

## ION EXCHANGE AND ADSORPTION FIXED BED OPERATIONS FOR WASTEWATER TREATMENT - PART II: SCALE-UP AND APPROXIMATE DESIGN METHODS

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**Abstract:** In part I of the study the theoretical models and hydraulics of fixed bed operations are presented. In part II the issues of scale-up and approximate design methods are presented. Part II could be considered as a presentation of the path from the laboratory scale to full scale units. Finally, the methods for derivation of the experimental data needed are discussed.

**Key words:** ion exchange, adsorption, fixed beds, scale-up

### 1. SCALE-UP CONSIDERATIONS

#### 1.1. General analysis

The term “scale-up” should not be understood only in the sense of increasing the dimensions while preserving similar systems. Often, the transposition of one system to another offers a better solution to a problem (for example, a pilot plant operating in upflow for industrial unit operating in downflow) [1]. While the rate of a given chemical reaction is independent of the size and the structure reactor, the physical processes involved in the overall rate e.g. mass and heat transfer are usually controlled by these factors [2]. Furthermore, scale up will have much better chance for success if the laboratory and large scale units are carried out in the same type of system e.g. fixed bed, fluidized bed, batch reactors etc [3]. By this point of view, keeping the same reactor type in different scales and thus the same reactor structure, the critical parameter is the reactor size, which incorporates its geometrical analogies and dimensions. Furthermore, in the case of wastewater treatment in adsorption fixed beds, no real chemical reaction occurs nor heat transfer is a crucial subject as these operations are essentially isothermal. Thus, scale up is mainly consider mass transfer phenomena and of course the hydraulic performance of the reactor (liquid distribution, small-scale dispersion of the flow and liquid holdup).

Here a distinction should be made between a system that can be fully modeled mathematically, for example treatment of one component solutions in adsorption fixed beds and a system that such a modeling is not practically feasible, for example treatment of a complicated multicomponent solution in the same operation. In the case where full modeling is applicable, scale up can be based on the model keeping adequate flow conditions and geometric analogies in order to avoid extensive particle attrition, partial fluidization and large scale maldistribution of the flow as channelling, dead-spaces and fluid re-circulation.

Even in one-component systems because of the nonlinearity associated with the equilibrium expressions, a full solution of the resulting partial differential equations of the models requires complicated numerical solutions. In our days with the aim of computers and sophisticated software these solutions are easily found. However, to obtain a priori prediction of the breakthrough curve independent experiments and/or reliable engineering correlations are required to estimate the numerous equilibrium, transport and sorption kinetic parameters. As a result some of the model parameters are evaluated by fitting the model to experimental breakthrough curves. Such multiparameter fitting may reduce the physical significance of the parameters. By this point of view, scale up is often following other routes.

In the case where modeling is not applicable, apart from flow conditions and geometric considerations, scale up should be also based on some additional rules, called similitude rules. Then, under specified conditions the data of a laboratory-scale unit can be used for the evaluation of the large-scale unit performance. The similitude rules can be withdrawn using the continuity equation, rates equations and hydraulic equations (for %h and  $Pe_d$ ). From continuity equation it is clear that for the same ion exchange or adsorption system and the same  $C_o$ ,  $q_{max}$ , the exit concentration (C) at each time interval (t) will be the same for any bed size if the equation terms are the same. These terms are shown in Table 1.

Table 1. Parameters derived from the analysis of the design equations.

Parameters	Equation	Parameter is a function of:
E	Continuity equation	$D/d_p$
$Z/u_s$	Continuity equation	-
$Pe_L$	Continuity equation	$Pe_d$ , $Z/d_p$ or equally: $Z/d_p$ , $Re_p$
$D_s$	Rate equation for solid diffusion	-
R (or $d_p$ )	Rate equation for solid diffusion	-
$k_f$	Rate equation for liquid film diffusion	$\varepsilon$ and $Re_p$
$a_u$	Rate equation for liquid film diffusion	$d_p$ , $\varepsilon$ and particle shape
E	Rate equation for liquid film diffusion	$D/d_p$
R (or $d_p$ )	Rate equation for liquid film diffusion	-
h%	Liquid holdup equation	$u_s$ , $d_p$ , $\varepsilon$
Isotherm – Equilibrium behavior	Isotherm	Maybe influenced by contact time ( $Z/u_s$ )

\*The parameter ( $\rho_b$ ) is found in the continuity equation but is eliminated when the rate equation is incorporated in its expression.

Under some circumstances, the parameters in Table 1 may have not the same influence on the bed performance. In Table 2 some critical comments are summarized.

Table 2. Comments on the critical design parameters.

Parameter	Comments
$Z/u_s$	Critical
$Z/d_p$	Minimal effect if is higher than 150
$D/d_p$	Minimal effect if is higher than 30
$Re_p$	Minimal effect if solid diffusion is the controlling mechanism and the bed is operated in near plug flow mode (or for $Z/d_p$ higher than 150).
$d_p$	Critical
$\varepsilon$	Minimal effect if is higher than 10 ( $d_p/D < 0.1$ )
$u_s$	Minimal effect if solid diffusion is the controlling mechanism and the unit is operated in upflow mode. The same for downflow mode if the liquid holdup is 100%.

As it will be analyzed in the next paragraphs, in the case of fixed beds the critical parameters are the contact time and the particle size.

### 1.2. Scale up of adsorption and ion exchange fixed beds

There are some basic scale-up rules for each operation. In fixed bed adsorption and ion exchange operations are scaled up from laboratory tests in small-diameter bed, and the large unit is designed for the same superficial velocity and particle size [4]. Particle size should be kept the same, as it is well known that is affecting the rate of the adsorption and ion exchange [5-7]. Liquid velocity is considered as an important parameter also in other

studies. As is pointed out due to geometric considerations, it is not possible to keep linear velocity constant during scale up and only if the pilot plant has a very high ratio of height to diameter, compared to the large unit the linear velocity could be similar in the two units. However, contact time is more critical than superficial velocity. Contact time is incorporated in the continuity equation, representing the physical time allowed for the contact of the phases. The physical meaning of this is that contact time is the time allowed for the “reaction”. However, it is known that in some systems contact time is possibly influencing the equilibrium state, leading in a “partial” equilibrium behavior, in the bed at least in reversible and of low kinetics liquid phase adsorption and ion exchange processes [8]. It is important to be noted that this effect is essentially another “non ideality” which is not incorporated in the continuity equation. Thus, scale up should be based on the same contact time and particle size.

Constant contact time has been used as an essential factor for scale up of adsorption columns, provided the degree of maldistribution of the flow is very small, and for scale up of ion exchange (resin) columns for the removal of radionuclides [9,10]. Contact time is also used as the basic scale up factor in two-phase flow (gas-liquid) fixed beds in the common case of liquid reactant limited systems [11,12]. Again, the full scale column, being taller than the experimental pilot or laboratory one, at the same contact time (or space velocity) has a much larger liquid mass velocity which causes improved contacting efficiency.

Contact time (or space time), is determined by the linear velocity as follows:

$$\tau = \frac{\varepsilon}{I} = \frac{V_o \cdot \varepsilon}{Q} = \frac{Z \cdot \varepsilon}{u_s} \quad (1)$$

Where; (I) is the relative volumetric flow rate commonly expressed in empty bed volumes per hour (BV/h). Essentially, (I) is equal to the reactor space-velocity [13].

From hydraulics perspective, if scale up is based on the same superficial velocity, in downflow operation  $Pe_L$  will be higher in large bed due to higher bed height, while liquid holdup will be low due to the low velocity, which is frequently used in laboratory scale beds (see Part I, eq. 32-34). This leads to problems and special efforts are needed in order to improve the liquid holdup e.g. special distributor design. These problems are absent in upflow operation.

On the other hand, keeping the same contact time, linear velocity is much higher in the large column and this is may be not a problem as velocity influences the controlling step (mass transfer in liquid film) and hydraulics (maldistribution and liquid hold-up): in the large column the higher velocity is leading the system to solid diffusion control, higher liquid holdup (for downflow operation) and higher  $Pe_L$  (nearer to plug flow), if of course liquid dispersion in the top of the column is well designed (see Part I, eq. 32-34). In this case, the results are expected better i.e. higher breakthrough volume will be achieved due to the fact that the liquid film resistance could be minimized and, for downflow operation, liquid holdup and  $Pe_L$  are expected high enough. Adsorption from liquid phase using porous solids is frequently controlled by the solid diffusion step. By this point of view, the influence of superficial velocity is expected to have a minimal effect on the bed performance, provided that the flow is near plug flow and the liquid holdup is near to 100%. Thus, if scale up is base on the same contact time the results from laboratory scale bed can be directly used in large bed and the performance of the bed can be accurately evaluated. Consequently, if we have a contact time-breakpoint volume relationship derived from the laboratory bed experiments, we can use this relationship to evaluate the breakpoint volume of the large-scale unit for the specified contact time.

**It should be noted that in this** case, when modeling is applicable in laboratory scale bed, the possible change in the controlling mechanism should be taken into account and different model type is likely to be needed in large-scale bed.

From the above analysis some important conclusions can be withdrawn:

- In the case of high liquid film resistance keeping the same contact time in large unit, the higher superficial velocity is leading to the elimination of this resistance and thus higher rates can be achieved

in the large bed. However, in the same time downflow operation is probably the best operation mode due to the fact that high superficial velocities in large bed could lead to partial fluidization and particle attrition in upflow operation. Furthermore, different type of model is probably needed in the different scales;

- In the case of low resistance in liquid film or/and unfavorable equilibrium (equilibrium-limited system) high contact time in large bed would be beneficial for the equilibration step while in the same high superficial velocity is not expected to lead in better results, as the liquid film resistance is minimal. Furthermore, the same type of model can be used in the different scales.

Considering the liquid distribution in downflow operation, several liquid distributors could be used as weir-through, simple slotted-pipe and perforated-pipe distributor [2, 14]. For the later the design procedure is rather simple and is presented in the literature [15]. Finally, reproducible and appropriate bed voidage should be ensured in large beds in order to avoid dead volumes. A suitable method called “snow storm filling” is presented in the literature [16].

During this part of the design it should be kept in mind that the same controlling mechanism in the different sized units will permit experimental data to be straight “transferred” from the small to the large unit. In most of the cases, diffusion of molecules or ions within the solid matrix is the controlling mechanism, especially in micro- and mesoporous materials as zeolites. Film diffusion resistance is reverse proportional to linear velocity and as linear velocity is increased in large scale unit film diffusion is not important in most of the cases. Consequently, if in laboratory scale units the controlling mechanism is solid diffusion the change in linear velocity (under the same contact time) is not influencing the kinetics of the system. This then allows flow rate to be varied during scale up. However, even in the case where in small columns the controlling mechanism incorporates liquid film resistance it is safe to consider that the adsorption rate will be higher in larger columns due to the increase of linear liquid velocity. Contact time in two scales will be the same only if the liquid holdup in both scales is the same and in the ideal case near to 100%. Since liquid holdup is expected high enough in large-scale column, provided that liquid distributors are designed properly especially in downflow operation, it is desirable to achieve high liquid holdup also in small columns. This, could be achieved only under upflow condition. Upflow reactor is also preferred in two-phase flow systems in the common case of liquid limited reactions [12].

In order to have near-ideal plug flow in small scale bed particle Peclet number should be high enough to have a high bed Peclet number, while in large scale unit this particle Peclet number is of minimal importance since it is multiplied by  $(Z/d_p)$  and thus the bed Peclet number is expected high enough. For example, in the typical case of 2 mm particle size and a small bed of 20 cm height the multiplier is 100, while in large bed of 200 cm height the multiplier is 1000. Thus the bed Peclet number is 10 times higher in the large column, provided that the particle Peclet number is similar in both beds. It should be noted here that particle Peclet number are not expected to be similar in both scales, as it is depended on the flow condition (up and downflow) and on Reynolds number. However, in the case of irregular-shaped (zeolite-like) particles using the correlations of Inglezakis et. al. [17] it is clear that particle Peclet number are close enough for both flow conditions in the range for  $4 < Re < 8.5$ . For spheres this is happen in a broader range e.g.  $Re < 10$  (see hydraulics analysis, Part I).

In order to ignore the geometrical similarity between the different unit sizes the following conditions should be satisfied [7]:

$$\frac{Z}{D} \geq 5 \dots \dots \frac{D}{d_p} \geq 30 \quad (2-3)$$

Also, the scaling factor for bed diameter should be kept equal or lower than 10 [18]. Using the above relationships the limits of volumetric flow rates could be evaluated. Here (I) is the relative volumetric flow rate in Bed Volumes/h: is ( $\beta$ ) for treatment and ( $\alpha$ ) for pretreatment/regeneration (generally is  $\alpha > \beta$ ):

$$Q = I \cdot V = I \cdot Z \cdot A \Rightarrow A = \frac{Q}{I \cdot Z} \Rightarrow D = \left( \frac{4 \cdot Q}{I \cdot \pi \cdot Z} \right)^{0.5} \quad (4)$$

Then the following equations hold:

$$\begin{aligned}
 D &\leq \frac{Z}{5} \Rightarrow \left( \frac{4 \cdot Q}{I \cdot \pi \cdot Z} \right)^{0.5} \leq \frac{Z}{5} \Rightarrow Q \leq \left( \frac{Z}{5} \right)^2 \cdot \frac{I \cdot \pi \cdot Z}{4} \\
 D &\geq 30 \cdot d_p \Rightarrow \left( \frac{4 \cdot Q}{I \cdot \pi \cdot Z} \right)^{0.5} \geq 30 \cdot d_p \Rightarrow Q \geq (30 \cdot d_p)^2 \cdot \frac{I \cdot \pi \cdot Z}{4} \\
 D_{FS} &\leq 10 \cdot D_{LS} \Rightarrow \left( \frac{4 \cdot Q_{FS}}{I \cdot \pi \cdot Z_{FS}} \right)^{0.5} \leq 10 \cdot D_{LS} \Rightarrow Q_{FS} \leq (10 \cdot D_{LS})^2 \cdot \frac{I \cdot \pi \cdot Z_{FS}}{4}
 \end{aligned} \tag{5-7}$$

Where: subscript (LS) is for laboratory scale bed and (FS) for the large unit. Using equations (5-7) the appropriate limits of working volumetric flow rates can be evaluated. Thus, the full scale bed diameter can be evaluated using equation (4) according to the chosen working volumetric flow rate.

### 1.3. Bed voidage considerations

In order to use safely the above analysis, the bed voidage should be approximately the same for both units. Bed voidage is a function of  $d_p/D$  [19]. However, for small  $d_p/D$  values (typically lower than 0.1) the bed voidage is practically constant. As in practical applications  $d_p/D$  is kept lower than 0.1, the bed voidage could be considered the same in both scales. For example using particles of 2 mm diameter the bed diameter should be greater than 2 cm. This is critical then only in laboratory scale units. In Figure 1 Dixon's correlations are presented for spheres and cylinders, in the case of  $d_p/D < 0.5$  [19]. For cylinders ( $d_p$ ) is equal to diameter of equal volume sphere.

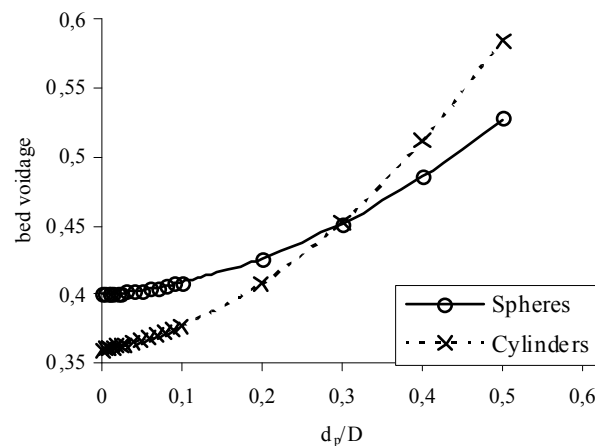


Fig. 1. Bed voidage v.s.  $d_p/D$ .

It is obvious that for  $(d_p/D) < 0.1$  the differences of bed voidage are small while for  $(d_p/D) > 0.1$  the bed voidage is greatly affected by diameter ratio. For irregular shaped particles measurements should be conducted in order to evaluate bed voidage and its dependence from  $(d_p/D)$ . For example, for irregular shaped particles of zeolite (clinoptilolite) the bed voidage was measured to be in the range of 0.48-0.51 for  $(d_p/D)$  between 0.019 and 0.074 [20]. Finally, as pressure drop is very sensitive to the bed voidage, the ratio  $(d_p/D)$  is greatly influencing pressure drop across the bed in the case of  $(d_p/D) > 0.1$  [16, 21]. Again this is happen because for  $(d_p/D) > 0.1$  bed voidage is changing considerably (Figure 1).

## 2. DERIVATION OF BASIC EXPERIMENTAL DATA

Some preliminary experiments in laboratory-scale beds are essential. In the laboratory, a series of measurements can be taken concerning all of the mechanisms that are independent of size. This applies in particular to

everything related to thermodynamics and chemical kinetics [1]. The laboratory reactor should not necessarily be similar to the idea we have of the industrial one but has to be designed in order to give the best information [22].

In order to investigate the chemical behavior of the system non-idealities in the flow should be minimized. For this reason, laboratory experiments should be made in upflow mode, since in such small scale beds in downflow mode high liquid maldistribution and low liquid holdup are expected and the results may be not usable [17]. For example in adsorption and ion exchange operations where zeolites or zeolite-like materials are used the need for high residence time and high liquid holdup (>90%) in downflow mode is leading to operations that should work at low relative flow rates (<25 Bed Volumes/h) and long columns (bed height higher than 100 cm) [23]. However, in laboratory scale beds (where bed height is frequently lower than 100 cm) the relative volumetric flow rate has to be high (>25 Bed Volumes/h) and improper for ion exchange and adsorption operations. In order to avoid this situation, upflow is recommended for small-scale laboratory columns and downflow in large columns. On the other hand in upflow mode liquid holdup is kept near 100%, and thus the theoretical contact time is also the real contact time of the system. Furthermore, if the packing material require a pretreatment or regeneration step (usual in zeolite applications) this should be also studied in full detail [24].

The minimal set of data from this step, that are needed for the proposed short-cut design are the bed diameter ( $D_{LS}$ ), particle diameter ( $d_p$ ), relative volumetric flow rates (Bed Volumes/h) for treatment ( $\beta$ ) and pretreatment/regeneration ( $\alpha$ ) and the achieved breakpoint volume (Bed Volumes) for the selected volumetric flow rate (treatment) and breakpoint concentration (typically this value is equal or lower than 10% of  $C_o$ ). Breakpoint concentration is the effluent pollutant concentration, which is equal to the allowable concentration set by the environmental laws and regulations. Obviously, the fixed bed operation is stopped at this point and the performance of the unit is based on the achieved treated volume (breakpoint volume). Simple correlations between contact time and breakpoint volume could be valuable information in scale up procedure. Breakthrough volume is considered to be a primary parameter for design purposes.

In experimental section it could be valuable to determine the following: the maximum adsorption capacity of the solid ( $q_{max}$ ), the diffusion coefficients of the solutes in the solid phase ( $D_s$ ) and the equilibrium isotherm. Generally, all the above measurements are made in batch systems using simplified models for kinetics, as Paterson's approximation for solid diffusion coefficients and Langmuir equation for isotherms and maximum adsorption capacity of the solid ( $q_{max}$ ) [7]. The maximum adsorption (or ion exchange) capacity of the solid ( $q_{max}$ ) is frequently measured separately by the "repeated equilibrations method" in batch systems [8].

Theoretically, the equilibrium isotherm, the maximum adsorption capacity and the solid diffusion coefficient, for a specific ion exchange or adsorption system, are independent of the experimental method used for their determination and independent of flow conditions [6,7]. However, experimental results obtained in ion exchange and adsorption systems showed that if the maximum adsorption capacity is measured in fixed beds is different from that measured in batch systems and it might be flow rate dependent [8]. Furthermore, as shown in the cases of adsorption on activated carbon and the removal of Cr (III) using zeolite NaX, the equilibrium isotherm is influenced by the type of reactor used for its measurement (fixed bed and batch reactor) [25,26]. Finally, the diffusion coefficient may be different when measured in fixed bed or batch systems and in some cases is found to be flow-dependent [8, 27]. All these observations seem to be a result of the limiting (low) contact time in fixed beds and of the different concentration gradients in fixed beds and batch reactors [8, 28]. It must be recognized that batch reactors do not approximate the hydrodynamic and contaminant removal patterns of fixed bed (column) reactors [27]. Another reason responsible for such differences between batch and fixed bed parameters is the lack of adequate mechanical strength of particles (compressed particles due to flow pressure) and permeability of particles to fluid flow [29]. Obviously, this case is possible only when resins are used.

The maximum adsorption (or ion exchange) capacity can be measured using the experimental breakthrough curve ( $C$  v.s.  $V_{eff}$ ) by integration [2, 6]:

$$q_{\max} = \frac{\left( V_{\text{eff},T} - \int_{V_{\text{eff},B}}^{V_{\text{eff},T}} X(V_{\text{eff}}) \cdot dV_{\text{eff}} \right) \cdot C_o}{\rho_b \cdot V_o} \quad (8)$$

Where: ( $V_{\text{eff},B}$ ) is the effluent volume until the first appearance of the solute in the exit stream and ( $V_{\text{eff},T}$ ) is the effluent volume until the exit solute concentration is equal to its inlet concentration.

The dependence of ( $q_{\max}$ ) by residence time have been noticed and modeled elsewhere, in the case of zinc and acid dyes adsorption by bone char and activated carbon respectively [30]. The following equations were successfully applied

$$\begin{aligned} q_{\max}^t &= q_{\max}^0 \cdot [1 - \exp(-\omega \cdot t_{\text{res}})] \\ q_{\max}^t &= q_{\max}^0 \cdot [1 - \exp(-\omega \cdot t_{\text{res}}^{0.5})] \end{aligned} \quad (9-10)$$

Where: ( $q_{\max,t}$ ) and ( $q_{\max,o}$ ) are the bed maximum capacity and the real maximum capacity (measured in batch reactor systems), ( $t_{\text{res}}$ ) is the residence time and ( $\omega$ ) is a system-specific constant. It is obvious that if residence time is infinite the bed maximum capacity is equal to the real maximum capacity, which is theoretically expected, as noticed elsewhere [8]. These equations have been also tested in  $\text{Pb}^{2+}$  adsorption by clinoptilolite (zeolite), and showed satisfactory results [20].

As a conclusion, the maximum adsorption capacity should be measured in fixed bed experiments under dynamic conditions and if models are applicable, diffusion coefficients should be also determined in fixed bed apparatus. Due to the fact that equilibrium isotherms requires extended data series and thus are time consuming experiments, are quite difficult to be conducted in fixed bed reactors and by this point of view, it is more practical to be evaluated in batch reactor systems. Then, it is known that when applying fixed-bed models using an equilibrium isotherm obtained in batch-type experiments, the equilibrium discrepancy (if exists) can be compensated by a different estimate for the solid diffusion coefficient [8, 25].

### 3. SHORT-CUT DESIGN METHODS

It should be clear that the presented design steps can not be followed in “linear” sequence. Some “loops” are necessary. For example no experiment is reliable if the hydraulics of the laboratory scale beds are not properly studied and applied.

#### 3.1. General approach

The general approach is usable when modeling is failing to represent the system or in the cases of a complicated multicomponent system. The short-cut design procedure can be described step by step as follows:

1. Conduct of laboratory fixed bed experiments using upflow operation working in near plug-flow conditions. Also, keep ( $d_p/D$ ) lower than 0.1. The following minimum set of parameters should be specified: the bed diameter ( $D_{LS}$ ), particle diameter ( $d_p$ ), bed voidage ( $\epsilon$ ), relative volumetric flow rates (Bed Volumes/h) for treatment ( $\beta$ ) and pretreatment/regeneration ( $\alpha$ ) and the achieved breakpoint volume (Bed Volumes) for the selected volumetric flow rate (treatment) and breakpoint concentration. Furthermore, evaluate ( $q_{\max}$ ) for each relative volumetric flow rate;
2. Correlate the contact time-breakpoint volume and contact time-( $q_{\max}$ ) data. The later could be correlated using equations of the form of eq. (9-10);
3. Using the appropriate approximate methods presented determine the controlling mechanism and specify the linear velocity limit above which the rate is determined by solid diffusion;
4. Determine the maximum working velocity, using eq. (40) for upflow and downflow operations. Then determine the maximum bed height using eq. (41-42), see Part I;
5. Select a bed height-linear velocity couple in order to have the same contact time as in laboratory scale bed for treatment and pretreatment (bed height should be the same). Examine if the controlling step is changing, using the appropriate method;

6. Determine the volumetric flow rate limits and select the appropriate rate according to the needs of the operation, using eq. (5-7). Then determine the bed diameter using eq. (4);
7. Evaluate the hydraulic performance of the bed e.g. Peclet number and liquid holdup, using eq. (32-36), see Part I. If these parameters are not satisfying then go to step 4 and change the working linear velocity. Repeat the steps 4-7 until all restrictions are satisfied;
8. Determine the number of beds to be used according to the maximum flow rate of the waste. If the number of beds is very high (impractical) the use another contact time from step 2. Repeat the steps 2-8 until the results are satisfying;
9. Determine the number of operation cycles for each bed according to the maximum (total) volume of the waste to be treated.

### 3.2. One-component mass transfer controlled systems

This procedure is applicable when one component systems or multicomponent systems which can be considered as one component are to be treated. When modeling ("diffusion" or Thomas models) is applicable the above procedure could be used with some modifications:

- Step 1. During this step, experiments should be conducted using different contact times (or linear velocities) and model equations should be applied in order to find the controlling mechanism and to evaluate model parameters. Also, work in upflow mode in near plug flow conditions and keep  $(dp/D)$  lower than 0.1. Several batch experiments should be used for the determination of isotherms in the case of "diffusion" models. Also, keep  $(d_p/D)$  lower than 0.1;
- Step 2. Only a contact time –  $(q_{\max})$  correlation is useful. Breakpoint volume could be evaluated using the model equations;
- Step 5. There is no need to keep the same contact time in two scales (except for pretreatment).

### 3.3. One-component equilibrium limited systems

This procedure is applicable when one component systems or multicomponent systems which can be considered as one component are to be treated. When equilibrium is highly unfavorable the General procedure (A) could be used with some modifications:

- Step 1. Batch experiments should be conducted in order to evaluate the equilibrium isotherm relationship. Bed experiments could also be used. In any case,  $(q_{\max})$  is better to be measured using the experimental breakthrough curves. Also, work in upflow mode in near plug flow conditions and keep  $(dp/D)$  lower than 0.1;
- Step 2. Only a contact time –  $(q_{\max})$  correlation is useful. Breakpoint volume could be evaluated using the model equations;
- Step 3 is not needed;
- Step 5. There is no need to keep the same contact time in two scales (except for pretreatment).

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