LOADING AND DRAINING OF PERIODICALLY OPERATED TRICKLE BED REACTOR

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ABSTRACT

Periodic interruption of liquid flow in cocurrent trickle beds appears to be an attractive mode of operation. For modelling these intermittent flow reactors, loading and draining times must be known. Experiments were undertaken using beds of activated carbon with water and air as the fluid phases. Loading time was taken as water breakthrough. The gas flow was continuous while the time between the end of drainage and the start of filling was varied to simulate different periods. Drainage experiments followed the liquid flow leaving the bed as a function of time. Liquid hold ups were determined after the filling and draining measurements. Variable considered were particle size, gas and liquid velocities. Loading closely follows the plug flow model; drainage shows tailing but does not follow literature models. Static and dynamic hold ups at zero gas flow agree with literature correlations for the larger particle size used. A gas velocity effect on both static and dynamic hold up was observed.

INTRODUCTION

Haire et al. (1983) found that periodic interruption of liquid flow in cocurrent trickle beds increased the rate of an exothermic catalytic reaction in which both reactants enter the reactor in the gas phase. Because the increase is about 40 to 50%, this type of periodic operation merits more detailed study. Periodic flow interruption was considered for the scrubbing of SO2 from stack gas (Haire et al., 1989) because sulfur can be recovered as a moderately concentrated sulfurous acid. A model for flow interruption has been proposed that neglects the time needed to fill the bed when liquid is readmitted and the time to drain the bed when the flow is closed off (Haire et al., 1990; Stegman et al., 1992). It is evident that filling and draining limit how short a cycle can be in operations with flow interruption. A further question that arises out of model development was how large should the hold up be when (i) only gas flows through the bed and (ii) both gas and

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liquid flow through it. In other words, whether periodic operation influence the hold up was questioned.

A literature survey encompassing the last 20 years failed to disclose any research on the flow time required to establish hydrodynamically steady flow through the column. Also, the character of the flow in the bed during the filling phase seems not to have been investigated. On the other hand, there is a rich literature on gravity drainage from packed beds. Unfortunately, this literature deals primarily with drainage from filter cake or centrifuge solids, the particle size of which is often more than two orders of magnitude smaller than trickle bed packing and thus capillary forces prevail. However, gas flow was either upward or countercurrent to the liquid flow. Cocurrent gas flow was treated by Wakeman (1979, 1982). Therefore, it seemed to be useful to study the drainage behaviour experimentally. The literature was helpful for interpreting the data obtained. The question of hold up in periodic flow stoppage, of course, had to be answered experimentally.

Objectives of this study were thus 1) to measure the filling time, defined as the time after flow starts to when liquid begins to fall from the bottom of the trickle bed, 2) to determine the flow character during filling, 3) to measure the rate of drainage as a function of time, and 4) to measure the liquid hold up when both gas and liquid flow concurrently through the bed (referred to as static hold up). In addition, the influence of particle size at to levels, gas velocity at three levels and liquid velocity and water saturation of the air at two levels on the above group of measurements were examined.

Experimental Equipment

The schematic diagram is shown in Figure 1. Because the experiments were carried out in support of experimental and simulation studies of periodic interruption, trickle bed dimensions, packing materials used and particle size, and gas and liquid velocities were chosen to mirror earlier periodic operation studies. Filling and drainage behaviour was observed using a 5.6 cm cylindrical trickle bed. The trickle bed wall was lucite, so that the filling and the draining flows could be observed. Granular active carbon with 0.8 and 3.5 mm particle sizes was used as packing. The bed was 40.4 cm high and supported by a fine stainless steel screen. The gas phase was saturated air, which was filtered.

Each activated carbon charge was dried and weighed. A stopwatch with a resolution of 0.1 s was used. Filling experiments were begun by passing air through the bed, then at time zero, liquid was fed to the bed. Air flow was continuous. The time was recorded when water first fell from the bottom of the bed. Flow thereafter was collected in a shallow graduated cylinder and the volume charge with time determined.

The drainage rate was monitored by accumulating the liquid leaving the bed in graduated cylinders and noting the increase in volume with time until liquid flow from the bed ceased. The volume collected during the drainage experiment measured the dynamic hold up, defined as the fraction of the interparticle void volume filled with liquid during liquid flow through the bed less the static hold up.

Static hold up was determined by weighing the bed after drainage was complete and subtracting the original weight of dry bed. Void volume can be calculated from the weight of carbon and the porosity of the BPL activated carbon.

Filling Behaviour

Figure 2 shows the filling time per meter of activated carbon vs the superficial liquid velocity, i.e., the volumetric rate of water feed divided by the bed cross sectional area. Other parameters in the figure are the carbon mean diameter and the superficial air velocity through the bed. The filling time is the time for the first liquid to drop from the bottom of the bed after water flow to the bed is started. The figure shows that this filling time is independent of the carbon particle size and the gas velocity.

Water leaving the bed was collected and measured as a function of time. Figure 3 shows that flow leaving the bed is a linear function of time and is identical to the water feed rate. This means that for the bed dimensions and flow rate used, liquid moved through the bed in plug flow. Within the limits of measurement accuracy, fingering does not occur. The plug flow character was observed in all experiments including those at higher gas velocities and experiments in which liquid was retained in the bed from previous filling and draining steps.

Figure 4 compares filling times for three different filling conditions. The bottom group of curves represents filling times when water is retained in the bed. These measurements were intended to simulate the technique of periodic flow interruption investigated by Hau et al. (1989, 1990). The gas flow is continuous in this operation so that after the liquid flow is interrupted, leaving residual water in the porous carbon particles and at particle interstices, evaporation continues. Evaporation removes this hold up and thus the filling times increases. The difference in filling times is discernible at low liquid velocities (i.e., between 5 min of flow interruption and 60 min), but at velocities 6 to 8 cm/s or greater, it cannot be measured. Data were obtained for only the 3.6 cm diameter particles.

The uppermost line in Figure 4 and the accompanying data points provide filling times for a dry bed of carbon. The line and the data points for the 3.6 cm particles were taken from Figure 2. It is evident from a comparison of the dashed line and the data points for periodic flow interruption (lower cluster of lines) that even after 60 min of flow interruption, the carbon bed contains almost all of the liquid retained from the previous dosing.

The solid line in Figure 3 above the dashed line for filling of the dry bed is the calculated line, assuming liquid plug flow and no gas flow in the bed. It represents the maximum filling time. The difference between the two lines is the gas hold up in the bed.

Filling times for any bed depth can be estimated from Figure 4. They can also be obtained by dividing the volumetric liquid flow rate into the product of the difference between the total hold up under liquid flow and the total hold up with low gas flow through the bed times the bed void volume. In terms of dynamic and static hold up:
Draining Behaviour

All the experimental data collected are shown in Figure 4 as the liquid collected vs. time after the water flow was halted. Draining is a slower and much more complex process. Figure 5a shows the draining behaviour when the gas flow has also been terminated. This is typical of gravity flow data in the literature (Wakeman and Vince, 1986) and shows a particle size effect that would be expected for laminar flow in the draining bed. In contrast to published data, there is an effect of the water flow rate just prior to flow interruption. This and the effect of the gas superficial velocity are examined in Figure 5b and 5c for the 0.8 mm and 3.6 mm particle respectively. Comparison of the curves for $V_g = 5.1$ mm/s and 13 mm/s at $V_v = 0$ illustrated the water flow-rate effect. This effect arises from dynamic hold up that is a function of the liquid loading. The effect of gas velocity can be seen by comparing the curves for $V_v = 0$ and $V_v = 60$ mm/s for $V_v = 17$ mm/s in Figure 5b. Visually the same behaviour can be seen in Figure 5c for the 3.6 mm particles using the curves for $V_v = 0.80$ mm/s and $V_v = 5.4$ and 13 mm/s.

Although drainage from beds in the presence of gas flow has been both measured and analyzed (Wakeman, 1979, 1982), conditions in this study differ because gas flow occurred prior to drainage and continued at the same velocity during drainage. In Wakeman's studies, gas flow begins with drainage and pressure drop rather than velocity is maintained constant.

Despite system differences, one would expect the same physical influences to be important. In Wakeman's studies, the knee of the drainage curve results from two processes: the dropping liquid head in the bed as drainage proceeds followed by a slower process of film or interstitial drainage from the particles (Wakeman and Vince, 1986). A sophisticated analysis of the $V_v = 0$ drainage is given by Wakeman and Vince, who allow for capillary pressure and for the effect of liquid retention in the bed on the Darcy's Law permeability. This leads to liquid retention or static hold up that varies with bed depth. The hold up is very small at the upper surface of the bed and increases slowly with depth before rising rapidly to almost the pre drainage hold up at the bottom of the bed. The Wakeman analysis assumes $b = 0$ at $t = 0$ and that only gravity acts on the liquid. Although the analysis could be modified for $b < 1$ and gas phase drag (following Wakeman, 1982) a numerical solution is necessary; thus, the analysis leads to a drainage chart specific to bed and liquid properties.

For purposes of modeling periodic liquid flow interruption, or determining the shorter practice cycle periods, the detail provided by the Wakeman and Vince treatment is unnecessary and generalized drainage charts are inconsistent. Thus, for the analysis of the data in Figure 5, an analysis presented by Nenninger and Storrow (1958) has been adapted. Only liquid external to the porous carbon particles is considered. This is justified by a mean pore diameter in the carbon of about 2 mm. Water wets carbon preferentially so that the particle interior is non draining. The hold up is assumed to be uniform through the bed after the drainage is complete. Following Nenninger and Storrow, this static hold up is assumed with capillary pressure that can be expressed as liquid height, $h_l$. If $h_l$ is the momentary liquid height in the bed, Darcy's law for liquid drainage through the bed of solids can be written as

$$ dQ/dt = (h_l - h_s)/h_p (s_k \cdot k \cdot A / g) $$

(2)

The dynamic-plus static hold up in the presence of liquid flow is re written as $Q(t)$ at $t = 0$ and is assumed independent of axial position. Thus, at any time $t > 0$

$$ Q(t) = (h_l - h_s)/h_p (s_k \cdot k \cdot A / g) $$

(3)

where $Q(t)$ is the external liquid remaining in the bed. Eliminating $h_l$ from Eq. (2), integrating this expression from $Q_0 = h_l h_p (s_k \cdot k \cdot A / g)$ at $t = 0$ to $Q(t)$ at $t$, substituting in the final liquid volume $Q_{l_e} = (h_l - h_s)/h_p (s_k \cdot k \cdot A / g)$ at $t = 0$ and $Q_l(t)$, yields

$$ Q_l(t) = (h_l - h_s)/h_p (s_k \cdot k \cdot A / g) $$

(4)

Equations (4) have two parameters that must be evaluated from experimental data and $Q_0$. In Eq. (4), $Q_0$ and $Q_l(t)$ can be read from the drainage curve so both $s_k$ and $Q_0$ should be calculable. Indeed, if $h_l > h_s$ then it can be calculated from the slope of the drainage curves $s_k > 0$.

When this model was tested against the data shown in Figure 4, $Q_0$ assumed a negative value. Therefore, it is concluded that the analysis of Wakeman and the simpler analysis of Nenninger and Storrow are not applicable to this situation. These authors deal with a bed of solids supported on a screen. For this reason, the capillary pressure expressed as a liquid height must be negligible. Indeed, the knees shown in Figure 5 is probably due to drop formation beneath the screen supporting the packed bed as the liquid drains from the bed.

Because the amount of liquid drained after the falling head approach the screen is quite small, this amount is neglected and it is assumed that the draining time can be approximated by the time it takes for the liquid head to move through the bed. This neglects the logarithmic term on the right-hand side. The draining behaviour can be described by means of the Darcy coefficient $k$, now. This was calculated from the first two measurements of the drainage collected within one minute of interruption of liquid flow.

Figure 6 shows values of the Darcy coefficient $k$ as a function of the superficial gas velocity for the 0.8 mm and 3.6 mm carbon particles. Individual data points are labelled with the liquid velocity. It is apparent that particle size has just a small effect on the Darcy coefficient at gas velocities above 32 mm/s. However, the coefficient increases with the gas velocity as expected, up to 22 mm/s, indicating that the higher drag of the gas flow forces the greater volume of liquid through the packed bed.
coefficients of Figure 5 permit an estimate of the draining time for any bed depth for the particle sizes and velocity ranges used in the experiments.

Draining experiments were carried out using dry air and water saturated air, because it was thought possible that draining might require along enough time for evaporation to influence the drainage behaviour. Air saturation, however, did not affect drainage and in Figures 4 and 5 data for flows of saturated and dry air have been averaged. Points for different liquid velocities prior to flow interruption as shown in Figure 5: V_L' is important when there is no air flow through the bed. The Darcy coefficient, k, increases with increasing V_L. As V_L increases, the effect of V_D on k decreases. Liquid velocity changes δ_L so the influence suggest that δ_L = δ_D should be used in Eq. (2) in place of p.

Hold Up

Static hold up is the liquid held in the interstices of the wick bed packing and retained in the bed by capillary forces when liquid flow goes to zero. It generally correlates with the Eotvos number, the ratio of gravitational and surface tension forces.

Figure 7 plots the static hold up as a function of bed volume vs. the Eotvos number. Because the Eotvos number is independent of liquid and gas velocities, results in this study are given at Eo = 0.17 and 3.4. Most published data, including points from other studies shown in the figure, are at V_D = 0. Experimental values in Figure 7 at V_D = 0 scatter at Eo = 3.4. If the mean our data at each Eotvos number is used, the static hold up displays a higher dependence on this parameter than do published data.

With gas flowing through the bed, δ_D is about 50% lower at δ_D = 0.8 mm. Experimental data at δ_D = 3.6 mm are close to zero at V_D = 0 mm/s and fall off the plot. With larger particles, V_L has a large influence on δ_D, and the relationship given by the solid line in Figure 6 significantly overestimates the static hold up.

Dynamic hold up at zero gas velocity is plotted against the particle Reynolds number based on liquid properties. The data for the 3.6 mm particles agree closely with data presented by Wammes et al. (1991), whose data were collected for a particle size of 380.5 mm. The measurements for the smaller particles are about twice the dynamic hold up predicted by Wammes et al. (1991). This suggests that the correlation against particle Reynolds number is inadequate. Dynamic hold up was also measured as a function of gas velocity. For the larger particles, the influence of the gas was negligible, but with the 0.4 mm particles, an increase of about 20% is dynamic hold up with gas velocity was observed. An explanation would be that with no gas flow, liquid channeling occurs in the bed, the preferred path being that which offers the least resistance. Liquid flows by gravity without pressure drop. With concurrent gas flow, the gas competes with the liquid for the preferred paths, causing redistribution of the liquid through less preferred paths. Pressure drop that now occurs provides the energy for this redistribution. Our data suggest that this phenomenon depends on bed porosity.

Wammes et al. (1991) also correlated their dynamic hold up data as the product of dynamic hold up and the Galileo number to the exponent 0.42. These experimental

results compared with the data of these authors in Figure 8. Now data of at zero and finite gas velocities are shown. Introduction of the Galileo number has just a small effect on the differences between the hold up for 3.6 and 0.8 mm particles. Data for zero gas flow are also shown and the -10% effect mentioned above remains evident.

Although these experimental results show particle size and gas velocity effects that have not been reported by Wammes et al. (1991), the agreement of the data suggests that cycling does not affect the static and dynamic hold up measured in these three phase reactor.

Conclusions

1. Loading or filling follows the plug flow model.
2. Draining does not follow literature model.
3. Static and dynamic hold ups at zero gas flow agree with literature correlations for the larger particle size used.
4. Static and dynamic hold ups are influenced by gas velocity.

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References

Hosokawati, W., 1993, Loading, Draining and Steady State Operation of a Trickle Bed Reactor, M.A.Sc Thesis, Department of Chemical Engineering, University of Waterloo, Waterloo, Ontario, Canada.


Notation

- \( A \): bed cross sectional area, \( m^2 \)
- \( d_a \): particle diameter, \( m \)
- \( E0 \): Estvös Number
- \( g \): gravitational acceleration, \( m/s^2 \)
- \( h \): bed height, \( m \)
- \( h_c \): liquid height due to capillary pressure in bed, \( m \)
- \( h_m \): momentary liquid height in bed, \( m \)
- \( K \): Darcy coefficient for packing, \( m^3 \)
- \( Q_0 \): momentary liquid retained by bed, \( m^3 \)
- \( Q_l \): liquid volume fed to bed, \( m^3 \)
- \( Q_s \): volume of liquid drained from bed, \( m^3/s \)
- \( Q_n \): initial liquid volume retained by bed, \( m^3 \)
- \( Q_u \): ultimate liquid volume retained by bed after draining, \( m^3 \)
- \( t \): time, \( s \)
- \( t_f \): filling time for the bed, \( s \)
- \( V_b \): volume of bed, \( m^3 \)
- \( V_g \): volume of bed occupied by gas, \( m^3 \)
- \( V_l \): volume of bed occupied by liquid, \( m^3 \)

Greek Letters

- \( \beta_b \): fraction in the bed space filled by liquid under flow conditions
- \( \beta_a \): fraction in the bed space filled by liquid under static conditions
- \( \varepsilon \): overall porosity of bed
- \( \mu_l \): dynamic liquid viscosity, \( kg/m/s \)
- \( \rho_l \): liquid density, \( kg/m^3 \)
Figure 2. Filling time at various superficial liquid velocities

Figure 3. Filling volume vs. time $d_p = 3.6$ mm and $V_L = 22.8$

Figure 4. Filling time at various liquid velocities and bed water content for $d_p = 3.6$ mm. Upper two lines are calculated from Equation (1). $N$ is the internal between the interruption of continuous liquid flow through the column and the beginning of the filling experiment.
Figure 6. Darcy coefficient $k$ and the height of the undrained bed as a function of the superficial gas velocity for the 0.5- and 3.6-mm carbon particles.

Figure 7. Static liquid hold-up as a function of the Eötvös number.
RAGAM PATAH SPELIMEN TARIK BERTAKIK
BUSUR LINGKARAN PADA BAHAN CFRP SERAT SERAH
Pengaruh rasio keketukan (kedalaman takik)
Hera Sari\textsuperscript{a} and B. Rochardjo\textsuperscript{a}

\textbf{ABSTRACT}

Tensile tests were carried out on unidirectional CFRP to observe the
effects of specimen geometry on fracture mode of the circular necked
specimen. Necked-on-thickness specimens having various thicknesses of the
necked region with constant radius of curvature were tested. The results
show that, as the thickness ratio between the necked and un-necked region
increases a transition in fracture mode from tensile failure at the necked
region to transverse inter-laminar shear failure spreading to the outside of the
necked region before final fracture was observed. This transition is attributed to the
change in the relation between shear fracture load and tensile fracture load
with changing thickness ratio.

1. PENGANTAR

Serat karbon adalah serat yang mempunyai kekuatan taring tinggi, dengan bobot
yang ringan. Dengan kata lain kekuatan spesifiknya, yaitu kekuatan taring dibagi berat
jusiuknya tinggi yaitu bisa sekitar 2800 m. Bandingkan dengan bahan konvensional,
meskipun baja padaan, yang hanya mempunyai kekuatan taring spesifik sekitar 500 m.
Karena itu bahan komposit yang mengandung serat jinis ini termasuk kedalam bahan
yang disebut bahan komposit lanjut (advanced composite). Tetapi kekuatan taring yang
tinggi ini membutuhkan kkesulitan pada pengujian bahan komposit serat searah, karena
sifatnya yang sangat anisotropic, dimana kekuatan pada arah sejajar serat sangat tinggi
sedangkan kekuatan pada arah tegak lurus serat sangat rendah. Hal ini menyebabkan
ujiannya pada ujiensi taring dengan ragam dan tempat taring yang tidak diinginkan. Bberapa
peng-
ujian dengan spektrum plat datar seperti dilakukan beberapa peneliti (misalnya
Madhukar, 1991) memberikan hasil bahwa kebanyakan spekimen patah pada daerah tab
di bawah pecahan. Kondisi ini menunjukkan adanya pengaruh proses pecahannya
pada spekimen. Berdasarkan analisis dengan metoda eleven hingga, Uemura (Uemura,
1970) menunjukkan bahwa pada daerah di sekitar pecahan timbul konsentrasi tegangan
yang besar.

Jadi, persoalannya adalah menerapkan bentuk spekimen sedemikian sehingga bisa
mengurangi sejauh mungkin tegangan-tegangan yang tidak dikehendaki, sehingga
spekimen patah di tempat yang diinginkan, yaitu pada daerah uji. Karena itu beberapa
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