# SMALL LOW-COST EQUIPMENT AND FAST EXPERIMENTS AT A UNIT OPERATIONS LABORATORY: ADSORPTION/BREAKTHROUGH CURVE

**Deovaldo de Moraes Júnior** – deovaldo@unisanta.br
**Luiz Renato Bastos Lia** – bastos@unisanta.br
**Flávio Pereira** – scecilia@unisanta.br
**Oswaldo Bonfanti** - scecilia@unisanta.br
Universidade Santa Cecília, Faculdade de Engenharia Química, Rua Oswaldo Cruz, 266, Santos, SP, Brasil, CEP 11045-100
**Edson Luiz Silva** – edsilva@power.ufscar.br
Universidade Federal de São Carlos, DEQ, Rod. Washington Luiz km 235, São Carlos, SP, Brazil

***Resumo.*** *The equipment in this work was designed and built on bench-scale with the participation of undergraduates. Its low cost, small size), and operation with non-toxic compounds (air, water and silica gel) make possible the accomplishment of several studies regarding adsorption in a fixed bed in less than two hours. The unit consists basically of an air source, an air flow meter, a humidifier, a column with silica gel, and thermometers to measure dry-bulb and wet-bulb temperatures. With this equipment several experimental investigations may be carried out: a) the capacity of the packed-bed tower through a breakthrough curve (by measuring the flow and temperature as well as observing the change in color); b) the scale-up by the length of unused bed; c) the influence of the packing on the occurrence of channeling and stagnant volumes; d) the use of the tanks-in-series model; and e) the pressure drop. The experimental variables can be easily assessed, e.g. the silica granulometry (grinding/sieving); the humidity at the inlet; the packing height; and the packing technique.*

***Key Words:*** *Adsortion, Unit operations, Breakthrough curve*

## 1. THEORY

Adsorption is a unit operation consisting basically of the retention of one or more components of a liquid, vapor or gas on the surface of a solid caused by the molecular mass, shape or polarity difference between the former and the solid surface. Several industrial applications attest the importance of this operation, e.g. the removal of impurities, such as color, flavor or smell, from vegetable oils, sugar solutions, pharmaceuticals, tap waters, wastewaters (such as phenol solutions and fermentation effluents). It is also employed in the removal of impurities from the air of ventilation systems; in the recovery of toxic fumes, e.g. ammonium, from fertilizer factories; in the separation of rare gases such as krypton and xenon; in the dehumidification of cooling gases from refrigeration and air conditioning equipment; and in the air dehumidification of the cryogenic process used to obtain oxygen, nitrogen and argon.

It is fundamental, when designing industrial adsorption columns, that the bed adsorption capacity (kg adsorbate, solute per kg adsorbent, solid) be known under the operating conditions. An important technique used to determine this capacity is the breakthrough curve McCabe, Smith and Harriott(1993); Geankoplis(1993); Coulson, Richardson, Backurst and Harker(1996); Perry, Green and Maloney(1997); Schweitzered(1998); Treybal (1980); King(1980); Macávek and Navratil(1992); Lukchis(1980) and Tejeda(1996).

With this technique the undergraduate students can determine experimentally the breakthrough curve of the air dehumidification carried out in a fixed bed with silica gel. The silica saturation capacity to adsorb the water present in the air ($w_{sat}$, adsorbate loading, g/g solid, at equilibrium with the fluid) may be estimated using the classic equation for an ideal breakthrough curve, McCabe, Smith and Harriott(1993).

$$w_{sat} = \frac{QC_0M\theta_m}{AL_T\rho_b} = \frac{u_0C_0M\theta_m}{L_T\rho_b} = \frac{F_A\theta_m}{L_T\rho_b} \quad (1)$$

where:
$Q$ = total feed rate (fluid and adsorbate), $m^3/h$
$C_o$ = concentration at the adsorbate column inlet per volume of mixture, $kgmol/m^3$
$M$ = molecular mass of adsorbate, kg/kgmol
$\theta_m$ = ideal adsorption time, h
$A$ = cross sectional area of bed
$L_T$ = total bed length, m
$\rho_b$ = bulk density of bed, $kg/m^3$
$u_0$ = superficial velocity of fluid, m/h
$F_A$ = feed rate of adsorbate per unit cross-sectional area of bed, $kg/h\ m^2$

In the case of air dehumidification the adsorbate feed rate per unit cross-sectional area ($F_A$) may be estimated through:

$$F_A = \phi_0 \frac{1}{V_H} u_0 \quad (2)$$

where:
$\phi_o$ = specific humidity at the column inlet, determined by humidity charts (psychrometric charts) for mixtures of air and water vapor with dry bulb temperature ($t_{bs}$) and wet bulb temperature ($t_{bu}$) (kg water vapor/kg dry air)
$V_H$ = humid volume, determined by humidity charts with the values of $t_{bs}$ and $t_{bu}$ ($m^3$ humid air /kg dry air)

Considering C the adsorbate concentration at the column outlet, and the graph of $C/C_o$ as a function of time ($\theta$) as the breakthrough curve, the area above the curve $\int_0^{\theta}(1-C/C_0)d\theta$ is the ideal adsorption time, which is proportional to the total volume of absorbed solute when the entire bed comes to equilibrium with the feed. This time may be obtained, approximately, introducing $C/C_o = 0.5$ in the breakthrough curve. The concentration $C_o$, in the case of air dehumidification, is the constant air humidity $\phi_o$ at the bed inlet obtained through humidity

charts with the values of $t_{bs}$ and $t_{bu}$. The concentration C, which increases with time, is the humidity $\phi$ at the bed outlet, also obtained with the values of $t_{bs}$ and $t_{bu}$.

## 2. MATERIALS

Figure 1 shows the built unit diagram.

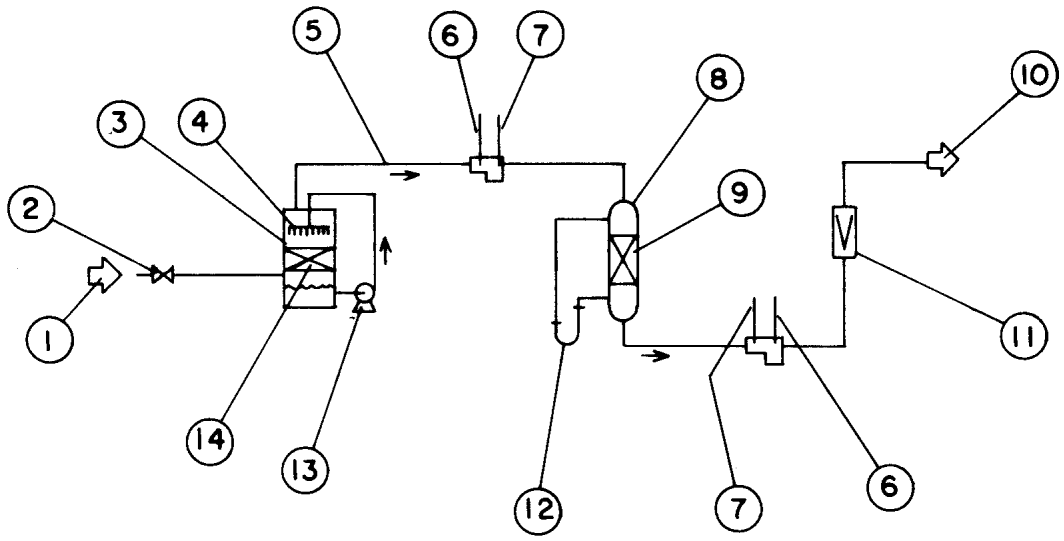


Figure 1 - Equipment for the study of air dehumidification through adsorption in fixed bed. 1) air from compressor; 2) pressure reducing valve utilized in spray guns (low-cost); 3) acrylic humidifier, 80mm diameter and 250mm height; 4) water distributor (shower nozzle); 5) humid air in 3/8 in. polyethylene tubing; 6) dry-bulb thermometer; 7) wet-bulb thermometer; 8) glass packed column, 30mm internal diameter and 200mm height; 9) silica gel packing; 10) air at column outlet with less humidity than that at the column inlet; 11) rotameter, flow rate ranging from 0 to $50ft^3/h$ ($\cong 1.4m^3/h$); 12) U-shaped tubing with mercury; 13) dishwasher pump with 30mm diameter rotor for water recycling; and 14) humidifier packing consisting of 10mm long pieces of 9.53mm diameter polyethylene tubing.

Some important points should be observed when assembling the unit: a) the diameter of the whole tubing system must be equal or wider than 3/8" (9.53mm) in order to prevent high load losses; b) the maximum diameter of the tubes should ensure that the air speed in them is greater than 1m/s in order to yield reliable values of $t_{bs}$ and $t_{bu}$. This procedure is vital to ensure that the rate of the heat flow by radiation from warmer surroundings to the bulb be negligible in comparison to the rate of sensible heat flow by conduction and convention from the air to the bulb, McCabe, Smith and Harriott(1993); c) the wet-bulb wick should be always wet and the reposition water should be at the wet-bulb temperature; d) the rotameter should be installed after the column (as shown in Figure 1) to prevent humid air from the humidifier from interfering with the feed rate measurements.

## 3. EXAMPLE OF RESULT

Figure 2 shows one of the curves that can be obtained in the system. It was plotted using the air feed rate of $35ft^3/h$ ($\cong 0.99m^3/h$); internal diameter of the column equal to 0.03m; superficial

velocity of 1,394.37m/h; 0.17m bed height, average size of commercial silica after grinding equal to 0.7mm (between 16 and 32mesh); dry silica mass (dried at 150ºC) of 0.082kg ($\rho_b$ = 679.37kg/m$^3$); constant dry-bulb and wet-bulb temperatures at the column inlet equal to 23ºC (saturated) ($V_H$ = 0.87m$^3$/kg and $\phi_o$ = 0.0176kg of vapor per kg of dry air, obtained from the humidity chart).

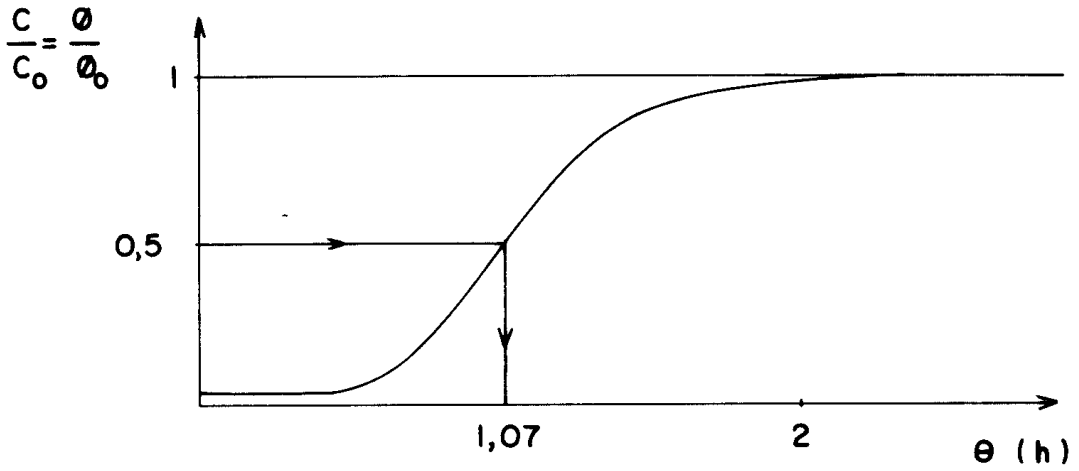


Figure 2 - Example of breakthrough curve. $C/C_o$ or $\phi/\phi_o$ as a function of time.

For the conditions previously mentioned, the area above the curve in Figure 2 yielded an ideal adsorption time for a vertical breakthrough curve equal to 1.07h ($\theta_m$). For the same data, Equation 2 gives $F_A$ = 28.21kg/m$^2$h, and from Equation 1 an approximate concentration in the solid phase ($w_{sat}$) equal to 0.26kg $H_2O$ (adsorbate, solute) per kg dry silica gel (adsorbent, solid) is obtained.

## 4. CONCLUSIONS

Besides the example given in this work for the determination of the column capacity, this equipment - however simple - may be useful in other experimental investigations, such as:

a) The influence of the size distribution, air humidity and superficial velocity on the pressure drop (see correlations) and on the adsorption capacity of the silica (or other commercial adsorbent such as the molecular sieve and unconventional adsorbents such as coffee refuse and peat).
b) The scale-up by the increase in the packing height, using for instance the length of unused bed technique (McCabe, Smith and Harriot, 1993 and Geankoplis, 1993)
c) The reproducibility of the packing by the analysis of the pressure drop constancy in each column assemblage for the same experimental conditions.
d) By analyzing the breakthrough curve it is possible to identify the formation of channeling by the occurrence of peaks and of stagnant volume by the occurrence of non-symmetrical curves. These undesired phenomena (channeling and stagnant volume) may be induced for the sake of instruction by: $d_1$) high velocity and bad distribution of the air at the inlet; $d_2$) column/packing particle diameter ratio smaller than 5; and $d_3$) packing with great size distribution. Vilermaux (1985), shows breakthrough curves with imperfections.
e) The quantification of the packing homogeneity by the breakthrough curve with the tanks-in-series model (Levenspiel, 1999).

## ACKNOWLEDGMENTS

Thanks are due to Gilmar Alcântara and Volnei de Lemos for their diligent collaboration in the assemblage of the experimental unit, and to the undergraduate students Affonso Carlos G. Alvarez, Carlos Roberto de Moraes, Daniela Luz Aguiar and Maria das Graças P. de Albuquerque for helping in the collection of the experimental data and in the improvement of the equipment. We also thank the engineers Antônio Salles Penteado and Áureo Emanuel P. Figueiredo for their financial support.

## REFERENCES

Coulson, J.M., Richardson, J.F., Backhurst, J.R. and Harker, J.H. Chemical Engineering, 4 ed., Butterworth-Heinemann, Oxford, England (1996).

Geankoplis, C.J. Transport Process and Unit Operations, 3 ed., Prentice Hall, Englewood Clifss, NJ (1993).

King, C.J., Separation Processes, 2 ed., McGraw-Hill, New York, NY (1980).

Levenspiel, O. Chemical Reaction Engineering, 3 ed., John Wiley & Sons, New York, NY (1999).

Lukchis, G.M. Adsorption Systems, Part I: Design by Mass-Transfer-Zone Concept. In: Ricci, L. and the staff of chemical engineering eds, Separation Techniques I – Liquid – Liquid Systems, McGraw-Hill, New York, NY (1980) p. 277-282.

Macávek, F. and Navratil, J.D. Separation Chemistry, Ellis Horwood, Czechoslovakia, (1992).

McCabe, W.L., Smith, J.C. and Harriott, P. Unit Operations of Chemical Engineering, 5 ed., McGraw-Hill, New York, NY (1993).

Perry, R.H., Gree, D.W. and Maloney, J.D. eds., Perry's Chemical Engineer's Handbook, 7 ed., McGraw-Hill, New York, NY (1997).

Schweitzer, P.A. ed, Handbook of Separation Techniques for Chemical Engineers, 2 ed., McGraw-Hill, New York, NY (1988).

Tejeda, A. et al. Introduction to Bioseparations Affinity Adsorption, Chemical Engineers Ed., 32(4), 124 (1996).

Treybal, R.E., Mass-Transfer Operations, 3 ed., McGraw-Hill, Singapore (1980).

Villermaux, J. Génie de la Réaction Chimique-Conception et Functionnement des Réacteurs, $2^{eme}$ tirage, Technique et Documentation (Lavossier), Paris, Fr (1985).