REFORMULATING THE LENGTH OF UNUSED BED (LUB) SCALE-UP METHOD FOR IMPROVED APPLICATION AND TEACHING

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INTRODUCTION

Air purification, respirators (i.e., masks), water purification, environmental engineering, drug manufacturing, and other engineering unit operations all use fixed-bed sorption and/or ionic exchangers. Therefore, the teaching of scale-up methods for fixed-bed sorbers is beneficial for a wide range of academic majors. The Length of Unused Bed or LUB methods of scaling-up sorption unit operations are widely used in industry.[1] Discussions initiated by this author on the American Institute of Chemical Engineers (AIChE) Engage Discussion Boards resulted in a number of positive anecdotes on LUB's success in scaling-up commercial applications using lab bench and pilot plant information. In addition, a number of textbooks for teaching separation science contain material on the LUB methods.[1-3]

This author has taught the LUB methods in various courses (general separations, bio-separations, and environmental engineering) and has always found the way textbooks present these methods to be confusing and unnecessarily complicated. In addition, different well-established textbooks present different LUB-methods that produce different results.

For example, all methods define the breakpoint time, t_b, as the time when the exit solute concentration reaches its maximum allowable value for the specified application. However, some textbooks (see Harrison et al.[3] or Ruthven[4]) calculate the breakpoint time, t_b, as the integral of a function from 0 to t_b, where the method treats the t_b in the limit of integration as a known (observed or read from the data) and the t_b on the left hand side of the equal sign as an unknown dependent variable:

$$t_b = \int_0^{t_b} \left(1 - \frac{C_{out}}{C_F}\right) dt \quad (1)$$

where C_{out} is the concentration (kg/m^3) exiting the bed at time t and C_F is the concentration (kg/m^3) in the feed entering the bed. Therefore, t_b is calculated by putting a known t_b into the limits of integration?! Students find this mathematically illogical. A fair hypothesis is that Eq. 1 resulted from a variable notation error, which this manuscript corrects below during the development of the Sorption Capacity method. However, since Eq. 1 appears in a number of textbooks published over a 30+ year period, students can encounter Eq. 1 in this mathematically illogical form.

Other textbooks do not use the lab bench or pilot plant data to determine the length of the used bed for a stated scale-up target time. Instead an equilibrium model estimates the length (Seader et. al.[1])

$$\text{Length of Equilibrium Section} = LES \approx \frac{C_F G_F t_b}{q_F \rho_b} \quad (2)$$

where G_F is the superficial feed velocity (m^3 of fluid/[m^2 s]), q_F is the amount sorbed by the sorbent (kg of solute/kg of sorbent) in equilibrium with the feed, and ρ_b is the bed density (kg/m^3 of bed). Ignoring the lab bench or pilot plant

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data for calculating LES is confusing since the reason for using the LUB method is that dispersion and other unquantifiable properties inside of the packed bed complicate accurate modeling of the sorption process. To quote McCabe, Smith, and Harriott on this point, "…predicting (from models) the concentration profiles and zone widths…may be inaccurate because of uncertainties in the mass-transfer correlations."[2] This author finds it pedagogically confusing to contradict a key student outcome on the value of physical data when scaling-up complex transport processes by using a scale-up method that ignores the transport information contained in the data.

In order to eliminate these pedagogically confusing elements from the successful LUB scale-up methods, this manuscript will first redevelop the equations for teaching LUB, followed by a comparison of scale-up results using this new redeveloped method against the prior LUB methods found in textbooks. The manuscript ends with suggested contents for a teaching module on this subject, including dimensionless numbers, characteristic quantities, and reduction-to-practice caveats.

LUB methods typically appear in textbook sections discussing the operation of fixed-bed adsorption processes. However, the LUB methods are not limited to adsorbents. The methods work with processes that have fixed-bed sorbents that operate as adsorbents, absorbents, or ion exchangers. Therefore, this manuscript will use the more generic terms of sorbents, sorption, and sorbers instead of adsorbents, adsorption, and adsorbers.

LUB METHODS

The key requirement for application of a LUB method is that the concentration versus abscissa co-ordinate shape of the sorption wave front is constant over time or distance traveled (Figure 1). This is the "self-shaping assumption" that results when the process occurs in confined beds (i.e., columns) and the sorption isotherm is "favorable," such as Langmuir, Freundlich, or Irreversible.[2] The velocity at which solutes advance through a bed, V_{Ci} (m/s), is directly related to the solute concentration for favorable isotherms as shown in Eq. 3. This means that as dispersion results in the movement of solutes ahead of the wave front, the lower concentration of these solutes ahead of the wave front will move slower than the solutes left behind and the higher concentrations will catch up with the forward-dispersed solutes.

$$V_{Ci} = \frac{G_F/\varphi}{1+\frac{\rho_S(1-\varphi)}{\varphi}\frac{\Delta q}{\Delta C}} = \frac{G_F/\varphi}{1+\frac{\rho_b}{\varphi}\left(\frac{\Delta q}{\Delta C}\right)} \quad (3)$$

where φ is the bed void fraction (m^3 of fluid/m^3 of bed), ρ_S is the sorbent density (kg/m^3 of sorbent), ΔC is the difference between the feed solute concentration, C_F, and the initial solute concentration in the bed (kg/m^3), C_o, and Δq is the increase in solute content on/in the sorbent after equilibrating with the C_F.[3]

To continue this discussion of LUB Scale-Up methods, it is necessary to define terms. Figure 2 assists in defining terms in reference to a self-sharpening concentration wave front versus bed length and the exit concentration of the solute concentration versus time. L is the length of the bed (m); t_M is the time (s) when the exit concentration is equivalent to the feed solute concentration. The ideal sorption time, t* (s), is

$$t^* \equiv \frac{L}{V_{CF}} \quad (4)$$

where V_{CF} is the velocity (m/s) at which solutes with the feed concentration advance through the bed (see Eq. 3). The design requirements (i.e., maximum allowable exit solute concentration) of the scaled-up unit operation defines the breakpoint time, t_b. In Figure 2, t_b is observed on the time axis when the concentration wave front crosses the maximum allowable exit solute concentration.

The development of scale-up equations starts with a mass balance on the solute entering and exiting the bed during a time interval, Δt:

$$Accumulated = "Sorbed" = \text{Mass in} - \text{Mass out} \quad (5)$$

$$"Sorbed" = AG_F C_F(\Delta t) - AG_F C_{out}(\Delta t) \quad (6)$$

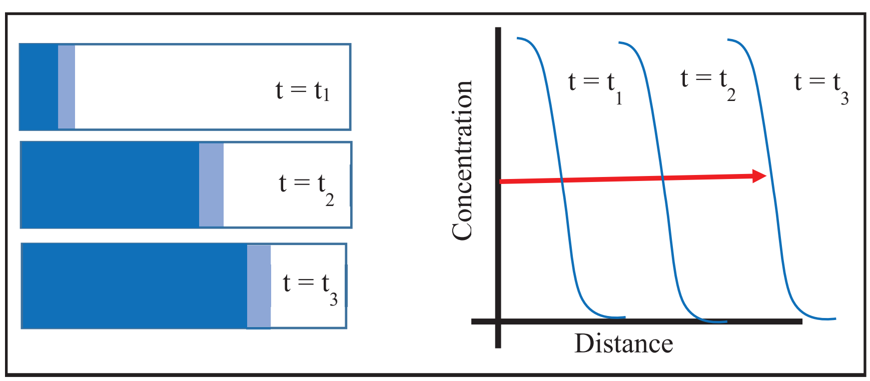

Figure 1. *Movement of a solute wave front in a fixed bed sorber with a favorable sorption isotherm. Left side is a sketch of the movement of the solute (shaded areas) over time in the bed; right side is a graph of solute concentration vs time and distance.*

where A is the cross-sectional area of the bed (m^2). Rearranging Eq. 6 gives

$$"Sorbed" = AG_F C_F \left[1 - \frac{C_{out}}{C_F}\right](\Delta t) \quad (7)$$

Taking the limit as Δt → 0 and starting the limits of integration at time = 0 results in

$$W_t = AG_F C_F \int_0^t \left[1 - \frac{C_{out}}{C_F}\right] dt \quad (8)$$

where W_t is the total amount of solute "sorbed" (kg) from time = 0 to time = t. Conventionally, the accumulation term in this mass balance is called "sorbed;" however, it is actually accumulation on/in the solid sorbent plus the accumulation in the void space between the sorbent particles. Most literature ignores the latter void space accumulation via a usually unstated assumption that it is negligible compared to the sorbed amount on/in the sorbent.

Next we will apply Eq. 8 to two types of wave fronts exiting identical beds (see Figure 3). The ideal wave front results when the following assumptions apply:

- Feed superficial velocity, G_F, equals the exit superficial velocity
- Instantaneous sorption
- Negligible mass transfer resistance for fluid to solid transport
- No dispersion (i.e., no channeling or diffusion)

The ideal wave front is a step function (see left side of Figure 3); however, dispersion, mass transfer resistance, and self-sharpening produce an "S-shape" wave front (see right side of Figure 3).

So two sorption beds, with the only difference being that one suffers from dispersion, etc. while the other one is ideal, will have the same maximum possible sorption loading. Therefore, applying Eq. 8 to both beds in Figure 3 with the upper limit of integration being t_M results in the maximum possible sorption loading, W_{Max} (kg):

$$W_{Max} = AG_F C_F \int_0^{t_M} \left[1 - \frac{C_{out}}{C_F}\right] dt \quad (9)$$

Eq. 9 applies to both beds; however, the step function of the ideal wave form simplifies the integral for the bed on the left resulting in

$$W_{Max} = AG_F C_F t^* \quad (10)$$

Noting that W_{Max} for the left (Eq. 10) and right (Eq. 9) hand side beds in Figure 3 are equal, we get Eq. 11:

$$W_{Max} = AG_F C_F t^* = AG_F C_F \int_0^{t_M} \left[1 - \frac{C_{out}}{C_F}\right] dt \quad (11)$$

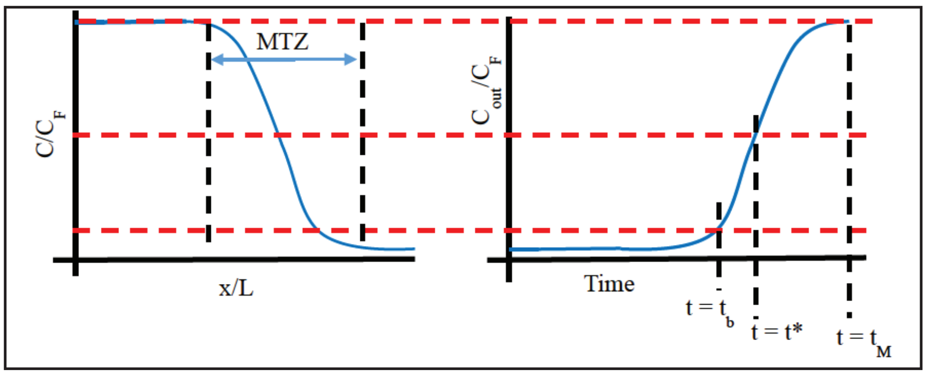

Figure 2. *Self-sharpening concentration wave front vs bed length (left) and the effluent concentration of the solute concentration vs time (right). The Mass Transfer Zone, MTZ, has a constant width but moves at V_{CF}, Eq. 3. Eq. 4 defines t*, and t_M is when $C_{out} = C_F$.*

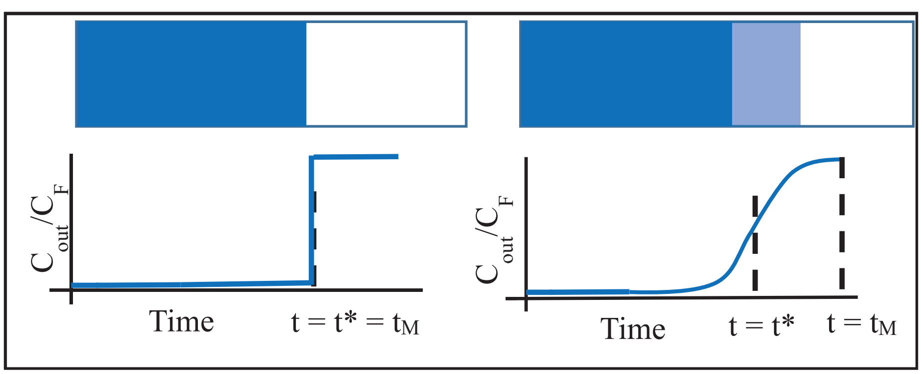

Figure 3. *Effluent data for two types of solute concentration waves. On the left, the step function of an ideal wave front; on the right, the self-sharpening "S-wave" resulting from the interplay of dispersion, mass transfer resistance, and favorable sorption isotherms. Note that for the ideal wave front $t^* = t_M$.*

Canceling the pre-integral term "AG_FC_F" and using the t^* definition (Eq. 4) gives

$$\frac{L}{V_{CF}} = t^* = \int_0^{t_M} \left[1 - \frac{C_{out}}{C_F}\right] dt \qquad (12)$$

Now we apply the favorable isotherm self-sharpening assumption that results in the concentration versus abscissa coordinate shape of the sorption wave front being constant over time or distance traveled. This assumption means that the time difference between t_b and t^* is constant for any time or distance of wave front travel in the bed

$$t^* - t_b = constant \qquad (13)$$

If the superficial velocity, G_F, sorbent material, sorbent particle size, temperature, void space, and solute feed concentration, C_F, are kept constant during a scale-up process, then V_{CF} is constant during scale-up (see Eq. 3). In addition, these scale-up conditions result in Eq. 13 having the same constant for the initial and scaled-up sorption beds. We can now complete the development of the scale-up equations using the following subscript notations: "data" for values obtained from a laboratory, bench, or pilot test; "scale" for the scale-up bed values:

$$(t^* - t_b)_{data} = (t^* - t_b)_{scale} = constant \qquad (14)$$

$$t^*_{data} - t_{b,data} = t^*_{scale} - t_{b,scale} \qquad (15)$$

$$t^*_{scale} = \frac{L_{scale}}{V_{CF}} = \frac{L_{data}}{V_{CF}}\left(\frac{L_{scale}}{L_{data}}\right) = t^*_{data}\left(\frac{L_{scale}}{L_{data}}\right) \qquad (16)$$

Substituting Eq. 16 into Eq. 15 gives

$$t^*_{data} - t_{b,data} = t^*_{data}\left(\frac{L_{scale}}{L_{data}}\right) - t_{b,scale} \qquad (17)$$

If the objective is to estimate the required bed length for a specified t_b, then solving Eq. 17 for L_{scale} gives

$$L_{scale} = L_{data}\left(1 + \frac{(t_{b,scale} - t_{b,data})}{t^*_{data}}\right) \qquad (18)$$

If the objective is to estimate the t_b for a new bed length, L_{scale}, then solving Eq. 17 for $t_{b,scale}$ gives

$$t_{b,scale} = t^*_{data}\left(\frac{L_{scale}}{L_{data}} - 1\right) + t_{b,data} \qquad (19)$$

COMPARISON OF LUB METHODS

For organizational purposes, we will call Eq. 18-19 the proposed "Constant Time Interval" or "Constant-Δt" method. Tables 1 and 2 summarize the scale-up equations for the Constant-Δt method along with equations for two other methods commonly found in textbooks. The Equilibrium Length method starts with Eq. 2 combined with Eq. 13 (see Seader et al.[1] for its development). The Sorption Capacity method starts with a ratio of two Eq. 8's, one applied at $t = t_b$ and one at $t = t_M$.[2,3] In the application of the Sorption Capacity method, the pre-integral term "AG_FC_F" cancels in the ratio of $W_{b,data}/W_{Max,data}$, resulting, according to some references, in Eq. 1 divided by t^*_{data}. More appropriately, the following equations state these steps in the development of the Sorption Capacity method:

$$LUB_{cap} \equiv L_{data}\left(1 - \frac{W_{b,data}}{W_{Max,data}}\right) = L_{data}\left(1 - \frac{AG_FC_Fb_{data}}{AG_FC_Ft^*_{data}}\right) = L_{data}\left(1 - \frac{b_{data}}{t^*_{data}}\right) = L_{scale}\left(1 - \frac{b_{scale}}{t^*_{scale}}\right) \qquad (20)$$

$$b_{data} = \int_0^{t_{b,data}} \left(1 - \frac{C_t}{C_F}\right) dt \qquad (21)$$

where b_{data} (s) and b_{scale} (s) are the stoichiometric breakpoint times.[3,4]

All three methods have the same key assumptions; namely, LUB is constant if the scaled-up bed has the same sorbent, particle size, void space, temperature, superficial velocity, and solute feed concentration as the data bed. In addition, all three methods assume favorable sorption isotherms. The Equilibrium Length method, further, assumes that the bed's initial solute concentration is zero and that the solute in the fluid phase of the saturated bed is negligible compared to the amount of the solute on/in the solid sorbent. An additional assumption is made during the development of the Sorption Capacity equations in Tables 1 and 2; namely, that $t_{b,scale} \approx b_{scale}$. We will show later that this Sorption Capacity assumption limits its application to systems where $t_{b,scale} < t^*_{scale}$.

The t^*_{data} equation is identical in all three methods. The development above, resulting in Eq. 12, shows that the t^*_{data} (in Tables 1 and 2) is also equal to Eq. 4. This means that $L_{data}/t^*_{data} = V_{CF}$ for all three methods. In contrast to t^*_{data}, each method has a different definition for LUB, which are not identical. One definition is the distance the wave front travels during the time interval starting at t_b and ending at t^*_{data}; specifically, $LUB_{eq} = V_{CF}(t^* - t_b)$. The other definition is the amount of the bed "not used" or $(W_{Max} - W_b)$ converted to units of length. Figure 4A shows the graphical trends of

TABLE 1 **Equations for estimating the scaled-up bed length, L_{scale}, given a specified $t_{b,scale}$.**		
Proposed "Constant-Δt"	**Equilibrium Length**	**Sorption Capacity**
$L_{scale} = L_{data}\left(1 + \frac{(t_{b,scale} - t_{b,data})}{t^*_{data}}\right)$	$L_{scale} = LUB_{eq} + \text{LES}$	$L_{scale} = LUB_{cap} + L_{data}\frac{t_{b,scale}}{t^*_{data}}$
	$LUB_{eq} = \frac{L_{data}}{t^*_{data}}(t^*_{data} - t_{b,data})$	$LUB_{cap} = L_{data}\left(1 - \frac{W_{b,data}}{W_{Max,data}}\right)$
	Length of Equilibrium Section = $LES = \frac{C_F G_F t_{b,scale}}{q_F \rho_B}$	$W_{Max,data} = \text{A}G_F C_F \int_0^{t_M}\left[1 - \frac{C_{out}}{C_F}\right]dt$
$t_{b,data} = Observation$	$t_{b,data} = Observation$	$W_{b,data} = \text{A}G_F C_F \int_0^{t_b}\left[1 - \frac{C_{out}}{C_F}\right]dt$
$t^*_{data} = \int_0^{t_M}\left[1 - \frac{C_{out}}{C_F}\right]dt$	$t^*_{data} = \int_0^{t_M}\left[1 - \frac{C_{out}}{C_F}\right]dt$	$t^*_{data} = \int_0^{t_M}\left[1 - \frac{C_{out}}{C_F}\right]dt$

TABLE 2 **Equations for estimating the breakpoint time, $t_{b,scale}$, given a bed length, L_{scale}.**		
Proposed "Constant-Δt"	**Equilibrium Length**	**Sorption Capacity**
$t_{b,scale} = t^*_{data}\left(\frac{L_{scale}}{L_{data}} - 1\right) + t_{b,data}$	$t_{b,scale} = IST\left(1 - \frac{LUB_{eq}}{L_{scale}}\right)$	$t_{b,scale} = \frac{t^*_{data}}{L_{data}}(L_{scale} - LUB_{cap})$
	$LUB_{eq} = \frac{L_{data}}{t^*_{data}}(t^*_{data} - t_{b,data})$	$LUB_{cap} = L_{data}\left(1 - \frac{W_{b,data}}{W_{Max,data}}\right)$
	Ideal Sorption Time = $IST = \frac{L_{scale} q_F \rho_B}{C_F G_F}$	$W_{Max,data} = \text{A}G_F C_F \int_0^{t_M}\left[1 - \frac{C_{out}}{C_F}\right]dt$
$t_{b,data} = Observation$	$t_{b,data} = Observation$	$W_{b,data} = \text{A}G_F C_F \int_0^{t_b}\left[1 - \frac{C_{out}}{C_F}\right]dt$
$t^*_{data} = \int_0^{t_M}\left[1 - \frac{C_{out}}{C_F}\right]dt$	$t^*_{data} = \int_0^{t_M}\left[1 - \frac{C_{out}}{C_F}\right]dt$	$t^*_{data} = \int_0^{t_M}\left[1 - \frac{C_{out}}{C_F}\right]dt$

these two LUBs. The LUB for the Sorption Capacity method tends to zero when the t_b definition is greater than t^*. In contrast, the LUB of the Equilibrium Length method, LUB_{eq}, is negative at higher values of t_b. Figure 4B shows the relationship between b_{data} from Eq. 21 and t_b plotted in reduced (dimensionless) variables.

In comparing the Constant-Δt and the Equilibrium Length methods, we note that they both have equivalent definitions of LUB. Eq. 14 is the Constant-Δt LUB definition:

$$(t^* - t_b)_{data} = (t^* - t_b)_{scale} = Constant\ A \quad (14)$$

The Equilibrium Length LUB_{eq} definition is

$$V_{CF}(t^* - t_b)_{data} = V_{CF}(t^* - t_b)_{scale} = Constant\ B \quad (22)$$

Because V_{CF} is constant during scale-up, Eq. 14 and Eq. 22 are equivalent, with *Constant B* = (V_{CF})*Constant A*.

The Equilibrium Length is the only method of the three in Tables 1 and 2 that uses equilibrium isotherm data via the variables, $q_F\rho_B$. The use of equilibrium isotherm data is a significant difference and sets the Equilibrium Length method apart from the other two methods. If the isotherm data are unavailable, then some authors estimate the $q_F\rho_B$ variables via mass balance for the ideal wave front at time = t^* where $C_{out} = C_o$ for all times:[3,5]

$$A(q_F - q_o)\rho_b L + A(C_F - C_o)\varphi L = AG_F C_F(t^*) - AG_F C_o(t^*) \quad (23)$$

where C_o is the initial concentration of the solute in the bed and q_o is the initial solute loading (kg/kg) on/in the sorbent. Defining $\Delta q = (q_F - q_o)$ and $\Delta C = (C_F - C_o)$, then Eq. 23 gives

$$Bed\ Loading\ at\ Saturation = (\Delta q)\rho_b + \Delta C\varphi = \frac{t^*}{L} G_F \Delta C \quad (24)$$

The definitions of LES and IST in Tables 1 and 2 assumed that the bed's initial solute concentration is zero (i.e., $C_o = 0$ and $q_o = 0$) and that the solute in the fluid phase of the saturated bed is negligible ($\Delta C\varphi = 0$) compared to the amount of the solute on/in the solid sorbent. The following equations result from removing these two simplifying assumptions:

$$LES = \frac{G_F \Delta C}{(\Delta q)\rho_b + \Delta C\varphi}\, t_{b,scale} \quad (25)$$

$$IST = \frac{(\Delta q)\rho_b + \Delta C\varphi}{G_F \Delta C}\, L_{scale} \quad (26)$$

Now if we insert Eq. 24 into Eq. 25 and Eq. 26, the results are

$$LES = L_{data}\frac{t_{b,scale}}{t^*_{data}} \quad (27)$$

$$IST = t^*_{data}\frac{L_{scale}}{L_{data}} \quad (28)$$

Using Eq. 27 for LES and Eq. 28 for IST results in the Constant-Δt and Equilibrium Length methods being algebraically equivalent. The proof of this algebraic equivalency is left up to the reader and illustrated in the predicted scale-up performance curves presented below.

Before leaving the comparison on how to use the LUB methods, we should discuss method application in special situations. One special situation is the need to scale up with incomplete effluent trace data. In this situation one assumes symmetry to complete the wave front curve up to C/C_F = 1.0.[2,3] This is what is done below for Case Studies B and D. Another special situation is that the wave front data stop after $t_{b,data}$. This special situation can occur with commercial units where the test needs to terminate at $t_{b,data}$.[5] There are two options for applying the Equilibrium Length method when the wave front data terminates at $t_{b,data}$:

I. Calculate $LUB_{eq} = L_{data} - V_{CF}(t_{b,data})$, where V_{CF} is Eq. 3
II. Calculate $LUB_{eq} = L_{data} - LES_{data}$

For the Constant-Δt method, when only $t_{b,data}$ is available, the only missing information is t^*_{data}, which Eq. 4 will estimate using Eq. 3 to calculate V_{CF}. Similarly for the Sorption Capacity method, Eq. 3-4 can estimate the missing t^*_{data} followed by estimating $W_{Max,data}$ from $W_{Max,data} = AG_F C_F t^*_{data}$.

COMPARISON OF CASE STUDY RESULTS

To compare LUB methods with actual numbers, we selected case studies previously used in various textbooks. Table 3 summarizes the systems of these case studies along with citations and the LUB method used by the cited textbook. The first two case studies in Table 3 have experimental information on both the data and as-built (scaled-up) columns, allowing comparison of the scaled-up results with the performance of actual beds. Case Study A-Collins forms the basis of the example problems in both Seader et al.[1] and the classic textbook by Treybal.[6] The original source material contains the adsorption isotherms, allowing comparison of all three LUB methods. The remaining case studies do not have sorption isotherms, forcing the Equilibrium Length method to use Eq. 24 resulting in the Equilibrium Length and Constant-Δt methods having identical performance curves in the presented figures.

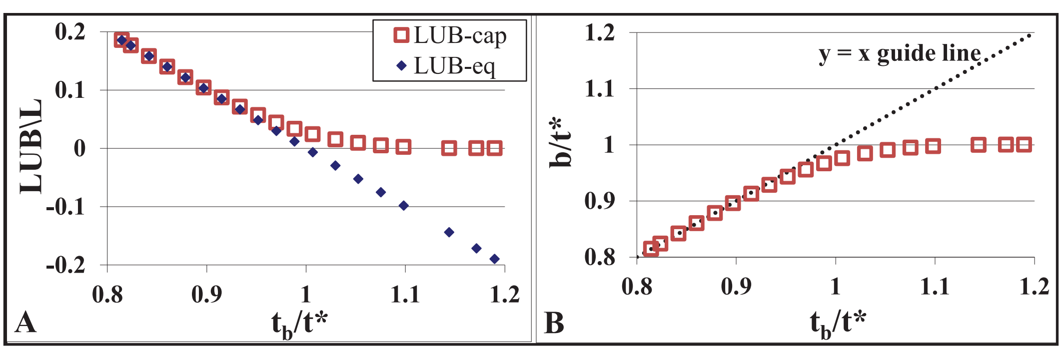

Figure 4. *Left hand graph (A) illustrates the difference in the definitions of the Length of Unused Bed for the Equilibrium Length (LUB-eq) vs the Sorption Capacity (LUB-cap) methods. The right hand side (B), illustrates the range over which the assumption $t_b \approx b$ is valid. Data for these illustrations taken from Case Study A-Collins in Table 3.*

TABLE 3 Summary of the Case Studies used to compare various LUB scale-up methods. Case Study C-Cephal data appear below in the example homework problem.				
Case Study	**System**	**Primary Reference**	**Textbook Reference**	**Method Textbook Used**
A-Collins	Drying of nitrogen gas with molecular sieve	Collins[5]	Treybal[6] and Seader et al.[1]	Equilibrium Length
B-MSH	Adsorption of n-butanol from air		McCabe, Smith, and Harriott[2]	Sorption Capacity
C-Cephal	Antibiotic recovery from fermentation broth	Belter et al.[7]	Harrison et al.[3]	Sorption Capacity
D-Pharma	Pharmaceutical capture		Harrison et al.[3]	Sorption Cap./Eq. Length

Case Study B-MSH is the example problem from McCabe, Smith, and Harriott.[2] Case Study B-MSH required assuming symmetry of the wave front around $C/C_F = 0.5$, since the wave front information for the data and as-built columns is limited to $C/C_F < 0.6$. The final two case studies are sorption of a product from liquid carrier fluids.

Figure 5 uses Case Study A to illustrate the trends of the three LUB methods. The Constant-Δt and Equilibrium Length methods have similar trends but with an offset. The offset is the difference between using equilibrium isotherms via the IST equation in Table 2 versus the test bed saturation data (i.e., Eq. 28). Conventional wisdom predicts that Eq. 28 (the Delta-t solid line) should match the as-built better than the IST equation in Table 2 (the Equilib dash/dot line); however, the reverse occurred. Unfortunately, the as-built conditions were not identical to the test bed; specifically, the as-built had a lower temperature (26.1 °C versus 28.35 °C), lower superficial velocity (4002 versus 4052 kg/m^2h), and higher feed concentration (1490 versus 1440 ppm). Even with this off-set, the Constant-Δt method's error in estimating the as-built effluent times ranged only from 6% to 8%. The other trend in Figure 5 is that the Sorption Capacity method gives equivalent predictions to the Constant-Δt at low C/C_F specifications for t_b; however, at higher C/C_F specifications, the Sorption Capacity method deviates and begins to level off.

Figure 6 is the comparison of the scale-up methods with as-built information for Case Study B-MSH. Figure 6 shows that the Constant-Δt and Equilibrium Length predictions for the scaled-up t_b's are in good agreement with the as-built information ($t_{b,scale}$ errors range from -1% to 3%). However, the Sorption Capacity method begins to deviate for $C/C_F >$ 0.1 specifications for t_b. Since there is no isotherm data for Case Study B, the Equilibrium Length method used Eq. 24 to estimate the missing isotherm data.

The comparison against as-builts involves estimating breakpoint times for a known increase in bed length. In reduction-to-practice, it is likely that the scaled-up bed length is unknown, but the time of desired unit operation is known.

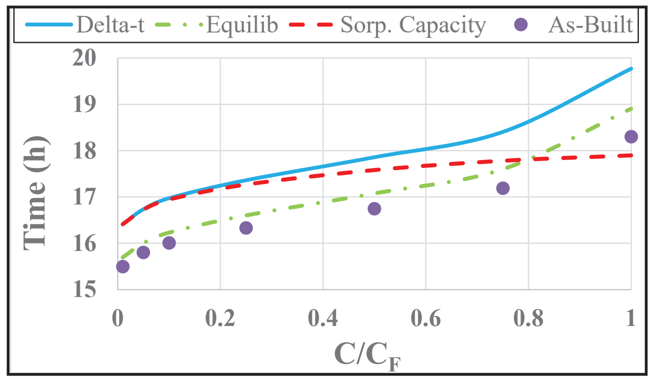

Figure 5. *Estimates of $t_{b,scale}$ vs a range of C/C_F specifications for the t_b. Data obtained from Case Study A-Collins (water vapor adsorption from nitrogen). Estimates for a 0.439 m bed length from 0.268 m test bed. As-built bed was 2.25 °C cooler than the test bed. The Constant-Δt error in estimating the as-built effluent times ranged from 6% to 8%.*

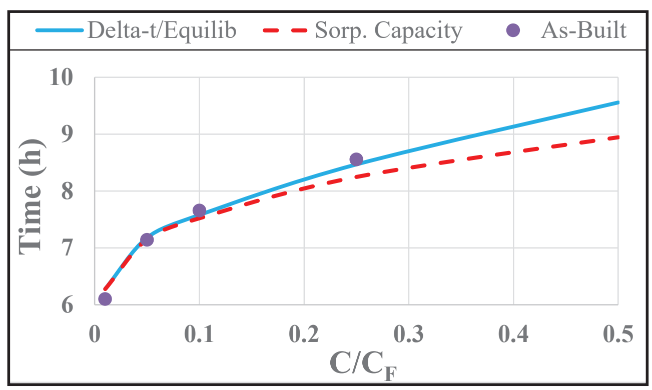

Figure 6. *Estimates of $t_{b,scale}$ vs a range of C/C_F specifications for the t_b. Data obtained from Case Study B-MSH (n-butanol adsorption from air). Estimates for a 16 cm bed length from 8 cm test bed. The Constant-Δt and Equilibrium Length (no isotherm data) methods give identical prediction curves. C/C_F limited to < 0.5 because of limited as-built information. The Constant-Δt error in estimating the as-built effluent times ranged from -1% to 3%.*

Therefore, Figure 7 compares scaling up unit operations so that the bed will need regeneration/replacement once per day (i.e., 24 hours). Two of the Case Studies are for loading the sorbent with a product; therefore, the economics of recovery of the product might lead to higher C/C_F specifications for t_b than environmental applications such as adsorption of a pollutant from a fluid. For this reason, we will compare the scale-up methods for the full range of potential C/C_F specifications. Since there is no isotherm data for the Case Studies B through D, the Equilibrium Length method used Eq. 24 to estimate the missing isotherm data. The result is that the Constant-Δt and Equilibrium Length methods give identical prediction curves in Figure 7.

In all four sorption systems in Figure 7, the Sorption Capacity method gives equivalent predictions to the Constant-Δt at low C/C_F specifications for t_b; however, at higher C/C_F specifications, the Sorption Capacity method deviates and begins to level off. The cause of these deviations in Figures 5, 6, and 7 is the Sorption Capacity assumption that $b_{scale} \approx t_{b,scale}$; a statement that Figure 4B clearly shows is limited to $t_b < t^*$. At low specifications of C/C_F for t_b, this assumption is adequate; but, at specification values of $C/C_F > 0.3$, this assumption is wrong.

POTENTIAL FIXES TO REDUCE THE PEDAGOGICALLY CONFUSING ELEMENTS

As stated in the Introduction, the existing LUB methods could be pedagogically confusing by either having the students calculate t_b by putting a known t_b into the limits of integration or telling the students to ignore the mass-transfer information contained in the bench or pilot scale data. For the Sorption Capacity method, the following are two alternative fixes. The first is to change the assumption to

$$LUB = L_{data}\left(1 - \frac{W_{b,data}}{W_{Max,data}}\right) \approx L_{data}\left(1 - \frac{t_{b,data}}{t^*_{data}}\right) \quad (29)$$

where $t_{b,data}$ is from observation instead of Eq. 1; this change means that the LUB of Eq. 29 equals LUB_{eq}. This change eliminates the need to limit the method to specifications of $C/C_F < 0.3$ and produces results equivalent to the Equilibrium Length and Constant-Δt methods. However, it is a much more complicated rubric of equations to get the same result as the simpler Constant–Δt method. The second alternative is to use Eq. 21 instead of the illogical Eq. 1 in teaching the method. This is mathematically less confusing;

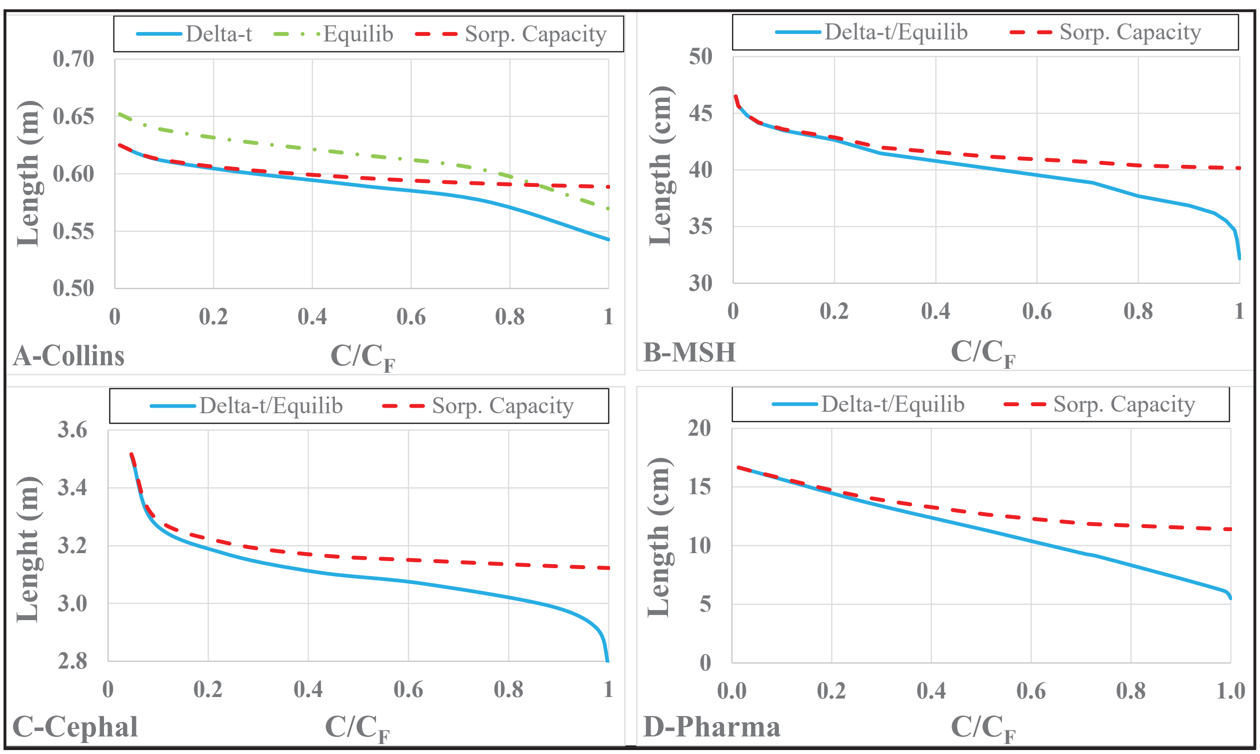

Figure 7. *Estimates of the required lengths of scaled-up beds to operate for 24 hours for each of the case studies in Table 3. In all four cases the Sorption Capacity method deviates for t_b specifications > 0.3; giving larger length estimates than the Constant-Δt method. Therefore, combining Figures 4B, 6, and 7 illustrates why the Sorption Capacity method is limited to t_b specifications < 0.3. When used without isotherm data, the Equilibrium Length method gives identical results to the Constant-Δt method.*

however, this would still limit the method to t_b specifications of $C/C_F < 0.3$. This author's final conclusion is that the limitation of the Sorption Capacity method to t_b specifications of $C/C_F < 0.3$ is a deal breaker.

The use of Eq. 27–28 in the Equilibrium Length method means that it will no longer ignore the test bed mass-transfer data. This change also eliminates the negligible solute content in the saturated void space assumption and the need to use isotherm data. In comparison to both the Equilibrium Length and Sorption Capacity methods, the proposed Constant-Δt method has fewer calculations and, therefore, potentially less error propagation (see Tables 1 and 2). The Constant-Δt method fixes the LUB's pedagogically confusing and complicated elements while giving equivalent results.

TEACHING MODULE

The following are some possible learning outcomes from a LUB Teaching Module:

- Use and program numerical integration
- Use dimensionless numbers for reporting data
- Work with imperfect or incomplete "real world" data
- Describe the scale-up concept of "Characteristic Quantities"
- Describe the value of bench scale or pilot plant data to design scaled-up systems that involve complex transport processes

The last bullet is clearer when using the proposed Constant-Δt method in contrast to the existing Equilibrium Length method.

Scale-Up Principles and Caveats That Should Be Included in the Teaching Module

Unlike many other scale-up methods in transport phenomena, the LUB methods do not depend on keeping dimensionless numbers, such as Schmidt or Reynolds numbers, constant. Instead, the LUB methods use "Characteristic Quantities" that need to be keep constant. The LUB methods assume that the fluid dynamics, mass transfer rates, and favorable sorption isotherms are constants. These three assumptions are valid if the following "Characteristic Quantities" are constant between the data and scale beds:

- Fluid dynamic and mass transfer rate quantities
  - Superficial velocity, G_F.
  - Sorbent particle size.
- Sorption isotherm quantities
  - Sorbent material.
  - Feed composition (i.e., concentration).
  - Temperature, especially for gas process streams. Ideally the initial bed and feed temperatures should be equal.[5]

An important outcome is noting that keeping characteristic quantities constant does not prevent changes in unit operation size or volumetric flow since G_F is volumetric flow divided by cross sectional area, neither of which are characteristic quantities.

The final quality of a LUB scale-up depends on the design of the test bed used to obtain the initial data. The on-line AIChE-Engage discussion groups highlighted that this step needs more weight in teaching LUB-methods. The result is the following content recommendations:

1. Use analytical solutions of fixed bed sorption to determine the appropriate test bed length and initial test range for the superficial velocity, G_F.
2. Ideal test bed length is greater than three mass transfer zones, Figure 2.
3. Ensure that the dimensionless Number of Transfer Units, N, is large enough to ensure "fast" mass transfer.
4. Set the ratio of test bed diameter to sorbent particle diameter large enough to eliminate wall effects.
5. If scaled-up bed will operate adiabatically, then the smaller scale test bed should be well insulated.[4]
6. Properly designed test beds result in scaling-up of only the loading step for the beds.[4] Unfortunately, the regeneration step is where the most deviation of the scaled-up bed from the test bed occurs.

Because textbooks present the analytical solution separated from the LUB method, there is a danger that students will not see the need to use both during a scale-up. The first recommendation, to place an analytical solution module before the LUB teaching module, will illustrate for the students the connection between bench scale testing and analytical models during engineering design via the dimensionless number

$$N \equiv \frac{k_{b.l.} a L}{G_F} \tag{30}$$

where $k_{b.l.}$ is the mass transfer coefficient (m/s) of the solute from the fluid to the sorbent particle and a is the mass transfer area per volume of the bed (m^2/m^3 of bed). The result is that the quantity $k_{b.l.}a$ has units of 1/s and represents the rate of mass transfer from the fluid to the sorbent. G_F/L, also, has units of 1/s and represents the rate of fluid movement past the sorbent particle. Therefore, N is the ratio of mass transfer to fluid flow, and the larger N is the more likely mass transfer is "fast" compared to the fluid flow. Unfortunately, the conventional name for N is "Number of Transfer Units," which does not communicate to the students its value in designing sorption beds with self-sharpening Mass Transfer Zones (Figure 2).

N, therefore, assists in the second recommendtion about appropriate length, L, in addition to helping with Recommendation 3, "fast" mass transfer. While obtaining data from the test bed, obtaining data for various G_F values will confirm the N is large enough for a valid LUB scale-up.

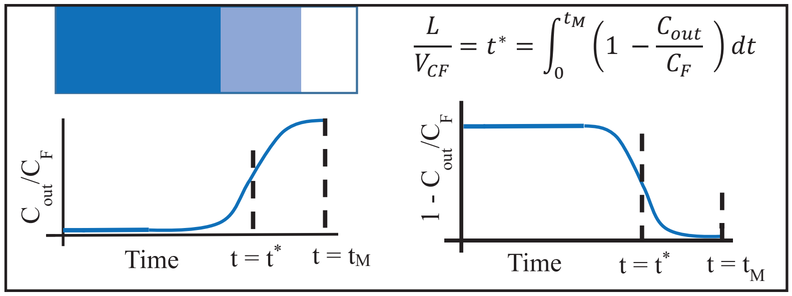

Figure 8. *A common misconception of students is to calculate the area under the left hand curve instead of the right hand curve when numerically integrating Eq. 12, the integral in the figure's upper right hand side.*

Student Difficulties Encountered

My experience, from teaching LUB Scale-up in numerous courses, is that the most difficult concept for students to apply is the numerical integration of Eq. 12. The difficulty is a misconception of which "area under the curve" they need to calculate (see Figure 8). A number of students will calculate the area under the left hand side curve in Figure 8 when they need to calculate the area under the right hand side curve.

Additional Suggested Module Content

A slide deck to aid in presenting a LUB Teaching Module would include elements of Figures 1-3 and 8 combined with Eq. 3-19 along with the bed loading equations, Eq. 23-24. Contact the author (scovazzo@olemiss.edu) for an example problem slide deck illustrating proper numerical integration.

The following modification of a homework problem from Harrison et al.[3] illustrates how to assess/reinforce some of the outcomes including the connection between t^* and Eq. 24.

Problem #1 (25%) <u>Scaling-up an adsorption column using Lab Data Method</u>

The following breakthrough data are from a laboratory scale adsorption process for the treatment of an aqueous solution containing 4.3 mg/liter of the antibiotic cephalosporin. The lab scale bed was 1 m long x 3 cm diameter, and the superficial velocity was 2 m/h. (Data modified from Belter, Cussler, Hu, *Bioseparations*, p. 174; Wiley, NY 1988.) Additional data: Sorbent Particle Size = 4 mm sieve size. Note: The absorbent is also manufactured in three different sizes (2 mm, 4 mm, and 6 mm).

TABLE P1
Breakthrough data

Time (hr)	4.7	6.5	7.3	7.8	8.1	8.7	9.3	10.3
C (mg/liter)	0.2	0.4	1.0	1.8	2.7	3.8	4.2	4.3

<u>Deliverables</u>

a. Calculate the break-point time for a scaled-up 3 m bed, defined here as occurring when $C/C_F = 0.1$, for the scaled-up column. Assume the same superficial velocity and a favorable adsorption isotherm.

b. Estimate the total cephalosporin recovered from the feed per column volume when the entire bed is in equilibrium with the feed, i.e., for times $\geq t_M$.

c. Part (b) is also an estimate of the equilibrium adsorbent loading, q_F, for the stated feed conditions (i.e., q_F for $C_F = 4.3$ mg/L), if you assume that the cephalosporin trapped in the void space, $\varphi = 0.3$, is negligible compared to the cephalosporin adsorbed on the adsorbent. Is this a good assumption? Numerically support your answer.

d. For the scaled-up bed, which particle size (2 mm, 4 mm, or 6 mm sieve size) will you specify and why? Here assume that the scaled-up diameter is > 2 times the laboratory scale bed diameter.

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