A TENSOMETRIC METHOD TO DETERMINE THE HOLD-UP OF LIQUID IN PACKED BED APPARATURES

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Results have been presented in the paper of the tests and calibration of a tensometric scale developed for weighing packed bed columns under operating conditions. The results have shown the tensometric method to be suitable for weighing packed bed columns under the two-phase flow of gas and liquid in the dynamic state. Experimental results have been presented of the steady state liquid hold-up and gas pressure drop obtained by the developed tensometric method in an experimental column 190 mm in diameter. The experimental data have been compared with those of other authors obtained by different experimental techniques.

Specific volume hold-up of the liquid phase is an important quantity from the view of the operation, productivity and the dynamics of the packed bed type equipment in its diverse applications as *e.g.* separation units, trickle bed reactors, *etc.*

The choice of the experimental technique of measurements of the liquid hold-up depends on eventual presence of the counter currently flowing gas, on whether the aim is to measure the static, dynamic or the overall hold-up, *etc.*

Less frequently used methods are based on the absorption of rays by liquid[1,2] or utilize a tracer[3,4]. The simplest method of determining liquid hold-up rests in sudden shut-off the flow rates of the phases[5]. That method, however, is inaplicable for dynamic studies.

A most frequently applied, continuous non-destructive method of determination of the hold-up is based on weighing the column under the operating conditions. This method has been used by numerous authors[6-11] and, as another advantage, it does not restrict the choice of the packing material and the liquid. Weighing on a mechanical scale is again out of question for dynamic studies due to the inertia forces and the dynamics of the scale proper. A qualitatively new step in perfecting this technique represents the use of tensometers (strain gauges) as shown by Standish[11]. For experiments on a small laboratory packed bed column, this author used a tensometer monitoring the deformation of a fixed-end beam supporting the suspended column.

Our aim has been to develop an experimental method of measurement of liquid hold-up in both the steady state and the dynamic operating conditions in a column of the total mass of about 200 kg. For current materials this mass enables measurements in columns up to 300 mm in diameter packed to about 1 m depth. The mass of the liquid hold-up for this system ranges roughly in the units of kilograms and for the requested relative accuracy of the hold-up of 1% there follows the need to determine the overall mass with the accuracy of about 10 g. In contrast to the experimental set-up developed by Standish[11] our task is different in that we require an equipment independent of the suspension gear and are faced with a large tare (dead weight). For these purposes the fixed-end beam appears not very well suited even from the stand point of the dynamic properties.

EXPERIMENTAL

The Tensometric Scale

The developed tensometric scale consists of a dynamometer and an electronic part. The dynamometer is essentially a mechanical deformation element shaped as a cross equipped with eight strain gauges forming individual branches of a DC Wheatstone bridge. The electronic part transforms the signal from the measuring diagonal of the bridge to two DC analog output levels of 0—5 mV and 0—1·5 V. The deformation element proper is based on the design of SVÚSS, Běchovice. A detailed description of the tensometric scale, design and construction has been presented elsewhere[12–14].

Calibration Procedure

The aim of the tests of the tensometric scale, *i.e.* the dynamometer together with the electronic unit, has been to determine its sensitivity to the changes of the mass of the load m (kg) suspended on the dynamometer. The tests consisted of: *a*) test of linearity of the output signal over the range of the expected load 0—200 kg, *b*) test of linearity of the output signal following an electronic balancing of the signal of the bridge due to the tare (dead weight), *c*) test of sensitivity of the scale for different settings of the supply voltage, amplification and the divider of the output voltage from the tensometric bridge, *d*) test of the drift of the base level of the signal from the differential amplifier, *e*) test of hysteresis of the dynamomter following gradual increase and decrease and decrease of the load.

The tests and the calibration of the dynamometer were all carried out on a simple test bench formed by a strong U-iron frame. In the upper horizontal edge of the frame there was a hanger for the dynamometer whose lower coupling hook carried a loading rod. The load of the could be varied stepwise up to the maximum; for small increments of the load, below 10 kg, there was a small pan. The dynamometer was connected to the electronic unit by a shielded five-conductor cable. The output signal 0—5 mV was recorded on an EZ-9 line recorder, the 0—1·5 V output was digitalized by a compuring data logger ADIMES. In order to suppress the effect of the fluctuation of the laboratory temperature the dynamometer was thermally insulated by polyurethane foam.

The Experimental Set-Up to Measure the Liquid Hold-Up by the Tensometric Scale

The scheme of the experimental set-up with the counter-current arrangement of the flows of the phases is shown in Fig. 1. The liquid is pumped from a thermostated storage tank 1 by a centrifugal pump 2 *via* a regulating valve 3 and a bank of rotameters 4 into a distributor 5. Excess liquid returns from the pump back into the tank 1 *via* a by-pass 6. After passing the bed of the packing 24 the liquid is separated from the gas in an external chamber of a syphon 7. The height of the overflow tube 9 returning the liquid from the column into the tank has been adjusted so as to maintain the height of the clear liquid above the level of the outlet 10. This eliminates, to a large extent, the effect of the fluctuation of the level of the draining liquid on the pressure drop of the gas. The temperature in the thermostated tank 1 is measured by a mercury thermometer 11; past the rotameters by a Pt-thermometer 12. At the inlet end the rotameters are equipped with closing valves 13. The distributor 5 has a venting cock 14 and is suspended, independently of the column suspension 15, from the supporting frame 46. Over the column cross section the liquid is distributed by a set of capillary tubes (internal diameter 1·5 mm, 250 mm in length) discharging 50 mm above the top of the packed section. The volume of liquid in the syphon 8 is constant. The correction on the buoyancy force due to the different depth of submersion is encopassed in the calibration of the pressure transducer. The height of the syphon chamber is 1 m.

Pressurized air 0·5 MPa was brought *via* an oil separator 17, a pressure reducing valve 18, a regulating valve 19 and a bank of rotameters 21 into the internal chamber 7 of the syphon. The outlet end of the gas tube reaches above the liquid level and it is shielded by a roof 22. After passing the bed 24 the air discharges into the atmosphere. Its pressure is gauged by U-tubes 28 filled with water and the temperature is measured by a Pt-thermometer 27.

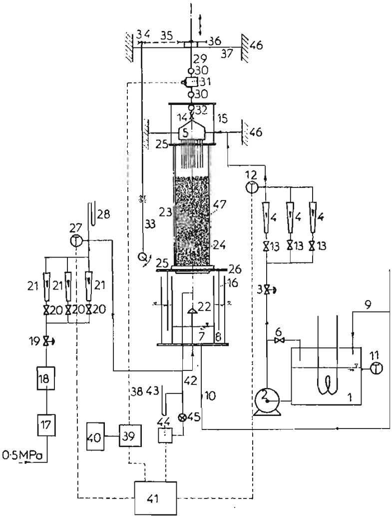

FIG. 1

Experimental set-up. For description see the text

The packed column is a 190 mm ID perspex glass cylinder 23 1·25 m high. The bars 47 connect flanges 25. The lower flange carries a grid supporting a 1 m high packed section of glass spheres 10 mm in diameter. This part of the set-up (the cylinder and the flanges) is replaceable and permits experiments with cylinders up to 300 mm in diameter. The lower flange 23 of the column is mounted to the upper flange 26 of the bell 16 which is a part of the syphone 8; the upper flange is mounted to the hanger 15.

The essential parts of the column suspension are adjustable screw 29 and hooks 30 mutually rotated by 90° forming together with the dynamometer 31 the Cardan joint. The lower hook 30 is coupled by screw, passing through the upper part of the hanger 15, to a ball journal 32. Arresting mechanism of the column is controlled by a shaft 33 with a tooth gear 34. Its rotation is transmitted by the Gall's chain 35 to a gear 36 equipped with an internal thread transforming the rotation to the translational motion in the direction of the column axis. Both toothed wheels 35, 36 are mounted on a firm bar 37 fixed to the frame 46. In the arrested position the column rests with the upper flange of the bell 26 on the external jacket of the syphon 8 and the ball journal is loosed of the upper edge of the hanger 15. In the active position all weight of the column, the bell 16 and the hanger 15 is transmitted by a ball journal 32 to the dynamometer; the bell is without contact with the internal and the external jacket of the syphon 8. The proposed design of the column suspension using a ball journal permits a reliable vertical positioning of the column.

Excepting the air tubing, which is made of steel, all remaining parts contacting the liquid are made of corrosion resistant materials (PVC, glass, brass, copper).

The packed column was suspended on the tensometric scale. The dynamometer 31 was connected by a coaxial cable 38 to the electronic part of the scale 39. The 0—5 mV output was fed to a strip chart recorder 40; the 0—1·5 V output was fed to a computing data logger ADIMES 41. The dynamometer 31 was thermally insulated by a polyurethane foam.

The pressure drop was measured by a capacitance transducer DISA 44, connected also to the ADIMES logger 41. The pressure transducer was fitted with a 0·14 mm thick membrane with the corresponding pressure range up to 0·01 MPa. The inlet end pressure port in the internal chamber of the syphon was connected by a tube 42 *via* a three-way cock 45 to the transducer 44 and to a U-tube 43 manometer gauging the height of liquid in the syphon. The effect of temperature on the reading of the pressure transducer was eliminated by a closed-loop control of the temperature in the transducer jacker and thermal insulation by polyurethane foam.

The experiments were carried out with the water/air system. Tap water was thermostated to 20 ± 0·5°C. The temperature of air from the piping ranged between 19 and 21°C.

The calibration of the pressure transducer was carried out by static pressurization of the column blinded at the top flange 25. From the known apparent loss of weight of the column due to the over-pressure within the bell 16 and from the known area of cross-section of the upper flange of the bell 26 one could calculate the over-pressure in the bell. Since the employed techniques encompasses correction on the buoyancy force due to the different submersion of the syphon, the dependence of the pressure gauge in the bell on the output signal of the transducer was correlated as a second-order polynomial. The measurement showed the constant of the quadratic term to have been an order of magnitude lower than that of the linear term. The repeated checks in the course of measurements confirmed long-term stability of calibration of the transducer.

RESULTS

Calibration Measurements

The results of the calibration measurements of the tensometric scale for different settings of the supply voltage, amplification and the output signal voltage divider are shown in Fig. 2. All curves display a linear course of the output voltage in the whole range of the load. The correlation coefficient of all dependences was better than 0·9997. From the ratio of sensitivities of individual measurements (Table I, the last but one column) one can asses the accuracy of the output voltage divider. This accuracy was found better than 1·5%. Also a comparison of the sensitivity of measurements at two different levels of amplification indicate that the error is less than 2%. Finally, the measurements have shown the ratio of sensitivies to be approximately proportional to the corresponding ratio of the supply voltage.

An important criterion for the assessment of suitability of the dynamometer and the electronic part for the given purpose is its sensitivity electronic balancing the signal of the bridge due to the dead weight. Table I shows the results of such measurements. The sensitivity in the last but one column corresponds to the loading of the dynamometer by large weights. The sensitivity in the last column represents loading of dynamometer after electronic componensation of the dead weight of 104·5 kg. The found sensitivities differ by 1·5% as a maximum.

Fig. 3 shows the measurements at various limiting levels of the supply voltage and amplification (Table I).

The measurements were carried out at constant load of 104·5 kg, while compensating this load electronically. The results show that electronic compensation of a large constant load did not affect the sensitivities at various settings of the supply voltage or amplification. From Fig. 3 it is also apparent that the hysteresis of the dynamometer, even at maximum sensitivities is at the limits of measurability.

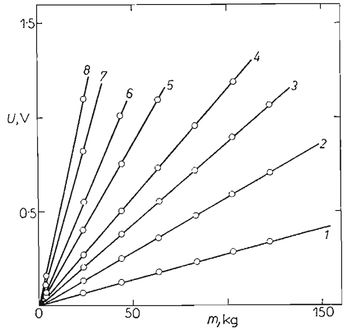

FIG. 2

Calibration curve of the tensometric scale. For description see Table I

At constant load the stability of the output voltage is significantly affected by the supply voltage and amplification of the differential amplifier. Higher supply voltage increases the sensitivity but also the thermal strain of the tensometers affecting

TABLE I

Measurement of sensitivity of the tensometric scale

Curve[a] No	Supply voltage V	Aplification	Output divider setting	Measured sensitivity, V/kg large load	Measured sensitivity, V/kg electronically compensated load 104·5 kg
1	2	10	1 : 16	0·0029	0·0029
2	2	10	1 : 8	0·0058	0·0058
3	6	10	1 : 16	0·0087	0·0087
4	2	10	1 : 4	0·0116	0·0115
5	6	10	1 : 8	0·0174	0·0171
6	4	10	1 : 4	0·0232	0·0231
7	6	10	1 : 4	0·0348	0·0345
8	2	10	1 : 1	0·0462	0·0461
9	4	100	1 : 1	0·0851	0·0849
10	6	10	1 : 1	0·1391	0·1391

[a] See curves in Figs 2 and 3.

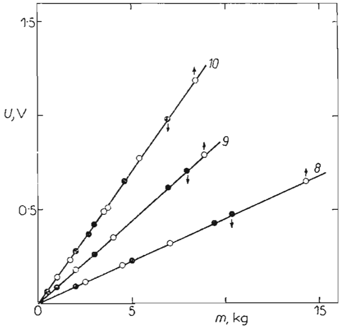

FIG. 3

Calibration curves of the tensometric scale for extreme settings of the supply voltage and amplification. ↓ ● direction of decreasing load, ↑ ○ direction of increasing load. For description see Table I their resistance and hence the output voltage of the bridge. Sufficiently long warm-up period of the apparatus (about 2 hours) and good thermal insulation considerably contribute to elimination of this phenomenon as well the effects of fluctuating temperature and stabilize the thermal regime within the dynamometer. The long-term measurements indicate that the best results are achieved when the instrument is kept with the power supply permanently switched on.

High aplification (100 times) is essential for accomplishing high sensitivities but at the same time increases the possibility of the shift of the zero line of the differential amplifier.

Both these phenomena compounded at the maximum sensitivity a maximum drift of 48 mV/hour with the corresponding change of the apparent load of about 50 g/hour. For long-term measurements it is advantageous to set a higher supply voltage and lower amplification; for short-tem measurement the reverse is more beneficial. During high sensitivity measurements, calling for a high resolution power, it is advantageous to check periodically the output signal of the arrested dynamometer and correct the measured data on this value. Using this method the maximum sensitivity of our set-up reaches 2 g, *i.e.* one can detect the addition of a 2 g load with the constant dead weight of 104·5 kg.

The Measurement of Liquid Hold-Up

In order to eliminate the start-up characteristics of the tensometric scale and the pressure transducer a 4 hour warm up period was standardized. Experience, however, showed that the best results are achieved when the instrument is kept constantly under power.

The calibration of the tensometric scale was periodically checked by the mass of the dry column plus the extra weight between 1 and 5 kg. No variation of sensitivity was detected during measurements.

The wetting of the column prior to the measurements proper was carried out, after degasing the distributor, by water at the rate of 2·5 m^3/hour. This method is termed in the literature as wetting by flooding[9,10].

After wetting the column the flow rate of gas was set to a required level and after steadying the regime the measurement of the column weight and gas pressure drop was carried out. The pressure drop was taken as the difference of the inlet gas pressure and the atmospheric pressure. The flow rate of gas was then gradually increased up to the flooding.

Each run represented the set of 300 data of each quantity scanned at the frequency of 10 channels per second. The mean and the standard deviation were computed from this set for each quantity. She measurement for each setting of gas and liquid flow rates was repeated three times.

The zero level of the output voltage of the tensometric scale and the pressure transducer was repeatedly checked in the arrested state of the column and with short-circuited chambers of the pressure transducer by the cock 45 (Fig. 1). The check of the zero level was performed prior to each measurement of the three sets of data for a given gas and liquid flow rate. The obtained set of 300 data was processed as described above.

The calculation of the mean and the standard deviation and the transformation to physical quantities, *i.e.* the specific hold-up, h (m^3/m^3) and the pressure drop Δp (kPa) in dependence on the superficial liquid mass velocity, Q_l, and gas mass velocity, Q_g, both in (kg/m^2 s), was performed directly during measurements by the arithmetic unit of the ADIMES data logger. The hold-up was computed from the apparent weight of the column under operation, the pressure force of the flowing gas, given by the pressure transducer, and from the weight of the dry column.

The dependence of liquid hold-up on superficial gas mass velocity, Q_g, with the liquid mass velocity, Q_l, as a parameter, is shown in Fig. 4. The form of the dependence is in agreement with the results published in the literature[6,9,10]. For low liquid velocities tue liquid hold-up is practically independent of the flow rate of air and increases only near the flooding point. At high irrigation rates the liquid hold-up increases immediately from the zero air rate.

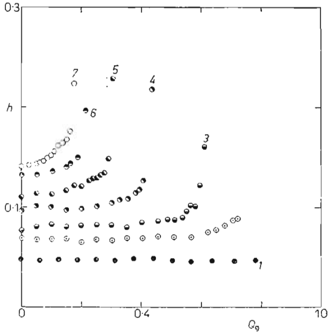

FIG. 4

Hold-up of water h as a function of superficial gass mass velocity Q_g with superficial water mass velocity Q_l as a parameter. *1* $Q_l = 0{\cdot}2939$ kg/m^2 s; *2* 1·4207; *3* 2·331; *4* 4·673; *5* 6·873; *6* 9·308; *7* 11·023

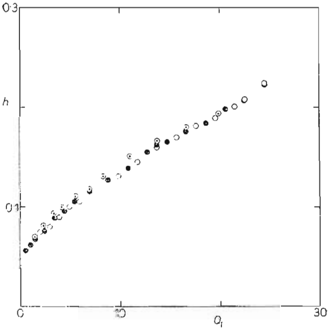

FIG. 5

Hold-up of water h as a function of superficial water mass velocity Q_l. ○ ● Measurement from this work and after a 7 day recess, ⊙ measurement of Brož[9]

Fig. 5 shows a comparison of our results with those of Brož[9], who measured with the aid of a mechanical scale. Both measurements were carried out in columns 190 mm in internal diameter and with a packing of glass spheres 10 mm in diameter,

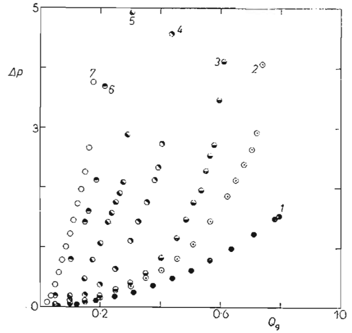

Fig. 6

Gas pressure drop Δp as a function of superficial gas mass velocity Q_g with superficial water mass velocity Q_1 as a parameter. *1* $Q_1 = 0{\cdot}2939$ kg/m s; *2* 1·4207; *3* 2·331; *4* 4·673; *5* 6·873; *6* 9·308; *7* 11·023

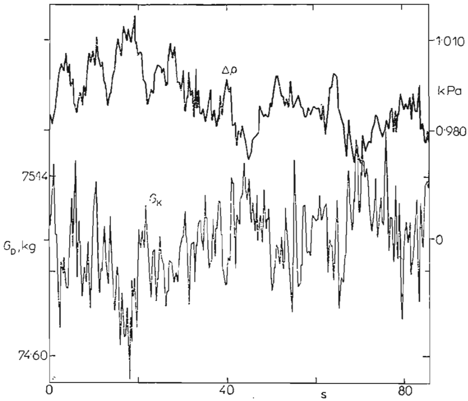

Fig. 7

A chart of oscillations of pressure drop Δp and of the apparent mass of the column G_D

packed 1 m deep and with the air/water system. Apart from the different method, Brož used also a rotating distributor for liquid. The comparison of both results shows a good agreement.

The results in Fig. 5 display a good reproducibility of measurement which suggests also a good reproducibility of the column wetting, of the formation of the hold-up under otherwise the same conditions even within the time span of seven days.

Fig. 6 shows the dependence of pressure drop on the flow rate of air for liquid rates corresponding to the dependences in Fig. 4. Also these results have and analogous course as the published data[9].

Fig. 7 depicts the time series of the instantaneous values of gas pressure drop Δp and the apparent weight of the column, G_D (*i.e.* not corrected on the pressure force of the air flow). In view of the used analog-to-digital converter the "instantaneous" values are averages over the period of intergration ($\tau = 80$ ms) of the AD converter. This figure gives a good assessment of the potentials of the developed method for the investigation of the dynamic behaviour of packed columns.

CONCLUSION

A tensometric method has been developed of weighing the packed bed columns under two-phase counter-current flow of gas and liquid, suitable both for the steady and dynamic measurements. The developed method enables continuous non-destructive measurements of instantaneous values of the overall liquid hold-up.

The calibration measurements have shown the tensometric scale to be able to determine the overall mas of 200 kg with the limiting sensitivities of 2 g, while the calibration curve is linear in the whole range of admissible load. The equipment permits electronic compensation of the dead weight and hence observation of small time variations of liquid hold-up with a high accuracy.

The obtained experimental dependences of the liquid hold-up on the flow rates of gas and liquid are highly reproducible and agree with the so far published data obtained by other methods.

LIST OF SYMBOLS

Δp	gas pressure drop, kPa
h	specific hold-up of liquid, m^3/m^3
Q_g	superficial gas mass velocity, kg/m^2 s
Q_1	superficial liquid mas velocity, kg/m^2 s
G_D	uncorrected mass of the column, kg
m	load, kg
U	output signal, V

REFERENCES

1. Hwa C. S., Beckman R. B.: AIChE J., *6*, 359 (1960).
2. Dombrowski H. S., Brownell L. E.: Ing. Eng. Chem. *46*, 1207 (1954).
3. Hrubý M.: Chem. Prům. *12*, 394 (1962).
4. De Maria F., White R. R.: AIChE J. *6*, 473 (1960).
5. van Swaay W. P. M.: Chem. Eng. Sci. *24*, 1083 (1969).
6. Shulman H. L., Ullrich C. F., Wells N.: AIChE J. *1*, 247 (1955).
7. Shulman H. L., Ullrich C. F., Proust A. Z., Zimmerman J. D.: AIChE J. *1*, 253 (1955).
8. Shulman H. L., Ullrich C. F., Wells N., Proust A. Z.: AIChE J. *1*, 259 (1955).
9. Brož Z.: *Thesis*. Czechoslovak Academy of Sciences, Prague 1965.
10. Brož Z., Kolář V.: This Journal *37*, 3551 (1972).
11. Standish N.: Ind. Eng. Chem. Fund. *7*, 312 (1968).
12. Research Report No 37/79. Institute of Chemical Process Fundamentals, Prague 1979.
13. Šmíd J., Jelínek V., Jiřičný V., Staněk V.: Chem. Listy *75*, 1303 (1981).
14. Jelínek V., Šmíd J., Jiřičný V., Staněk V.: Chem. Listy *75*, 1298 (1981).

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