The Behavior of Counter-current Packed Bed in the Proximity of the Flooding Point Under Periodic Variations of Inlet Velocities

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Original scientific paper
Received: November 11, 2005
Accepted: February 1, 2006

An experimental study has been carried out of the two phase counter-current gas-liquid flow in a packed bed column operated in the proximity of the flooding point under periodic variations of inlet velocity of gas or liquid. Additional experiments have been focused on evaluating axial dispersion characteristics in the proximity of the flooding line in both liquid and gas phase using inert tracers.

The transient flow experiments have revealed hysteretic behavior of liquid holdup and gas pressure in the bed. The tracer RTD experiments have shown that no deterioration of axial dispersion in both gas and liquid place takes place unless the flooding phenomenon has already prevailed. In fact, axial dispersion in the gas phase lessens with increasing gas velocity and so does axial dispersion in liquid phase at higher liquid loads.

Key words:
Packed bed, counter-current flow, flooding point, axial dispersion, oscillating flow, pressure drop, liquid holdup

Introduction

Counter-current packed bed columns have been employed in a variety of technological operations ranging from chemical industry and metallurgy to environmental protection and waste water treatment.

Operations in separation columns, etc., must carefully avoid the regime known as flooding, that poses serious technological problems. However, it has been known that gas/liquid mass transfer coefficients, representing often key system parameters, dramatically improve as the column regime moves into the region of strong gas-liquid interactions above the loading point and into the neighborhood of the flooding point.

Our research work has been focused for several years on counter-current columns operated in the proximity of the flooding line and under transient flow conditions in particular.[1–11] Our team has observed for the first time the phenomenon of pressure and liquid holdup overshoots[1,2] following a sudden change of gas inlet velocity[3] and liquid inlet velocity.[4] The conditions for the appearance of overshoots have been theoretically analyzed[5] and mathematically defined.[6] Our recent analysis[7] of countercurrent beds has proven that periodic oscillations of either gas or liquid inlet velocity may lead to unstable hydrodynamic conditions.

The aim of this study is to explore experimentally the hydrodynamic behavior of the column exposed to periodic oscillations of inlet fluid phase velocities that bring the regime close to the flooding point because pulsed operations receive ever increasing interest in the literature.[12–14] Additional experiments explore the degree of axial mixing in the liquid and gas phases as the system approaches the flooding line. The presented results should contribute to the theoretical understanding of the counter-current flow system in the high-flow interaction region and possibly open its potential benefits for use in industrial operations.

Experimental

Two types of experiments have been chosen to achieve the above outlined goals. In one type of the experiments the column was exposed to forced periodic oscillations of either gas or liquid inlet velocity while we measured the transients of the pressure profile and of the liquid holdup in the bed. In another type of the experiment we measured the residence time distribution (RTD) responses of the column to injecting tracers in either the liquid or gas inlet stream.

The employed[8] experimental counter-current packed bed column is 0.2 m in diameter packed to 1 m height by 10 mm glass spheres. The column is suspended on a tensometric balance (Eilersen Elec-

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tric A.S.) that allows its continuous weighing under operating conditions.

Water as the liquid phase is circulated in closed loop by a centrifugal pump via electronic flow meter, liquid distributor, packed bed and water siphon back to a temperature-controlled tank. Air from 0.5 MPa pressurized distribution network is used as the gas phase. The air is brought via an oil separator, a pressure reducing valve, regulating valve and electronic flow meters into the water siphon at the bottom of the column. After passing the column the air is discharged into the atmosphere.

Thermometers are installed in the flow lines of both phases. Electromagnetic valves in parallel supply lines of liquid as well as gas phase enable forced step or periodic changes of input flow rates. Pressures drops and pressure profiles along the column are measured directly by piezoelectric pressure sensing probes located in ports along the column height 0.2 m apart. Data are collected by a PC equipped with the Labview data logging system that also provides programmed control of the experiment and actuation of the solenoid valves.

Additional gear is installed inside the cylinder for residence time distribution (RTD) measurement in the gas and liquid phase.

Pressure drop and holdup transient measurement

In the pressure drop and holdup transient measurement the system was exposed to forced periodic oscillations of liquid or gas inlet velocity. These forced changes of inlet velocity were realized using the electromagnetic valves in the parallel supply lines described above. Instantaneous values of the liquid holdup were computed on-line from the data supplied by the tensometric balance, the overall pressure drop transducer, from the dry-column weight and the column diameter. The frequency of liquid holdup and pressure data sampling was 100 Hz. All data were recorded onto hard disk of the PC and the frequency of the forced oscillation was set in the Labview system program written for this purpose.

Residence time in liquid phase measurement

The RTD in liquid experiments consisted of injecting a small amount of concentrated solution of NaCl into the inlet liquid stream. The tracer was injected at time equal 10 s after the onset of the experiment and the duration of the injection was 2 s. The outlet concentration of NaCl was monitored by measuring electric conductivity of liquid in a cell of special construction.

The electric conductivity cell was formed at the top by the metal grid supporting the packing in the column and a brass wire mesh with 8 mm gauge square openings located 5 mm below the supporting grid. The supporting grid and the brass mesh served as two electrodes in the electric conductivity measuring system circuit. Just as a safety precaution between shortcutting the two electrodes there was an expanded plastic sheet placed between the two electrodes.

Residence time in gas phase measurement

The RTD in gas experiments consisted of injecting a small amount of Helium into the gas liquid stream. The concentration of He in the outlet gas stream was monitored by measuring the thermal conductivity of the gas. The data were logged on the hard disk of the PC.

Pressure and liquid holdup at flooding-results

The hydrodynamics of the countercurrent packed bed systems exhibits two important states called loading and flooding points. Below the loading point there is almost no interaction between the two flowing phases and liquid holdup is primarily a function of the liquid flow rate. Past the loading point, the interactions between the flowing phases become appreciable and, at the flooding point, the liquid is being entrained by the gas while a gas-liquid mixture appears above the top of the packing.

The Figure 1 plots the liquid flow velocity (full squares and LHS vertical axis) when, for the given gas flow velocity, flooding of the column begins to

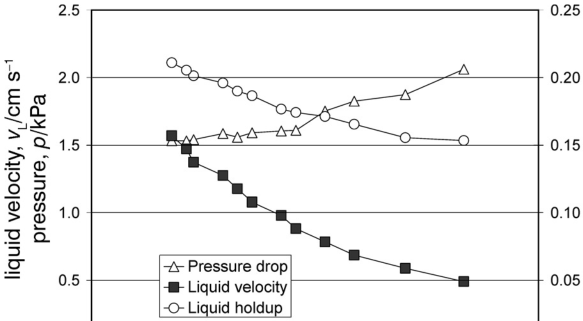


Fig. 1 – *Liquid velocity, pressure drop and liquid holdup at flooding as a function of gas velocity*

appear in a packing of 10 mm glass spheres. The flooding was indicated by non-zero reading of the pressure transducer, located level with the top of the packing, due to the presence of gas-liquid mixture there. Expectedly, the liquid velocity that leads to the flooding for a given gas velocity decreases with increasing gas velocity.

Apart from the flooding line Fig. 1 further shows corresponding values of the liquid holdup and pressure drop at flooding. It is seen that at the flooding point pressure drop per unit packing height (empty triangles and LHS vertical axis) slightly increases with increasing gas velocity. At the same time liquid holdup at flooding (empty circles and RHS vertical axis) slightly decreases with the gas velocity.

For subsequent measurements of the transients of pressures profiles and liquid holdup under forced periodic oscillations of inlet velocity we always chose combinations of gas-liquid velocity that would bring the system close to the flooding line shown in Fig. 1 (full squares).

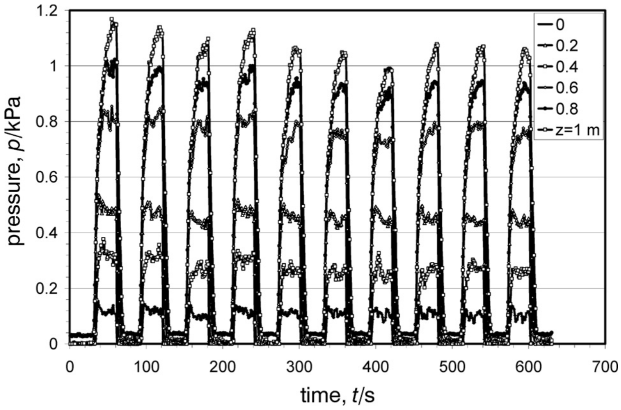
Transient pressure and liquid holdup development-results

Fig. 2-5 show the experimental time development of gas pressure and liquid holdup as functions of time for the case where the gas velocity was kept constant and the liquid velocity was changed periodically in rectangular pulses. The experimental points plotted are those obtained by smoothing the data collected at 100 Hz to 1Hz.

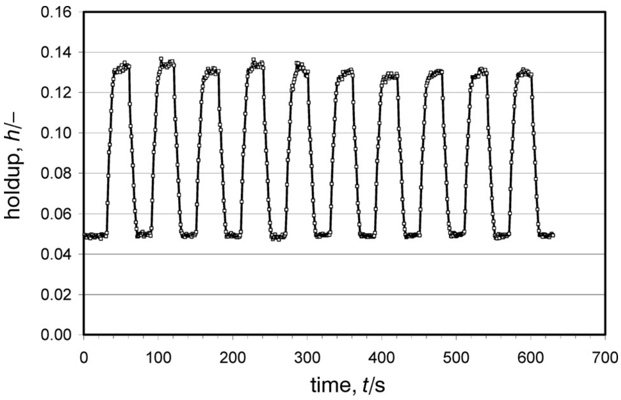
In Fig. 2-5 the upper and lower limit of liquid rate pulse was each maintained for 30 s making a total length of the time period of the pulse equal 60 s. The lower liquid rate in these four cases was set to 0.0039 m s^{-1} (see the captions) while the upper limit was selected so as to bring the system, for the given gas rate, close to the flooding line seen in Fig. 1.

Individual lines in Fig. 2a, 3a, 4a and 5a plot the profiles of pressure at various levels of the coordinate z measured from the top of the packed bed. The level designated as z=1 m thus represents the overall pressure drop. Fig. 2b, 3b, 4b and 5b plot the mean holdup of liquid in the column.

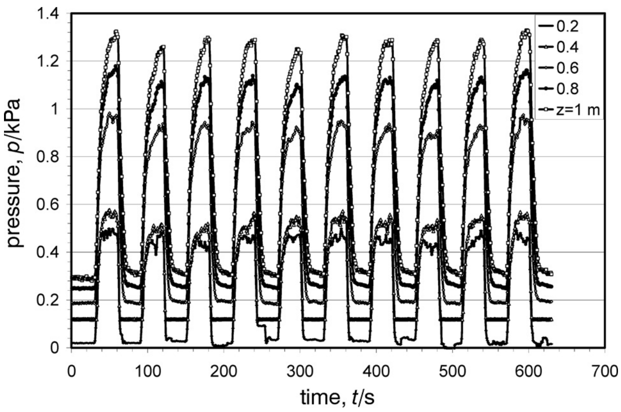
In all four Fig. 2-5 one can observe the same phenomenon: Namely that the rate of increase of pressure and liquid holdup (following the stepwise increase of liquid inlet velocity) is less than the rate of decrease of the pressure and liquid holdup (following the stepwise decrease of liquid inlet velocity).



F i g . 2 a – *Transient response of gas pressure at various levels of z to a step change in liquid velocity from 0.0039 to 0.0123 m s^{-1} at gas velocity 0.054 m s^{-1} at various locations along the bed shown in legend*

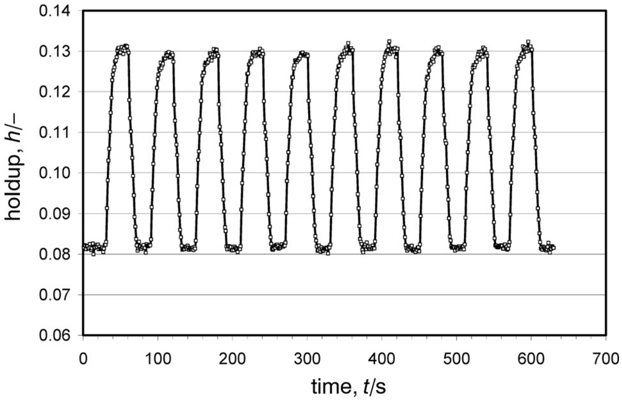


F i g . 2 b – *Transient response of liquid holdup to a step change in liquid velocity from 0.0039 to 0.0123 m s^{-1} at gas velocity 0.054 m s^{-1}*

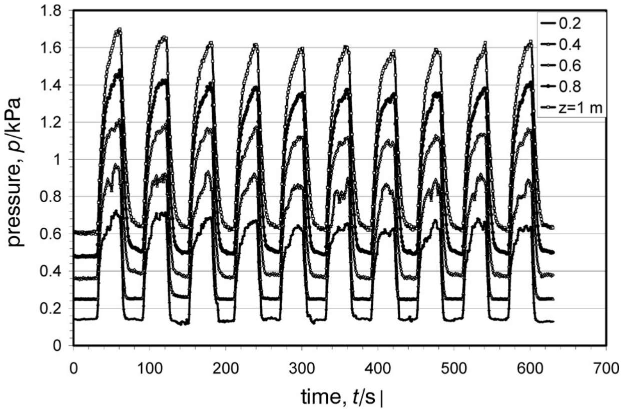


F i g . 3 a – *Transient response of gas pressure at various levels of z to a step change in liquid velocity from 0.004 to 0.0082 m s^{-1} at gas velocity 0.123 m s^{-1} at various locations along the bed shown in legend*

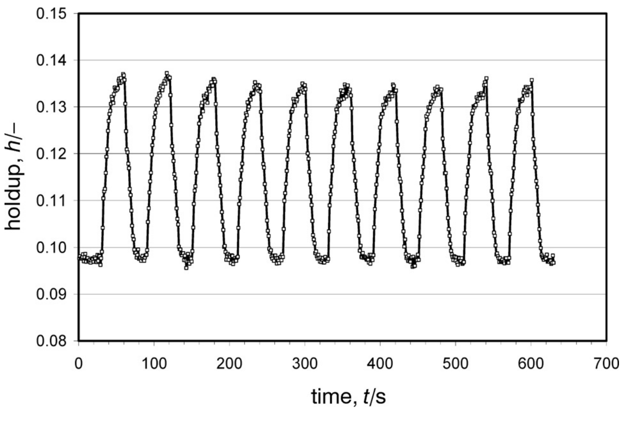
One can further observe that as we gradually move from Fig. 2 to Fig. 5 the gas-to-upper-liquid-velocity ratio increases. In other words, Fig. 2 represents the regime at low gas but high liquid velocity, in contrast to Fig. 5 where the situation is



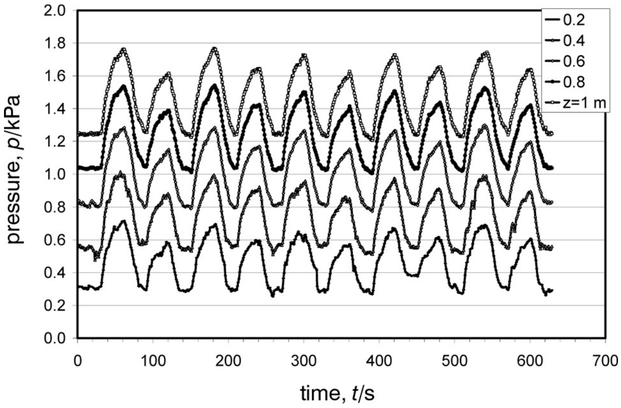
F i g . 3 b – *Transient response of liquid holdup to a step change in liquid velocity from 0.004 to 0.0082 m s^{-1} at gas velocity 0.123 m s^{-1}*



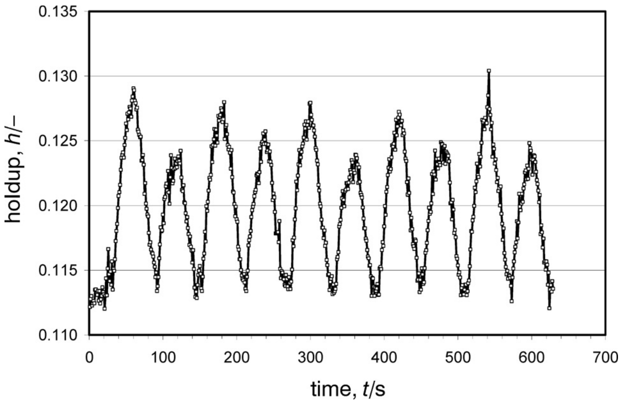
F i g . 4 a – *Transient response of gas pressure at various levels of z to a step change in liquid velocity from 0.004 to 0.0067 m s^{-1} at gas velocity 0.160 m s^{-1} at various locations along the bed shown in legend*



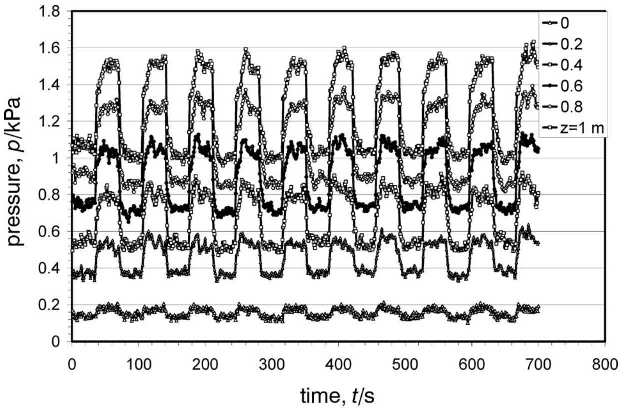
F i g . 4 b – *Transient response of liquid holdup to a step change in liquid velocity from 0.004 to 0.0067 m s^{-1} at gas velocity 0.16 m s^{-1}*



F i g . 5 a – *Transient response of gas pressure at various levels of z to a step change in liquid velocity from 0.004 to 0.0048 m s^{-1} at gas velocity 0.220 m s^{-1} at various locations along the bed shown in legend*



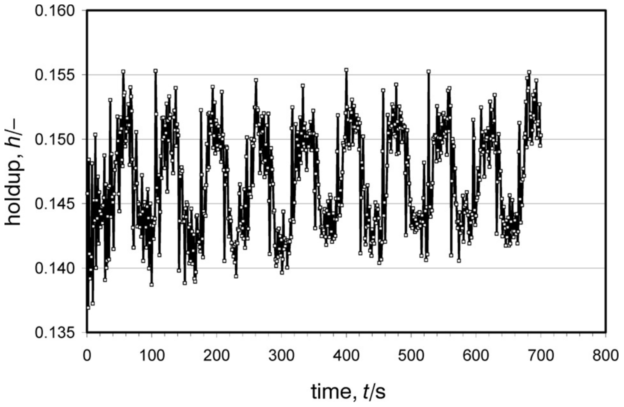
F i g . 5 b – *Transient response of liquid holdup to a step change in liquid velocity from 0.004 to 0.0048 m s^{-1} at gas velocity 0.220 m s^{-1}*



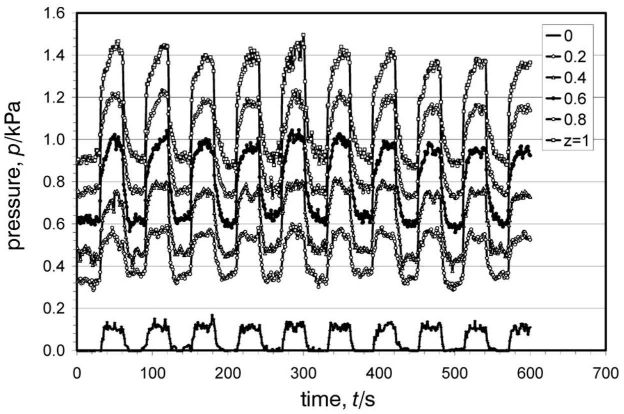
F i g . 6 a – *Transient response of gas pressure at various levels of z to a step change in gas velocity from 0.038 to 0.0724 m s^{-1} at liquid velocity 0.013 m s^{-1} at various locations along the bed shown in legend*

opposite. Keeping this in mind we can see that the hysteresis effect of the pressure and liquid holdup of transient curves becomes more manifest at higher gas rates.

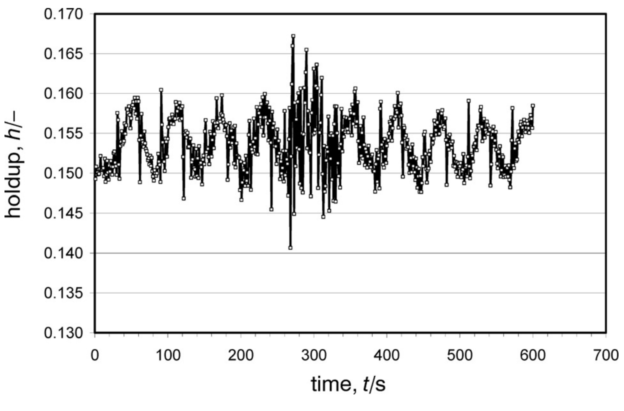
Fig. 6 and 7 show again the pressures and liquid holdup as functions of time, now, however, for the case that that the liquid rate was kept constant and the gas rate was changed periodically in rectangular pulses. In these figures the upper and lower limit of gas was each again maintained for 30 sec making a total length of the time period equal 60 sec. The upper limit of gas velocity was again



F i g . 6 b – *Transient response of liquid holdup to a step change in gas velocity from 0.038 to 0.072 m s^{-1} at liquid velocity 0.0132 m s^{-1}*



F i g . 7 a – *Transient response of gas pressure at various levels of z to a step change in gas velocity from 0.108 to 0.143 m s^{-1} at liquid velocity 0.0091 m s^{-1} at various locations along the bed shown in legend*



F i g . 7 b – *Transient response of liquid holdup to a step change in gas velocity from 0.108 to 0.0143 m s^{-1} at liquid velocity 0.0091 m s^{-1}*

selected so as to bring the system, for the given liquid velocity, close to the flooding line shown in Fig. 1.

Individual lines in Fig. 6a and 7a plot the profiles of pressure at various levels of z measured again from the top of the packed bed. Fig. 6b and 7b plot the mean holdup of liquid in the column.

The same hysteretic phenomenon as that observed in the experiments with variable liquid velocity can now be also observed at least on the transient curves of gas pressure in Fig. 6a and 7a: The buildup of pressure within the bed in the period of increased gas velocity is seen to be distinctly slower than its decay after the inlet gas velocity was decreased. The same phenomenon, however, is difficult to discern on the transient curves of the liquid holdup. The reason is that the imposed changes of gas velocity caused relatively minor changes of liquid holdup (see the scale on the abscissa). Nevertheless, similarly as before the hysteresis appears to be stronger in Fig. 7a than 6b because the (upper) gas velocity is higher for the case in Fig. 7a.

RTD curves in liquid phase-results

The RTD curves were measured on the same column with 1 m high bed of 10 mm glass spheres. The measurements were carried out in several series. Each series had a fixed liquid rate while the gas rate was gradually increased from zero up to the proximity of the flooding point while making measurements of the column response for each pair of the gas and liquid flow velocity.

The experimental responses measured by the electric conductivity probe were sampled at 100 Hz. These raw values were then smoothed to 1 Hz to facilitate processing and plotting of the results. The obtained data were further scaled so as to make the total input of the NaCl tracer into the bed equal unity.

The theoretical response curves were computed from the solution of the following equation:

$$\frac{\partial c}{\partial t} + v\frac{\partial c}{\partial z} = D\frac{\partial^2 c}{\partial z^2}$$

Its solution in terms of the dimensionless concentration response to a Dirac impulse at time equal zero at the inlet for an L long bed is given[9] by:

$$c = \frac{1}{2\sqrt{\dfrac{\pi}{Pe}\dfrac{t}{t_0}}}\exp\left[\frac{Pe_{\mathrm{x}}\left(1-\dfrac{t}{t_0}\right)^2}{4\dfrac{t}{t_0}}\right]$$

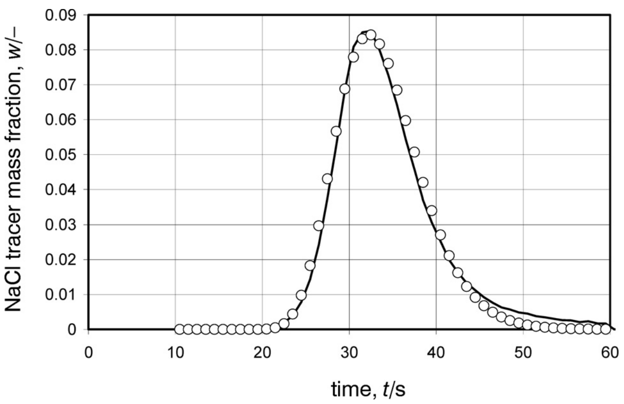
where

$$Pe_{\mathrm{x}} = \frac{vL}{D}$$

the time t is measured from the instant of tracer injection and v designates the linear velocity of the flowing phase in which the impulse was implemented (v_G, v_L).

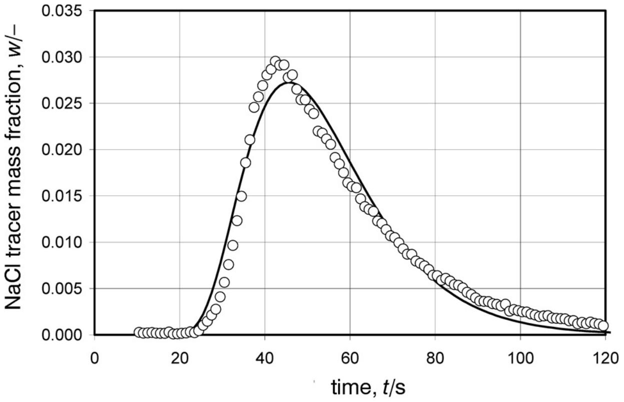
The theoretical profiles computed from the above model were fitted to the experimental profiles by optimizing the parameters t_0 and Pe_x using the Microsoft Excel software. It is noted that t_0 represents the mean residence time in the column measured from the instant of tracer injection (10 s).

Fig 8 shows the results of optimization for an impulse in the liquid phase at liquid velocity 0.0039 m s^{-1} and gas rate 0.039 m s^{-1}. The response is seen to form a very "narrow" peak indicating relatively weak axial dispersion in the liquid phase.



F i g . 8 – *Predicted (solid line) and experimental (circles) results of the response of the column to an impulse in concentration in liquid at t = 10 s for V_L = 0.0039 m s^{-1} and V_G = 0.039 m s^{-1}*

In contrast, Fig. 9 showing the results of the fitting the theoretical curve at the same liquid velocity, but substantially higher gas velocity (V_L = 0.0039 m/s and V_G = 0.314 m s^{-1}), indicates distinctly higher axial dispersion as it becomes manifest from the lower and wider peak and the lack of its symmetry. Distinctly worsened accuracy of the fit by the model testifies to the presence of other mechanisms than pure axial dispersion. All that is apparent due to the elevated gas velocity that brought the system close to the flooding point.

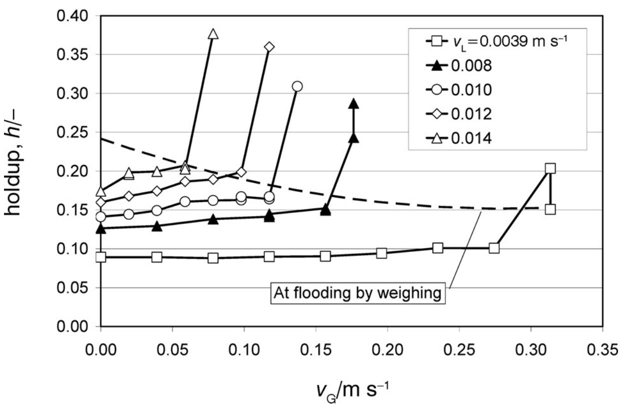


F i g . 9 – *Predicted (solid line) and experimental (circles) results of the response of the column to an impulse in concentration in liquid at t = 10 s for V_L = 0.0039 m s^{-1} and V_G = 0.314 m s^{-1}*

The set of the optimized results provided us with the "optimum" values of the mean residence time of liquid in the column, t_0, and the Peclet number Pe_x. The mean residence times were further processed to yield liquid holdup using the following formula:

$$h = \frac{V_\mathrm{L}}{v_\mathrm{L}} = \frac{V_\mathrm{L} t_0}{L}$$

The evaluated liquid holdups are plotted in Fig 10 as a function of gas velocity with liquid velocity as a parameter. The figure exhibits typical form when liquid holdup, for given liquid velocity, is initially at low gas velocity only a very weak function of the gas velocity. However, liquid holdup tends to change dramatically with the gas rate as the regime approaches the flooding line. Also typically, the region of weak gas velocity effect diminishes with increasing liquid rate.



F i g . 1 0 – *Liquid holdup evaluated from the RTD measurement in liquid as a function of gas velocity with liquid velocity as a parameter; broken line shows the course of holdup at flooding measured by weighing the column*

The broken line in Fig. 10 shows the course of liquid holdup at flooding measured in our experiments (see Fig. 1) by direct weighing of the column under operating conditions, while the flooding was detected by non-zero gas pressure at the top of the packed section. The figure shows a fairly good agreement of the indication of the flooding by two different experimental techniques.

The "optimum" values of Pe_x served to evaluate the Peclet number in the liquid phase, Pe_L, defined on the basis of superficial velocity of liquid as:

$$Pe_\mathrm{L} = \frac{V_\mathrm{L} d_\mathrm{P}}{D_\mathrm{L}} = Pe_\mathrm{x} \frac{V_\mathrm{L}}{v_\mathrm{L}} = Pe_\mathrm{x} V_\mathrm{L} \frac{t_0}{L}.$$

The obtained values of Pe_L are shown in Fig. 11 as a function of gas velocity with the liquid velocity

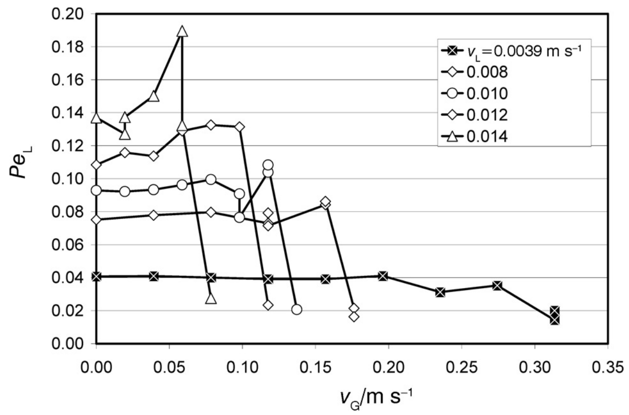


Fig. 11 – *Peclet number Pe_L in liquid as a function of gas velocity with the liquid velocity as a parameter*

as a parameter. The figure shows that the Peclet number in the liquid phase strongly varies with the liquid velocity, while the gas velocity, at least at low and modest liquid rates, has only weak effect until the flooding phenomenon has taken place.

It appears from the figure that Pe_L, for given gas velocity, increases almost in direct proportion to liquid superficial velocity. This is confirmed in Figure 12 plotting D_L/d_P equaling V_L/Pe_L which remains virtually constant until the flooding prevails.

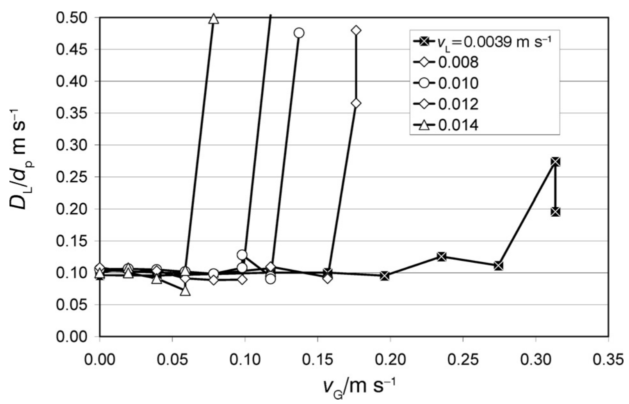


Fig. 12 – *D_L/d_P m s^{-1} as a function of gas velocity with the liquid velocity as a parameter*

In general, the above results show that axial mixing remains low and unchanged at low and intermediate gas rates; increasing gas rate at high liquid rates actually reduces axial mixing. Drastic increase of axial mixing (decrease of Pe_L) takes place only when flooding prevails.

RTD curves in gas phase-results

The breakthrough curves were measured again on the same column with 1 m high bed of 10 mm glass spheres.

The obtained experimental data were fitted by the above theoretical curve except that t_0 is related now to the holdup of gas and the Peclet number in the gas phase, based on the superficial velocity of the gas, is defined as:

$$Pe_G = \frac{V_G d_P}{D}.$$

Typical fit of the theoretical and experimental responses is shown in Fig 13 where the experimental results (circles) were obtained by smoothing the data measured at 100 Hz to 1 Hz. Furthermore, the thermal conductivity data were scaled so as to make the total input of the He tracer into the bed equal unity.

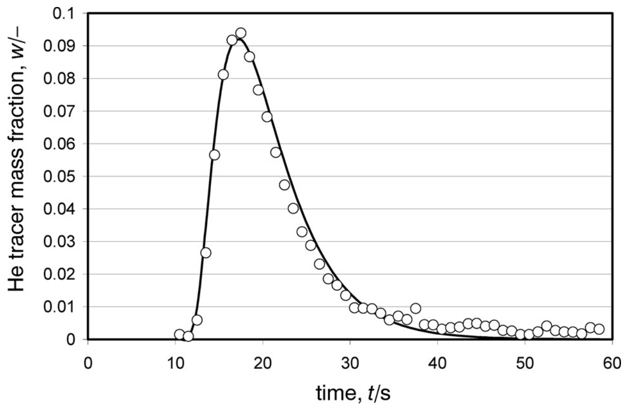


Fig. 13 – *Predicted (solid line) and experimental (circles) results of the response of the column to an impulse in concentration in gas at t = 10 s for V_L = 0.0137 m s^{-1} and V_G = 0.0392 m s^{-1}*

It turned out that the evaluation of gas holdup from the fitted response curves is very unreliable, because the residence time of gas in the bed is very short due to the gas velocities being much higher compared to those of liquid. This is evident from Fig. 13 where the peak of the dimensionless He concentration begins to rise very shortly after the time of injection (t = 10 s).

For this reason the Peclet numbers in the gas phase, Pe_G, were evaluated from the form Pe_x using gas holdup computed from the known void fraction of the bed and the liquid holdup, evaluated from the response curves in liquid, i.e. from:

$$Pe_G = \frac{V_G d_P}{D_G} = Pe_x \frac{V_G}{v_G} = Pe_x h_G = Pe_x(\varepsilon - h)$$

The results are shown in Fig. 14 plotting Pe_G as a function of gas velocity with liquid velocity as a parameter.

A comparison of the Peclet numbers in gas and liquid phase shows significant differences: In the

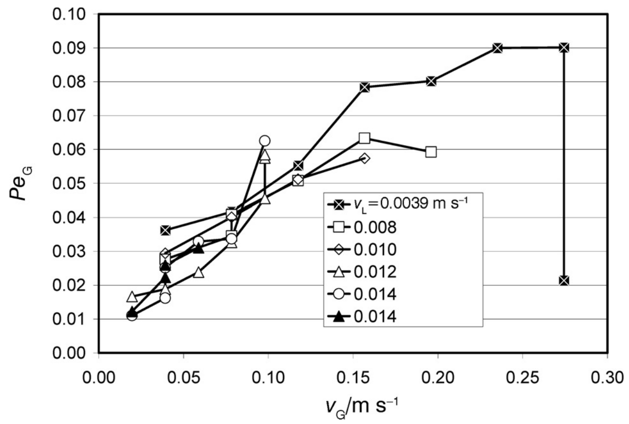


Fig. 14 – *Peclet number Pe_G as a function of gas velocity with the liquid velocity as a parameter*

liquid phase the Peclet number changed little with gas velocity until the onset of flooding. In contrast, the gas phase Peclet number appears to increase approximately linearly with the gas velocity, indicating a decrease of intensity of axial mixing. A test of this linear relationship is shown in Fig. 15 plotting D_G/d_P equaling V_G/Pe_G versus gas velocity with liquid velocity as a parameter. From that figure one can see that the lines for different liquid rates are not as closely packed together at it was in the case of liquid dispersion and D_G/d_P increases with gas velocity.

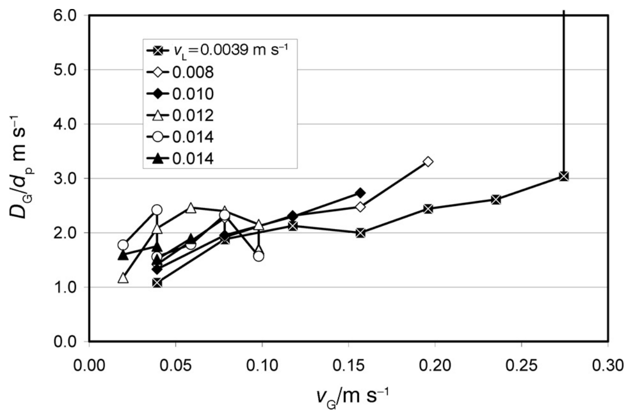


Fig. 15 – *D_G/d_P m s^{-1} as a function of gas velocity with the liquid velocity as a parameter*

Additional inspection of Fig. 14 and comparison with the flooding line in Fig. 1 indicates that axial mixing actually does not become a problem until the real onset of flooding.

Conclusions

The experiments with forced periodic pulsations of inlet liquid or gas velocities in counter-current packed bed have revealed that the rate of build up of liquid holdup as well as of pressure within the column is slower than the rate of their decay at least in the range above the loading line.

This hysteretic behavior has a potential for practical utilization of the periodic operation of the column in those situations where lowered liquid holdup and pressure represent an advantage. Even more important aspect may be that the periodic operation close to the flooding line offers high interfacial mass and heat transfer rates while simultaneously avoiding actually flooding the column.

The experiments with breakthrough curves measured in the proximity of the flooding line have revealed that axial dispersion does not pose a problem unless the flooding actually has taken place. In fact, axial dispersion in the gas phase actually weakens as we move toward the flooding line by increasing the gas velocity. Weakening of axial dispersion was observed also in the liquid phase at high liquid rates as we moved toward the flooding line by increasing the gas velocity.

These findings suggest that operation of the counter-current columns beyond the loading line and close to the flooding line can provide significant benefits thanks to increased mass and/or heat transfer coefficients, while low axial dispersion characteristics further contribute to high separation efficiency of the column. All these benefits are fully preserved all the way to the flooding line as long as the flooding does not take place.

ACKNOWLEDGEMENT

The authors gratefully acknowledge financial support of the project by the Grant Agency of the Czech Republic under the grant number 104/03/1558.

Symbols

D_G, D_L – coefficient of axial dispersion in gas and liquid phase, m^2 s^{-1}

d_P – particle diameter, m

h ,h_G – holdup of liquid and gas, –

L – length of the bed, m

Pe_G, Pe_L – Peclet number for gas and liquid, –

V_G, V_L – superficial velocity of gas and liquid, m s^{-1}

v_G, v_L – linear velocity of gas and liquid in the bed, m s^{-1}

t – time coordinate, s

t_0 – mean residence time of gas or liquid in the column, s

z – axial coordinate, m

ε – void fraction of bed, –

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