauf and Coates (*7*) for solid shell resistance, were developed for a constant separation factor and do not rigorously apply to this molecular sieve adsorption.

If one knows or can calculate the value of Z_{MTZ}, the height of the mass transfer zone, for a given set of operating conditions and f, the fractional ability of the adsorbent in the MTZ to still adsorb water, it is possible to predict the average percent saturation of a given adsorbent bed at the breakthrough point by using Equation (3). In design calculations it then becomes possible for example to determine the pounds of air a given bed can dry before moisture will appear in the exit air stream, or if one knows the amount of air to be dried, to calculate the bed height (Z) required.

DESIGN VALUES FOR MOLECULAR SIEVES

In this work MTZ lengths and shapes were obtained for various values of the independent variables. Primary emphasis was placed on the effect of inlet air moisture content on the MTZ. These runs and data are detailed in the research of Nutter (*11*).

Table 1 summarizes the important run conditions and calculated values. MTZ heights were calculated by Equation (2). The shape of the breakthrough curve is characterized by the value of f which was found to be constant at 0.592 ± 0.025 for type 4A molecular sieves. It is calculated by dividing the integrated area under the breakthrough curve between W_B and W_E, by the total area between W_B and W_E, and between C/C_o values from 0 to 1.0.



Run conditions:
Molecular sieves – 0.078" to 0.093" beads
Inlet concentration – 0.01542 lb. of H_2O/lb. air
Flow rate – 0.0328" lb. dry air/min.
Bed height – 19.875"
Bed temperature – 89.9°F

Fig. 4. Adsorption mechanism models for an air-water-molecular sieves (type 4A) system.

Table 1. Principal Run Conditions and Calculated Values

Run	Bed weight, g.	Bed temperature, °F.	Flow rate, lb. dry air/ hr./sq. ft.	Inlet concentration C_o, lb. H_2O/ lb. air	Equilibrium solid concentration X_o^*, lb. H_2O/ lb. adsorbent	MTZ height, Z_{MTZ} (in.)
2	50.4926	67.5	1,096	0.01101	0.2395	11.62
3	50.6472	71.1	1,118	0.01137	0.2167	11.18
4	50.7439	65.8	1,131	0.00988	0.2100	10.72
5	49.3793	67.3	1,131	0.01042	0.2255	12.62
6	71.1840	65.9	1,097	0.00988	0.2250	10.05
7	92.6092	68.8	1,107	0.01065	0.2273	11.29
8	90.6634	65.3	469	0.00988	0.2353	5.11
9	70.8088	66.8	469	0.01065	0.2273	5.24
10	50.3872	66.4	471	0.01119	0.2260	5.83
11	50.5833	80.9	1,139	0.01750	0.2288	11.67
12	70.1531	80.3	914	0.01244	0.2235	7.67
13	71.0289	80.6	913	0.01811	0.2540	11.26
14	49.6284	79.9	912	0.01841	0.2662	9.92
15	49.1948	80.4	912	0.01222	0.2395	8.14
17	91.6680	79.7	915	0.01870	0.2692	11.17
19	91.5228	80.0	914	0.01291	0.2268	9.40
20	91.9565	80.0	913	0.00784	0.2080	8.29
21	70.5793	80.2	913	0.00799	0.2228	7.77
22	49.7957	79.6	900	0.00772	0.2200	8.40
23	49.1825	90.0	913	0.01870	0.2197	9.41
24	48.2662	90.9	913	0.01542	0.2320	9.52
25	70.4699	90.4	914	0.01634	0.2259	11.66
26	70.9017	89.9	915	0.01542	0.2250	10.91
27	90.2971	90.0	914	0.01572	0.2285	10.65
28	49.2386	90.0	914	0.01083	0.2183	9.50
29	91.2397	90.0	912	0.01750	0.2198	10.46
30	89.8467	90.1	912	0.01083	0.2155	9.89
31	69.8996	90.0	909	0.01119	0.2215	8.77
33	70.8209	70.5	915	0.00988	0.2370	8.11
34	70.7236	70.4	915	0.00571	0.2225	7.25
35	69.4490	70.3	915	0.01222	0.2319	8.77
36	70.9255	70.6	913	0.00860	0.2292	7.74
38	70.3577	70.4	915	0.00860	0.2279	7.76

Run no. 26 was selected to demonstrate the type of analysis that can be carried out with these data. The overall solid phase mass transfer coefficient $K_s a$ was found to be 3.695 hr.$^{-1}$, when evaluated from the slope of a plot of $\ln(1 - C/C_o)$ vs. W. From the graphical procedures of Eagleton (*3*) the solid phase mass transfer coefficient $k_s a$ averaged 4.0 hr.$^{-1}$. A gas phase coefficient of 486 lb. air/(hr.) (lb. adsorbent) was calculated from Equations (4) and (5) by using the average $k_s a$ value and an experimental C/C_o value close to zero. Breakthrough curves were then calculated from the above mass transfer coefficients as constants. These are shown in Figure 4.

Solid diffusivities can be calculated from the shell resistance mass transfer coefficients by the following equation of Glueckauf and Coates (*8*):

$$k_s a = \frac{60\, D_p}{d_p^2} \tag{10}$$

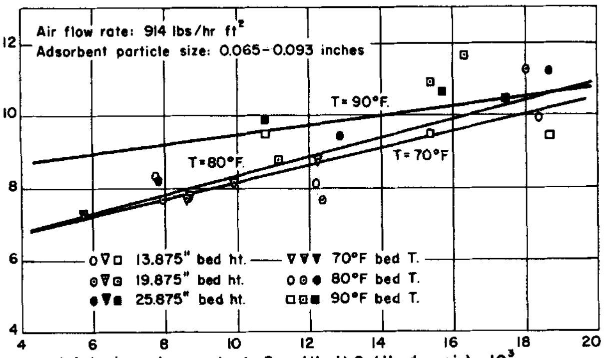


Fig. 5. MTZ height vs. inlet air water content for type 4A molecular sieves.

The calculated particle diffusivity (D_p) is 3.382 × 10^{-6} sq. ft./hr. This can be compared with a Knudsen diffusion diffusivity (D_k) of 3.09 × 10^{-3} sq. ft./hr. for type 4A molecular seives at 90°F. The equation given in Smith (*12*) was used to obtain the Knudsen diffusivity.

The empirical pore diffusion model [Equation (8)] was developed by assuming a pore diffusivity equal to the reaction kinetic particle diffusivity ($D_{pore} = D_p = 3.382 \times 10^{-6}$). The numerical constants were evaluated from the experimental values $C/C_o = 0.70$ and $C/C_o = 0.80$. Equation (8) was then used to calculate the remaining portions of the breakthrough curve. The result is shown for comparison in Figure 4.

Figure 5 shows the MTZ heights vs. inlet air water contents for the bed temperatures used in this research, namely 70°, 80°, and 90°F. The data for each of these curves were statistically evaluated. Almost all of the variation shown is attributed to the inability to reproduce precisely experimental conditions rather than to errors in measurement. For example it was difficult to maintain a given humidity at the high inlet air concentrations.

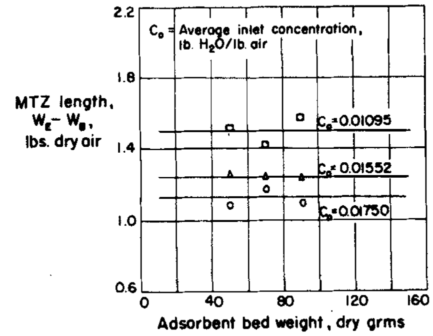


Fig. 6. Effect of bed height (weight) on MTZ length at a flow rate of 914 lb. dry air/hr. sq. ft. and a bed temperature of 90°F.

Expressions relating the height of the mass transfer zone Z_{MTZ} to inlet concentration are developed by combining the mechanism model equations with the Z_{MTZ} equation [Equation (2)].

Using Equation (6) the overall solid film resistance model of Eagleton and Bliss (*4*), for calculating W_B and an empirical expression obtained from run no. 26 data for W_E, one finds

$$Z_{MTZ} = \frac{2.148\, C_o G Z}{0.279\, C_o G + W_r\, \alpha\, K_s a} \tag{11}$$

where

$$W_E = \frac{X_o^*\, W_x}{C_o} + 1.2\,\frac{X_o^*\, G}{\alpha\, K_s a} \tag{12}$$

This represents the relative effect of C_o on Z_{MTZ} as shown in Figure 5. It appears that the slope of Z_{MTZ} vs. C_o line reflects the nonconstant character of the separation factor (r). Similarly one can develop other expressions using the different mechanism models.

Equilibrium data for each run are shown in columns 5 and 6 of Table 1. The equilibrium isotherm for water adsorption of type 4A molecular sieves at 90°F. is given in Figure 3. Complete equilibrium data for molecular sieves are available from the Linde Company (9).

The constant MTZ length independent of fixed bed height is shown in Figures 6 and 7. The stated limitation, requiring that the MTZ length be independent of bed height for the application of the method of Michaels (*10*), closely fits the experimental data. Deviations within each group of different bed heights are random and are attributed to the inability of exactly duplicating run conditions. The limitation stating that dilute feed streams be used was met, as is apparent in Table 1 from the listed inlet air water concentrations. The equilibrium data (Figure 3) for molecular sieves show extreme self-sharpening tendencies.

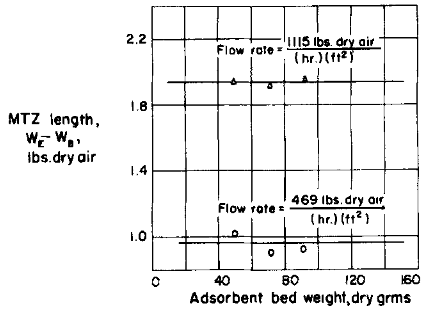


Fig. 7. Effect of bed height (weight) on MTZ length at a bed temperature of 66.5°F. and an average inlet concentration of 0.01045 lb. H_2O/lb. air.

SUMMARY

A constant MTZ length independent of fixed bed height was found for all values of the independent variables studied. The shape of the breakthrough curve and the fraction of the MTZ still able to adsorb water were also found constant. A combined external and internal diffusion or pore diffusion model can be used to interpret molecular sieve breakthrough data and to calculate MTZ lengths ($W_E - W_B$). It is proposed that commercial fixed bed units can be designed with MTZ heights which are calculated or known as functions of the usual design variables such as flow rate, temperature, particle size, and inlet air water concentration. The variations of MTZ heights with inlet air water contents were obtained in this work as one step toward this end.

NOTATION

a = surface area of particles, sq. ft./lb. adsorbent
C = air stream water concentration at time t, lb. H_2O/lb. dry air
C^* = air stream water content in equilibrium with X^*, lb. H_2O/lb. dry air
C_o = inlet air water concentration, lb. H_2O/lb. dry air
C_D = concentration in air at point of discontinuity, lb. H_2O/lb. dry air
d_p = particle diameter, ft.
D_k = Knudsen diffusion diffusivity, sq. ft./hr.
D_p = particle phase diffusivity, sq. ft./hr.
D_{pore} = fluid phase pore diffusivity, sq. ft./hr.
f = fractional ability of adsorbent in MTZ to still adsorb water
G = mass flow rate of air, lb. dry air/hr.
k_g = gas film mass transfer coefficient, $\frac{\text{lb. } H_2O \text{ adsorbed}}{(\text{hr.})(\text{sq. ft.})(C \text{ units})}$
k_s = solid phase mass transfer coefficient, $\frac{\text{lb. } H_2O \text{ adsorbed}}{(\text{hr.})(\text{sq. ft.})(X \text{ units})}$
K_s = overall mass transfer coefficient based on solid phase, $\frac{\text{lb. } H_2O \text{ adsorbed}}{(\text{hr.})(\text{sq.ft.})(X\text{-units})}$
r = equilibrium parameter or separation factor
t_B = time of appearance of breakthrough point, hr.
t_E = time required for MTZ to establish itself and move out of the fixed bed, and the time of appearance of bed saturation point, hr.
t_{MTZ} = time required for MTZ to move its own length down the column, hr. (for example $t_{MTZ} = t_E - t_B$)
v = fixed bed volume, cu. ft.
W = total dry air accumulated up to time t, lb. dry air
W_B = total dry air accumulated up to breakthrough point, lb. dry air
W_E = total dry air accumulated up to bed saturation point, lb. dry air
W_{MTZ} = total dry air accumulated during breakthrough curve period, lb. dry air (for example $W_{MTZ} = W_E - W_B$)
W_x = weight of adsorbent in fixed bed, lb.
X^* = water content of adsorbent in equilibrium with C^*, lb. H_2O/lb. adsorbent
X_o^* = water content of adsorbent in equilibrium with C^*, lb. H_2O/lb. adsorbent
Z = height of fixed bed, ft.
Z_{MTZ} = that part of bed in which the water concentration change from C_B to C_E is occurring, ft. (C_B and C_E are arbitrarily chosen as 0.05 C_o and 0.95 C_o respectively)
α = intercept value of X^* for linear isotherm approximation, lb. H_2O/lb. adsorbent
ρ_{air} = density of air, lb./cu. ft.
ρ_b = bulk packed density of dry adsorbent, lb./cu. ft.

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