AN EXPERIMENTAL SET-UP FOR THE IDENTIFICATION OF PARAMETERS OF THE TRICKLE FLOW IN A PACKED BED

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An experimental technique has been developed to identify parameters of the trickle flow of liquid under the counter-current flow of gas. The technique has been based on measurement of the response in the outlet stream to a periodic concentration perturbation of the inlet liquid stream. A two-chamber distributor for liquid has been developed effecting a virtually ideal rectangular concentration pulse uniformly across the whole column inlet. The nearly perfect shape of the inlet pulse permitted us to replace the measurement of the inlet concentration by a two-level indication of the instantaneous function of the distributor.

An experimental device has been developed for sampling the outlet stream of liquid. The device permits samples to be taken of feasibly small volume and yet large enough to eliminate the stochastic nature of individual rivulets draining from the bed.

The function of both devices was tested by measurement of the frequency dependences of the amplitude ratio and the phase lag between the outlet and the inlet stream.

For the experimental determination of the hydrodynamic quantities characterizing the flow of liquid and gas in a packed bed column one can apply either the steady state or the dynamic methods. The steady state methods have been used by number of authors to determine *e.g.* the hold-up of liquid in a packed bed column. For instance, Brož[1], Brož and Kolář[2] and Shulman and coworkers[3-5] applied the method of direct weighing of the apparatus under operating conditions on a mechanical balance. Standish[6] suspended the column on fixed-end beam equipped with tensometers; Jiřičný and coworkers[7] hanged the column on a special tensometric dynamometer. By static methods, detecting the concentration profiles, one can measure also additional quantities, *e.g.* back mixing as a particular manifestation of axial dispersion of the flow in packed columns.

The dynamic methods provide a more detailed insight into the character of the measured quantities and a more exacting criterion for testing validity of formulated mathematical models of the flow, incorporating various nonidealities such as stagnant zones, preferential paths, *etc.*

The advantages of the dynamic measurements stem from the fact that corresponding mathematical models contain terms with time derivatives which better discriminate between individual models. Under the steady state conditions some of the phenomena such as *e.g.* the existence of the stagnant zones do not show at all and as such cannot be identified by steady state methods.

The dynamic methods rest in perturbing the inlet stream and detection of the response at the outlet. The perturbation may be effected *e.g.* by a sudden change of the flow rate of liquid or injection of a suitable tracer. As tracers serve various radioactive substances, dyes, electrolytes and others. Injection of a tracer into the inlet stream may lead to different inlet concentration

signals such as *e.g.* impulse, pulse, step change, sinusoidal change *etc.* For identification may serve also random signals and noise present virtually in all real process streams[17-19]. In the former case the measurement consists of detecting the concentration change in the outlet stream. Danckwerts[8] introduced for the response to a step change the term F-curve and for the response to an impulse the term C-curve. As an advantage of the impulse appears the economy of the experiment. The concentration step change corresponds to a transition from one steady state to another. For the latter one needs a relatively large amounts of the tracer. A sinusoidal input calls for a more complicated equipment than other signals. A definite disadvantage is the laboriousness and the necessity of very accurate measurements. For the sake of the economy of the time necessary to obtain a single frequency response, Cusset and Mellichamp[9] perturbed the inlet stream by a signal composed of all requested frequency components

$$x(t) = x_0 + A \sum_j \sin \omega_j t \,. \qquad (1)$$

This method yields as much as 70% savings on experimental time compared to the standard frequency methods. The necessity of forming a defined shape of the input periodic signal may be avoided by the harmonic analysis of the signal as in Eq. (*1*). For the study proper one can utilize only some of the harmonic components present in the inlet. As a disadvantage, of course, remains the necessity to measure the input signal.

A general feature of all dynamic methods is the high requirements on the quality of the measuring detectors. This concerns particularly the satisfactory accuracy of measurement of the transient signal especially at higher frequencies of the perturbation signal. The availability of suitable sensors presents even today a serious constraint on the choice of the experimental method and the system. The most frequently utilized method for the gas phase analysis is thermal conductivity, while electric conductivity is most popular for liquid phase analyses. Goodridge and Bennett[10] used a scintillation detector to measure the concentration of CO_2 marked by C^{14} carbon. Miyanami, Tojo and Yano[11] subjected the liquid (solution of NaBr with methyl orange) prior to entering the column to electrolysis, while the released bromine caused a change of colour of the originally pink solution. The response was measured by photometry at the column exit. In the liquid phase the concentration of the tracer may be detected also by measuring pH (ref.[12,13]). Gunn and Pryce[14] measured the concentration change in the gas phase by a ionization detector. Linek and coworkers[15] measured the concentration of oxygen in the gas and the liquid phase (N_2, H_2O) by means of the oxygen electrode. This electrode operates on the principle of O_2 reduction on the cathode and measurement of the resulting current.

In the experimental determination of the response to an inlet signal experimental difficulties may arise associated with the need to preserve the dynamic information content of the signal. This may become of particular importance during sampling the outlet from the packed bed column. Although the system is usually described by a continuous function (*e.g.* radial velocity profile), the real stream consists of individualized rivulets, the properties of which must be statistically smoothed with minimum damage to the transient characteristic of the signal.

The aim of this work has been to develop a method of generating the signal in the inlet stream with sufficient fidelity eliminating the necessity of measuring the inlet signal by a special detector. In addition, the aim has been to develop a method of sampling the outlet stream with special reference to the specific features of the

outlet stream from a packed bed column, *i.e.* suppression of the stochastic character of individual rivulets while preserving the dynamic information in the sample.

The Realization of the Ideal Periodic Inlet Signal

Generation of the ideal periodic signal calls for a special construction of the distributor which alternates the feed of two liquids into the column that are analytically distinguishable. This method differs from the currently used methods of "tagging" the stream of liquid fed into the column. Tagging is inherently associated with the problem of sudden change of the quality of the stream which is impossible to solve perfectly by technical means owing to the non-zero time required for the mixing of the tracer with the bulk stream.

The construction and the function of the two-chamber distributor for generating the ideal periodic pulse developed in this work is apparent from Fig. 1. The liquid discharges onto the packing through the set of hypodermic needles uniformly distributed over the column cross section. The total number of needles 1·6 mm in diameter was 74. One half of the total number of the needles was connected with one of the two chambers of the distributor, the other half was connected with the second chamber. One of the two chambers was fed with water, the other with a solution of the sodium chromate solution. Each of the two chambers had its own degassing valve. The good function of the distributor depended strongly on the perfect degas-

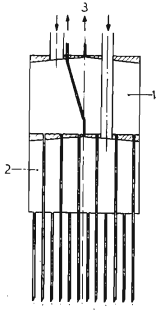

FIG. 1

Construction of the two-chamber distributor. 1 Water, 2 sodium chromate solution, 3 degassing vent

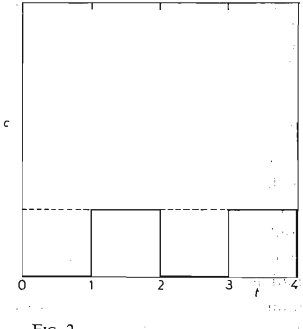

FIG. 2

Inlet concentration signal

sing of the chambers. The degassing ensured the uniform discharge of liquid through all needles of the distributor. The feed lines for water and the tracer solution were equipped with magnetic valves controlled by a time relay which served to generate the requested concentration pulses in the inlet stream. At the time of actuation of the magnetic valve, the other valve was automatically closed. The column was thus fed either by water or the sodium chromate solution. The intervals for actuating the magnetic valves were taken between 5 and 200 seconds. This method generated the inlet periodic signal shown in Fig. 2. With the aid of the just described two-chamber distributor an exactly defined concentration signal was generated eliminating the need for analytical determination of the concentration in the inlet liquid. The nstantaneous function of the distributor was monitored merely by a two-level analog signal: In the period of feeding the column with sodium chromate the corresponding output channel was connected to a 5 V voltage. In the opposite function of the distributor there was a zero voltage in the indication channel. Eventual small deviations of the function from the ideal shape of the rectangular pulse occured due to the inertia forces at the instant of flipping the function of the magnetic valves which caused an additional short-lived weeping of the closed needles. The uniform distribution of both sets of needles over the column cross section lead to the uniform distribution of liquids at either of the two functions of the distributor.

Sampling of the Outlet Stream

Practical sampling of the outlet stream from the packed bed columns poses technical difficulties. The liquid discharges from underneath the supporting grid in the form of individual separated rivulets of liquid and hence methods based on detecting concentration changes in the whole outlet stream cause inherently dynamic distortion of the dynamic properties of the signal in the process of collecting all individual rivulets into a single stream and its transport to the analytical detector.

Individual rivulets, on the contrary possess strongly individualized properties and hence sampling of a single rivulet suffers from strongly stochastic character calling for repeated experiments. This of course, in case of dynamic experiments is very impractical.

The method developed in this work combines both previous approaches while minimizing the disadvantages of both. A sample consisting of a larger number of rivulets is being taken for analysis from positions localized along the column diameter.

This approach achieves sufficient averaging of the outlet stream while the rate of sampling (the flow rate of the sample) is confined to a level technically manageable without serious damage to the dynamic information in the sampled liquid. Technical construction is shown in Fig. 3. In the bottom part of the column there is a grid supporting the whole weight of the packing of glass spheres. The grid, in addition,

facilitates distribution of the gas phase. The area of free cross section of the grid was higher than the area voidage of the packing. One of the bars of the grid shaped as a protruding fin served as a collector of liquid for analysis of the outlet stream. Just below the collecting bar there was a cone continuously diverting the liquid to the analyser. The flow rate of the sampled liquid was regulated by a pinchcock (Fig. 4) to a maximum value below which no entrainment of gas bubbles into the tube diverting the liquid into the analyzer occured. The design and shape of the middle collecting fin effectively averaged the sample and suppressed the individualized character of the rivulets trickling down the packing and their spatial fluctuations without causing undue difficulties associated with the analysis of the entire outlet stream. This collected sample represented the liquid averaged across the column diameter and hence represented both the central regions as well as regions adhering to the column wall.

EXPERIMENTAL AND RESULTS

The function of the devices for generation of the ideal pulse and for sampling the draining liquid was tested experimentally. The experiments were carried out in a glass column 106 mm in diameter packed to a height of 2·1 m by glass spheres 10 mm in diameter and fed with the water–air system. A scheme of the apparatus is shown in Fig. 5. Pressurized air was drawn from the distri-

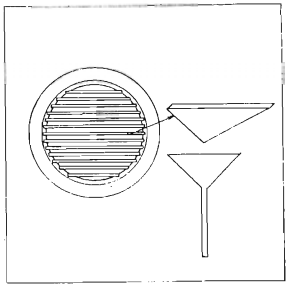

FIG. 3
Sampling of the outlet stream

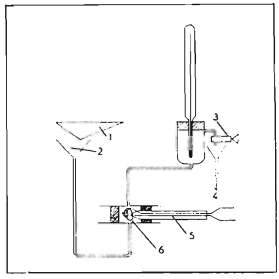

FIG. 4
Analysis of the outlet stream. 1 Collecting fin of the grid, 2 cone, 3 pinchcock, 4 drain, 5 electrode, 6 conductivity cell

buting pipe *via* a pressure reducing valve *1* to a humidifying column *3* packed with ceramic Rashig rings and further to the packed column proper *4*. The flow rate of air was metered by a rotameter *2*. Water of desired temperature was obtained by mixing warm and cold water from taps using a magnetic valve *5*. Water was then fed into the overflow tank *6a* ensuring constant pressure head. Water from the overflow tank was brought *via* a magnetic valve *9b* into the column *4*. The magnetic valve served (together with the magnetic valve *9b*) to generate concentration pulses in the entering liquid. The flow rate of water was metered by a rotameter *10*. Solution of sodium chromate of concentration $5 \cdot 10^{-4}$ kmol/m^3 was prepared in a storage tank *11*. From here the solution was pumped by a pump *12* to an overflow tank *6b*. From this tank the solution proceeded *via* a magnetic valve *9a* and a rotameter *13* to the column. The drain for water was constructed in such a manner so as to a provide a hydraulic syphon closing the bottom of the column.

Electric conductivity method was chosen to monitor the concentration of the tracer in the outlet stream of liquid. The scheme is shown in Fig. 4. Electric conductivity depends significantly on temperature and it was therefore necessary to keep the temperature of the inlet stream constant. As an electrode for the conductivity measurements we used the modification proposed by Khang and Fitzgerald[16] for the measurement of conductivities of electrolytes. An advantage of the electrode of this type is the elimination of the potential due to the grounding of the conductivity cell via the draining stream of liquid. This grounding exercised harmful effects for other types of electrodes. The elimination of this effect is particularly important for the dynamic measurements when the resistance of the grounding stream is time-variable and as such hard to compensate accurately. The conductivity measurements were carried out using an instrument on the principle of the Wheastone bridge developed in the Institute of Chemical Process Fundamentals. Its measuring range was between 500 and 2 000 ohms and the corresponding output analog signal -1 to $+1$ V was processed by a data logger. The concentration in the outlet liquid from the column was recorded on a punched paper tape together with the two-level indication of the instantaneous function of the inlet distributing device. The sampling frequency of the recording was 1 channel per 0·1 second. The concentration of sodium chromate solution was $5 \cdot 10^{-4}$ kmol/m^3. Up to this limit the concentration dependence of electric conductivity was found linear.

Data processing. The Fourier expansion of the outlet or the inlet signal is

$$c(t) = b_0 + \sum_{n=1}^{\infty} a_n \sin(n\omega t) + \sum_{n=1}^{\infty} b_n \cos(n\omega t) . \qquad (2)$$

In this study we shall make use of only the principal frequency (*i.e.* for $n = 1$)

$$c'(t) = b_0 + a_1 \sin(\omega t) + b_1 \cos(\omega t) . \qquad (3)$$

For our inlet signal we have

$$c''(t) = \frac{4}{\pi} \sum_{n=1}^{\infty} \frac{\sin[(2n-1)t]}{2n-1} . \qquad (4)$$

From the last equation it is apparent that the dominant frequency has $(4/\pi)$ times greater amplitude than the perturbing pulse and three times greater amplitude than

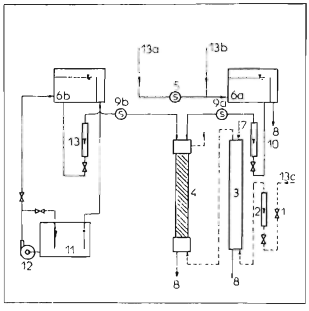

FIG. 5

Scheme of experimental set-up. 1 Pressure reducing valve, 2 rotameter for air, 3 humidifying column, 4 packed bed column, 5 magnetic valve, 6a,b overflow tanks, 7 water feed of the humidifying tower, 8 drain, 9a,b magnetic valves, 10 rotameter for water, 11 storage tank, 12 pump, 13 rotameter for Na_2CrO_4 solution, 13a feed of warm water, 13b feed of cold water, 13c feed of air

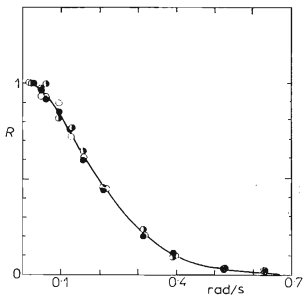

FIG. 6

Amplitude ratio as a function of the frequency of the input signal for liquid: $v = 7{\cdot}864 \cdot 10^{-3}$ m/s; gas: $v = 0$ ●; $1{\cdot}259 \cdot 10^{-1}$ m/s ◑, $1{\cdot}763 \cdot 10^{-1}$ m/s ○

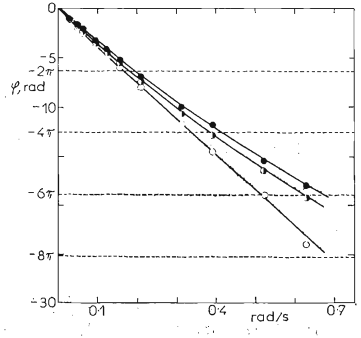

FIG. 7

Phase lag as a function of the frequency of the inlet signal for liquid: $v = 7{\cdot}864 \cdot 10^{-3}$ m/s; gas: $v = 0$ ●, $1{\cdot}259 \cdot 10^{-1}$ m/s ◑, $1{\cdot}763 \cdot 10^{-1}$ m/s ○

the nearest higher harmonic frequency component. Using familiar formulas the inlet and the outlet data were used to compute the amplitude ratios and phase lags of the output to the input signal.

The results were processed only for the dominant frequency. Fig. 6 and 7 show examples of experimental frequency dependences of the amplitude ratio and the phase lag. The smoothness and reproducibility of the obtained dependences confirms the correct function of the devices for the generation of the signal and the sampling of the outlet stream.

CONCLUSION

An experimental method has been developed to generate periodic concentration pulses in the inlet stream with a negligible departure from the ideal rectangular shape across the whole inlet cross section of the inlet stream. The measurement of the inlet concentration was replaced by a two-level indication of the state of the inlet stream without the necessity of resorting to sampling the inlet stream. A method has been developed of sampling the liquid draining from the packing below the supporting grid. The results obtained in the form of amplitude ratios and phase lags confirm the correctness of the developed methods.

LIST OF SYMBOLS

a_n, b_n	Fourier coefficients
A	amplitude
c, c', c''	concentration (kmol/m^3)
t	time (s)
$x(t)$	inlet signal
ω	frequency (rad/s)
R	amplitude ratio
φ	phase lag (rad)

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