Exchange between the stagnant and flowing zone in gas-flowing solids-fixed bed contactors

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Abstract: In countercurrent gas – flowing solids – fixed bed contactors, a fraction of the flowing solids is in motion (dynamic holdup), while the other fraction is resting on the fixed bed elements. In this study it was experimentally proved that the stagnant zone should not be considered as a dead part of the column, but that there is a dynamic exchange between these two portions of flowing solids particles. Combining a mathematical model with tracer experiments, the rate of exchange was determined and it was shown that only a small part (*ca*. 20 %) of the stagnant region should be considered as a dead one.

Keywords: gas–flowing solids–fixed bed contactors, gas–solids trickle flow, multiphase contactors, tracers.

INTRODUCTION

Contercurrent flow of gas and fine solids through a fixed bed of solids seems to be a promising basis for novel types of heat and mass transfer operations, as well as for chemical reactors with separation *in situ*. The idea[1] was patented in 1948 and the first recorded industrial realization[2] occurred in 1965 for heat recuperation. In gas – flowing solids – fixed bed contacotrs, gas is introduced at the bottom and fine solids particles (flowing solids) at the top of the column, and they flow countercurrently inside a packed bed of solids (second solids phase). The packing redistributes and carries the flowing solids, facilitating mass transfer, while the axial mixing of the both phases is reduced. Numerous applications were planned and investigated, both in separation processes, based on the different adsorption properties of the gas componenets, and in chemical reactors, where the reaction product could be removed from the reaction zone with the adsorbing flowing solids.

Gas – flowing solids – fixed bed columns could be used for contacting of two or three active phases. In heat exchange operations, drying, adsorption and other

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separation processes, the two phases (gas and flowing solids) are active. In these cases the design of packing elements depends on fluid dynamic demands only, because the packing serves only to redistribute both of flowing phases and to carry a part of flowing solids. In catalytic reactors with separation *in situ*, the packing elements (third active phase) are the catalytic pellets. In previous studies[3] gas – flowing solids – fixed bed columns were compared with other gas – solid countercurrent equipment and it was concluded that gas – flowing solids sistem have advantages over spray columns, moving beds and multistage fluidized beds.

The fluid dynamics of gas – flowing solids – fixed bed systems has received considerable attention over the years.[3–17] It was concluded that the flow of particles through the packing, countercurrently to the gas flow, exhibits similar behavior to gas – liquid trickle flow in packed beds. Three flow regimes were observed: preloading, loading and flooding. In the loading zone the interaction between the gas and the flowing solids is much more expressed than in the preloading zone. When the gas flow rate reaches the terminal velocity of the flowing particles, the solids phase has a tendency to accumulate in the upper part of the packing and these conditions are called flooding. The flooding point is characterized by a sudden increase in both pressure drop and solids holdup and the behavior of the system is unstable.

DYNAMIC AND STATIC HOLDUP

Flowing solids holdup is one of the basic design parameters and represents the fraction of the column volume occupied by flowing solids. Similar to gas – liquid systems, it can be split into dynamic and static holdup. Static holdup represents the fraction of the flowing solids resting on the packing elements, while dynamic holdup represents the fraction of flowing particles that are suspended in the voids between the packing elements. After shutting simultaneously the solids and gas inlets, dynamic holdup is determined from the quantity of particles that will drain out, while the mass of particles that remaining in the bed gives the static holdup.

Dynamic holdup is usually assumed to be the operating holdup. However, visual observation leads to the conclusion that there exists an interchange between these two portions of the solids particles. The objective of this study was to investigate if such exchange between the stagnant and flowing zones exists, and what is the rate of such an exchange.

EXPERIMENTAL

A tracer technique with a step signal at the inlet of the flowing solids was used for the experimental investigations. Polyamide powders in two colors, grey and red, were used as the flowing solids phase. All the properties (density, mean particle diameter, particle diameter distribution and sphericity) of these two powders were the same, except color. The characteristics are presented in Table I.

A sketch of the apparatus is presented in Fig. 1. A 2.47 cm inner diameter glass column was used. It was packed with 5 mm glass spheres, supported by a grid.

Two flowing solids dosage systems, for each powder separately, were used. A constant powder flow during the experiments was obtained by the use of two funnels, one above the other, while the
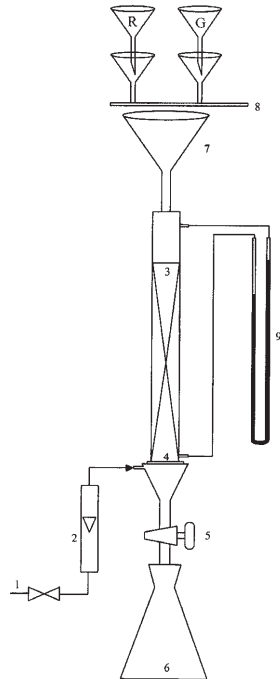
Fig. 1. Apparatus. (1-gas inlet, 2-rotameter, 3-column with fixed bed, 4-supporting grid, 5-exit valve, 6-container, 7,8-flowing solids inlet with dosage system, 9-manometer).

flow rate was adjusted by using plates with different orifice diameters, previously calibrated. By shifting the plate, the orifice changes its position closing one dosage system, and opening the other one, which corresponded to the step input. In these experiments solids flow rates was maintained at 0.17 kg/m^{2}s.

TABLE I. The characteristics of the flowing solid phase

Particle size distribution of the flowing solid phase		
Diameter/μm	Weight fraction/%	Cummulative fraction/%
<1.4	0.7	0.7
1.4–1.6	0.4	1.1
1.6–20	0.3	1.4
20–22	0.5	1.9
22–25	1.3	3.2
25–28	1.0	4.2
28–32	0.6	4.8
32–36	0.0	4.8
36–40	0.0	4.8
40–45	0.4	5.2
45–50	1.3	6.5
50–56	2.6	9.1
56–63	4.4	13.5

TABLE I. Continued

Particle size distribution of the flowing solid phase		
Diameter/μm	Weight fraction/%	Cummulative fraction/%
63–75	9.1	22.6
75–90	12.8	35.4
90–106	15.2	50.6
106–125	18.2	68.8
125–150	18.5	87.3
150–175	9.6	96.9
175–200	3.1	100.0

Mean diameter 105.27 μm; Skeletal density 1040 kg/m^3

The gas flow rate was adjusted with precision valves and determined by using a calibrated rotameter. In these experiments, the gas superficial velocity was kept at 0.0827 m/s.
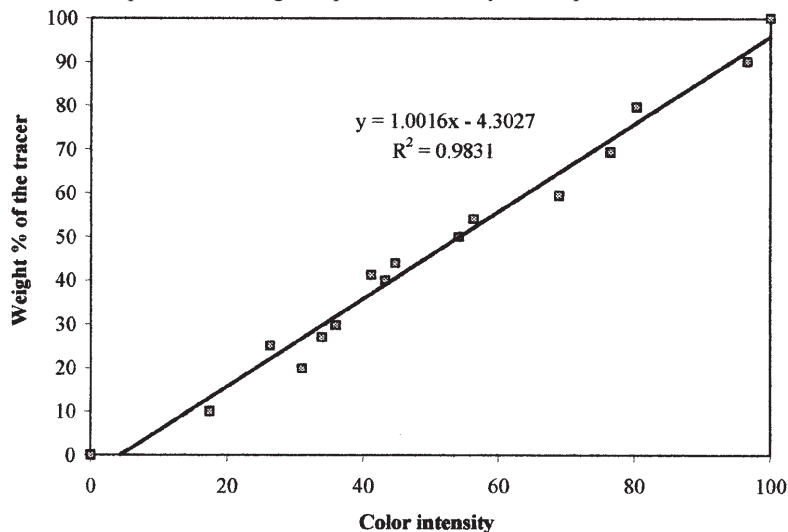

$$y = 1.0016x - 4.3027$$
$$R^2 = 0.9831$$

Fig. 2. Calibration curve.

The measurement technique was as follows. After reaching steady state, the step signal was introduced at the entrance of the flowing solids. At different times after the step signal, the inlets of gas and flowing solids were simultaneously closed, and a part of flowing solids drains out. The static holdup was determined by discharging the content of the column, sieving and weighing the solids powder that had remained in the bed. The powder that remained in the bed was then well mixed, spread between two glass plates and scanned using "Hewlett Packard Co. IIcx" Scanner together with the application of "DeskScan II Hewlett Packard Co.". Software "SigmaScan/Image" was used for color intensity analysis. The fraction of the red colored powder (tracer) was obtained from a previously prepared calibration curve (Fig. 2).

RESULTS AND DISCUSSION

The fraction of the exchanged flowing particles, represented as the fraction of red powder in the stagnant zone, is presented in Fig. 3 as a function of time after the
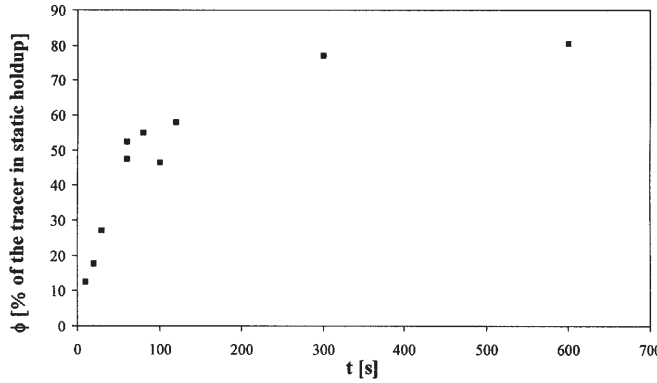
Fig. 3. Fraction of exchanged flowing solids as a function of time.

step signal. It shows that an exchange between the static and the flowing zones does exist, and that the static zone is not a dead volume. However, the curve representing the fraction of exchanged particles has an asymptote at the value of approximatelly 0.8, which means that 20 % of the stagnant volume is a real dead volume without any exchange.

The static zone without the dead part represents the active part of the static zones. Due to the exchange of flowing solids particles, the inlet of tracer particles into the active static zone is given by:

$$v_{T,\text{in}} = fV_c \tag{1}$$

where f is the volumetric rate of exhange of solids particles per unit column volume, and V_c is the column volume. It was assumed that after the step signal, all the particles entering the static zone were tracer particles. This is based on the fact that the mean residence time was much lower than the check times in our experiments, while the flow of flowing solids was close to the plug flow. Consequently, at a time after the signal, equal or greater than the mean residence time, all the particles suspended in the voids were tracer particles.

The outlet of tracer particles from the active static zone is given by:

$$v_{T,\text{out}} = fV_c \, \varphi \tag{2}$$

where φ is the volumetric fraction of tracer particles in the active part of the static zone. Eq. (2) was derived under the assumption that the active part of the static zone was well mixed, so that the fraction of tracer in the outlet fluid was the same as in the zone.

The total volume of tracer particles in the static zone is given by:

$$\varphi(\beta_{\text{st}} - \beta_{\text{dead}})V_c = \phi \, \beta_{\text{st}} V_c \tag{3}$$

where β_{st} and β_{dead} are the static holdup and dead part of the static holdup, respectively. The balance over the active part of the static zone gives:
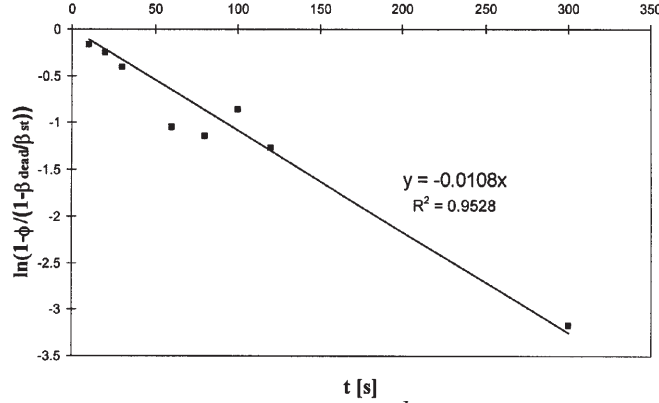
Fig. 4. Plot of equation (7) – Determination of the exchange rate.

$$(\beta_{st} - \beta_{dead})V_c \frac{d\varphi}{dt} = \nu_{T,in} - \nu_{T,out} = fV_c(1 - \varphi) \tag{4}$$

The solution of the differentail equation (4) is:

$$\ln(1 - \varphi) = -\frac{f}{\beta_{st} - \beta_{dead}}t + C \tag{5}$$

where the constant $C = 0$ for $t = 0$, $\varphi = 0$. The tracer volumetric fraction in the whole static zone, which was measured in our experiments, can be found from Eqs. (3) and (5) as:

$$\varphi = \left(1 - \frac{\beta_{dead}}{\beta_{st}}\right)\left(1 - e^{\frac{f}{\beta_{st} - \beta_{dead}}t}\right) \tag{6}$$

Equation (6) can be rearranged to give:

$$\ln\left(1 - \frac{\phi}{1 - \beta_{dead}/\beta_{st}}\right) = -\frac{f}{\beta_{st} - \beta_{dead}}t \tag{7}$$

where the rate of exchange between zones f can be found from the slope of the plot.

When the obtained results are presented in this form, Fig. 4 is obtained. From the slope, the value of the exchange rate was found to be $f = 0.274 \times 10^{-3}$ 1/s (*i.e.*, in m^3 of flowing solids per m^3 column and second).

CONCLUSIONS

An exchange between the stagnant zone and the flowing zone for flowing solids in a countercurrent gas – flowing solids – fixed bed column was experimentaly proved in this study. From the comparison of the experimental results and mathematical model, an exchange rate of 0.274×10^{-3} 1/s was found under these experimental conditions. This investigation has shown that dynamic holdup can not be

assumed to be the operating holdup.

Furthermore, the effect of the exchange between the dynamic and static portion of flowing solids is expected to influence the residence time distribution of the flowing solids.

NOMENCLATURE

f – Volumetric exchange rate of flowing solids between the flowing and stagnant zone per unit column volume, (m^3 / m^3s = 1/s),

t – Time, (s),

$v_{T,in}$ – Volumetric flowrate of particles entering the static zone, (m^3/s),

$v_{T,out}$ – Volumetric flowrate of particles exting the static zone, (m^3/s),

V_c – Column volume,

β_{dead} – Dead portion of the static volume (volume of "dead" particles per unit column volume),

β_{st} – Static flowing solids holdup

ϕ – Tracer volumetric fraction in the stagnant zone,

φ – Tracer volumetric fraction in the active part of the stagnant zone

ИЗВОД

РАЗМЕНА ЧЕСТИЦА ИЗМЕЂУ ПОКРЕТНЕ И НЕПОКРЕТНЕ ЗОНЕ У КОНТАКТОРИМА ГАС–ПОКРЕТНА ЧВРСТА ФАЗА–ПАКОВАН СЛОЈ

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Контактори гас–покретна чврста фаза–покован слој представљају нови тип уређаја за сепарационе процесе, пренос топлоте и хемијске реакторе. У овом типу контактора гас струји на горе кроз покован слој у колони, док ситне честице покретне чврсте фазе струје на доле. Покретну чврсту фазу карактеришу динамички и статички садржај, јер се део честица налази у кретању, док други део честица мирује наталожен на елементима паковања. У овом раду је предпостављено и експериментално доказано да наталожене честице на паковању не представљају неактивни (мртви) део покретне чврсте фазе, као што се до сада предпостављало, већ постоји стална размена између покретних честица у току (динамички садржај) и оних које су наталожене (статички садржај). Применом технике са обележеном супстанцом показано је да само мали део наталожених честица на пуњењу (око 20 %) не бива замењено ни после веома дугог времена и заиста представља "мртву зону". Комбиновањем изведеног математичког модела са експерименталним резултатима одређена је брзина размене покретних и непокретних честица.

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