An Experimental Study of Absorption of Oxygen in Water in Co-Current Packed Bed Column by Transient Technique

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Parameters are evaluated by mathematical models using measured experimental data on unsteady state absorption of oxygen as a gas poorly soluble in water in a co-current packed bed column. The most complex model is an eight-parameter model with axial dispersion in gas and liquid phase and a stagnant zone in liquid phase. The other models are various asymptotic cases of the basic model. Transfer functions for the five utilized models have been derived in an earlier paper.

The carried out experiments continuously measure the response in the gas and liquid column outlet streams to a periodic change of oxygen concentration in inlet gas in a 0.105 m diameter column. Using various frequencies of the periodic input concentration signal the frequency characteristics have been obtained and in turn used to evaluate model parameters such as holdups of gas, dynamic liquid and stagnant liquid, mass transfer coefficients to dynamic and static holdup, and axial dispersion coefficients in gas and liquid phase in the form of Peclet numbers. Simple correlations of these results on gas and liquid Reynolds numbers were evaluated. The ability to evaluate quantities of models of various complexities was assessed based on the experimental data obtained.

Keywords:
Packed bed, co-current down flow, oxygen absorption, periodically variable input, parameter evaluation

Introduction

Depending on the character of an experiment existing methods of study of mass transfer in two-phase packed bed columns may be classified as either static or dynamic. Static methods measure steady state concentration profiles of a species along the column height to evaluate model parameters. This approach has been used for instance by *Burghardt* and *Bartelmus*[1,2] and *Hutton* and *Woodborn*[3] studying the effect of axial mixing on interfacial mass transfer in packed columns. Static methods, however, suffer from certain disadvantages such as the necessity to place sampling probes into the packed section. Also the absence of accumulation terms in the balance equations causes that certain parameters cannot be studied.

The mentioned difficulties are absent with the use of dynamic methods.[4] These methods monitor the course of outlet concentration of a species following a change at inlet end. A disadvantage of the dynamic methods is the need for measurement of unsteady-state concentrations which calls for fast and accurate analytical probes. With the technical progress of data acquisition and on-line processing dynamic methods become increasingly popular.

The method of parameter evaluation depends on the employed mathematical model,[5,6] characteristic of the analytical probe, and the type of the input signal. These methods may be classified as either momentum or regression methods. The evaluation may be carried out either directly in the time domain, in the Laplace, or the frequency domain. Existing methods of evaluation are described in detail in papers[7,8]. A comparison of the momentum and regression methods[8,9] showed, the latter carried out in the frequency domain to be more accurate, but the penalty is an increase of computer effort.

Most of the existing papers relate to a single-phase flow and the experiments were carried out without exception in small-diameter columns packed with small-dimension packings. The reason is obvious. Such experiments are easier to realize and less demanding. The results obtained in small-diameter columns pertain, perhaps, more to catalytic processes.[10] Unfortunately, the experiments were carried out, with few exceptions,[10] with water as the irrigating liquid and at atmospheric pressure, which is a poor model for catalytic trickle bed reactors.

Unlike the literature data this work undertook experiments with a column and packing of larger diameter. This should enhance the relevance of the results to industrial size columns. Unfortunately us-

ing water as liquid phase will inherently worsen eventual agreement with the data obtained with small-dimension packings. This is due to the high static holdup of water held as bridges between individual small pieces of packing.

Theory

The derived relationships valid for individual mathematical models have been presented in our preceding paper.[5] Table 1 gives a review of the employed models, their characteristics and conditions of quantity evaluation. The last column indicates constraint that was used in parameter evaluation.

Experimental set up

The water-air system was selected for experiments with oxygen being the absorbed species. The measurements were carried out on a setup shown schematically in Figure 1. A column (1) of internal diameter 0.105 m was packed to a height of 2.05 m by glass spheres 0.01 m in diameter. The voidage of the packed section was 0.4. The scheme of the apparatus is shown in Figure 1.

Water as the liquid phase was thermostated in a tank (2) and its constant flow rate was kept by means of a constant head overflow tank (3) located about 5 m above the column top. Water flowed from the overflow tank (3) into the liquid distributor securing uniform irrigation of the top of the packing. The volume of the top space above the
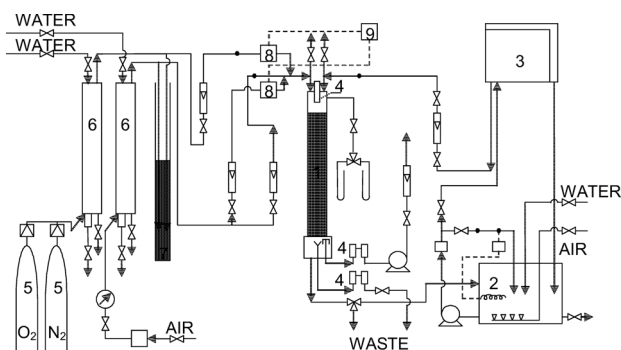

F i g . 1 – *Scheme of the experimental setup*

packing was minimized in order to restrict the dead volume that could affect the shape of the inlet oxygen concentration signal.

The air as the gas phase was taken from the pressurized-air pipeline. The tracer (oxygen or nitrogen) was taken from pressure cylinders (5) and lead via humidifying columns (6), a manostat (7), and magnetic valves (8) into the column head. The magnetic valves were controlled by a programmable data logger (9) creating periodic signals in the gas phase. The measuring oxygen concentration probes (4) were located at the gas inlet and outlet end and at the outlet liquid end.

The generated oxygen concentration signal was created in the form of a rectangular periodic pulse and the responses to this signal were monitored at the outlet ends of the gas and liquid phases. The concentration probes were oxygen electrodes of the polarographic type.

T a b l e 1 – *Review of employed mathematical models*

Model	Quantities			Constraint
	designation	number	free	
PF-PF	h_G, h_L, $k_L a$	3	2	$h_L+h_G = 0.4$
PE-PF	h_D, h_G, h_S, $k_{LS}a_S$, $k_{LD}a_D$, q	6	4	$h_L+h_G = 0.4$; $h_S+h_D = h_L$ h_L from PF-PF
AD-AD1	E_L, E_G, $k_L a$, h_G, h_L	5	4	$h_L+h_G = 0.4$
AD-AD2	E_L, E_G, $k_L a$, h_G, h_L	5	3	$h_L+h_G = 0.4$; h_L from PF-PF
PE-AD	E_G, h_G, h_D, h_S, $k_{LD}a_D$, $k_{LS}a_S$, q	7	5	$h_L+h_G = 0.4$; $h_S+h_D = h_L$; h_L from AD-AD
PDE-AD	E_D, E_G, h_G, h_D, h_S, $k_{LD}a_D$, $k_{LS}a_S$, q	8	5	$h_L+h_G = 0.4$; $h_S+h_D = h_L$; h_L from AD-AD $q = 0.003$

Experimental procedure

Initially, the apparatus had to be brought to the pseudo-steady state for which the solutions of the mathematical models have been derived.[5]

The measurements properly consisted of two parts: First the measurements of the signals of the output oxygen electrodes under steady state conditions and extreme (from the standpoint of the given experiment) concentrations of oxygen were carried out.

The added gas species were either oxygen or nitrogen which both cause a change of oxygen concentration in the air. With the use of oxygen to generate concentration pulses the upper limiting value was obtained by measurement in the air-oxygen mixture and the lower limit by the measurement in pure air. If the used species was nitrogen then the upper limit was measured in the pure air and the lower limit in the air-nitrogen mixture.

The measurements (checks) of the steady state limiting values were carried out always at the beginning of each series of experiments with variable frequency of the generated concentration signal, then after each fifth frequency measurement, and finally at the end of the series. Each check was measured at least twice and average values were used for evaluation. Steady state values for individual frequencies were then interpolated using the three measurements.

In the following part the measurement of the transient oxygen concentration at variable frequency of the input oxygen concentration signal, was carried out. This series of measurement started from the lowest frequency toward the highest frequency of the input periodic concentration signal (from the period of 240 to 4.2 s).

For each frequency selected for the measurement we waited for several periods of the change (min) in order for the column to attain the pseudo-steady state. Then the logging of the transient concentration data was actually initiated. The logged data actually covered several periods in order to suppress the error of measurements that could show particularly at high frequencies of the input signal. The product of each measurement were the coefficients of the Fourier transform for individual frequencies. Each experimental set was concluded by the test of linearity of the employed oxygen electrodes.

In view of the fact that the polarographic oxygen electrode is not a probe with instantaneous response it was necessary, before further data processing, to determine the dynamic characteristics of the oxygen probe itself. The test of the electrode dynamics was analogous to that of the measurement of the column itself. The experimental setup, however, was much simpler and enabled simultaneous tests of two electrodes.

The tests of the dynamics of the electrodes were carried out with pure oxygen and nitrogen. The periodic signal was generated in an analogous manner. We have tested the electrodes at zero frequency (steady state values) first. The linearity of the electrodes was tested by using oxygen-air mixtures.

The range of frequencies for testing the oxygen electrodes was wider and ranged between the length of the period of 3 and 240 s. The flow rates of gases were kept constant (60 l h^{-1}) and the temperature was kept constant and equal to that of the laboratory.

Since the dynamic properties of the polarographic electrodes lack long-term stability and are temperature dependent, the frequency characteristics of the electrodes themselves were repeated at least once a week.

A total of 55 sets of experiments of absorption of oxygen in co-current packed bed column under dynamic conditions, were carried out. These experiments covered seven different liquid flow rates in the range of superficial velocities between 0.002 and 0.014 m s^{-1}. For each liquid flow rate the experiments were carried out for 6 to 8 gas flow rates covering the whole range of velocities available with our experimental setup.

The range of superficial gas velocities was between 0.12 and 0.37 m s^{-1}. The minimum superficial gas velocity (0.12 m s^{-1}) was used for all above given superficial liquid velocities. In view of the way of generating the input concentration signal lower gas rates were not feasible. For each of the superficial liquid velocity there was a maximum superficial gas velocity limit due to the change of the pressure conditions in the column head. The flow rate of either oxygen or nitrogen generating the concentration pulse was chosen so as to keep the difference of the electrode reading for the extreme concentration above the minimum of 0.2 V from the available range of readings 0 – 1 V. This flow rate amounted to 1400 l h^{-1}.

The temperature of the liquid phase during the experiments ranged between 19.5 and 20.8 °C. The gas phase was not thermostated. The equilibrium constant was evaluated for the temperature of the liquid phase.

The flow rate of the gas samples at the exit was the same for all experiments and equal to 60 l h^{-1}. The flow rates of sampled liquid at the outlet ranged between 8.6 and 27.3 l h^{-1} correspondingly to the employed liquid rate in the column.

Data processing and evaluation

Evaluation of the model parameters from experimental responses of oxygen electrodes took place in three steps. In the first step the outputs of all oxygen electrodes were processed on-line by the data logger which converted these signals to the form of the Fourier transform coefficients.

In the second off-line step the frequency characteristics of the transfer functions, gas-to-gas X_{GZ}/X_{G0} and gas-to-liquid X_{LZ}/X_{G0}, were calculated. The amplitudes and phase lags were computed from the Fourier coefficients. The amplitude ratios, measured by the oxygen electrodes, were scaled by corresponding amplitude at zero frequency.

In the third step the frequency characteristics were processed by the least-square technique to evaluate parameters of the selected models. Both transfer functions were used simultaneously in this process.

Results and discussion

Preliminary theoretical analysis of the problem indicated improved ability of the experiment for parameter evaluation for the case of co-current flow arrangement in contrast to the counter-current flow.[8] Practical work, unfortunately did not confirm this expectation. Processing of co-current data proved more difficult because, among other things, experimental values of liquid and gas holdups obtained by an independent method were not available for co-current flow arrangement.

The transfer function X_{GZ}/X_{G0} served to evaluate gas holdup and total liquid holdup that were constrained by the condition that their sum must equal the void fraction (0.4). The results may thus be affected by the fidelity of the model of the liquid flow. In real conditions part of the liquid is kept as a "stagnant phase" while the dynamic measurements respond to the dynamic portion of the liquid phase.

Figure 2 plots the dependence of the holdup of gas, h_G, as a function of the gas Reynolds with the Reynolds number for liquid as a parameter as it was evaluated from the PF-PF and AD-AD1 models. In accord with the literature h_G decreases with increasing gas flow rate. Gas holdup from our experiments is also seen to decrease with increasing liquid velocity. The evaluated gas holdups do not differ appreciably for the two models used for evaluation.

Figure 3 shows the dependence of liquid holdup h_L on the Reynolds number for liquid for the AD-AD and the PF-PF models together with the correlation of *Rao* et al.[11]. Liquid holdup increases with increasing liquid velocity while it decreases with increasing liquid velocity. The figure also indicates lower h_L obtained from the AD-AD model.
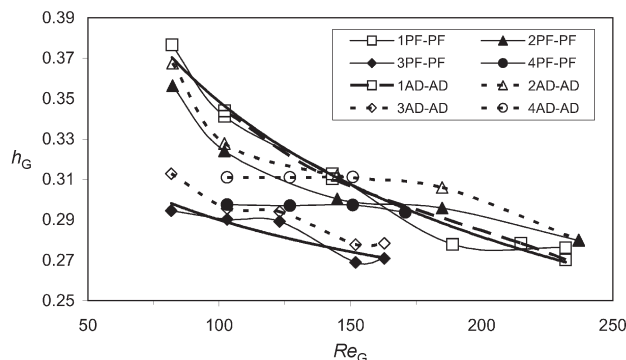

F i g . 2 – *The dependence of h_G on Re_G for the PF-PF and AD-AD1 models: Curve 1) Re_L = 38.6; 2) Re_L = 57.9; 3) Re_L = 124; 4) Re_L = 138*
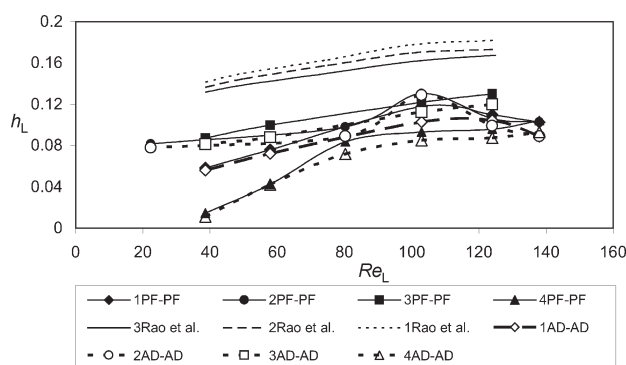

F i g . 3 – *The dependence of liquid holdup, h_L on Re_L for the AD-AD1 and PF-PF models and the correlation of Rao et al.[11] with Re_G as a parameter: Curve 1) Re_G = 103; 2) Re_G = 124; 3) Re_G = 143; 4) Re_G = 164*
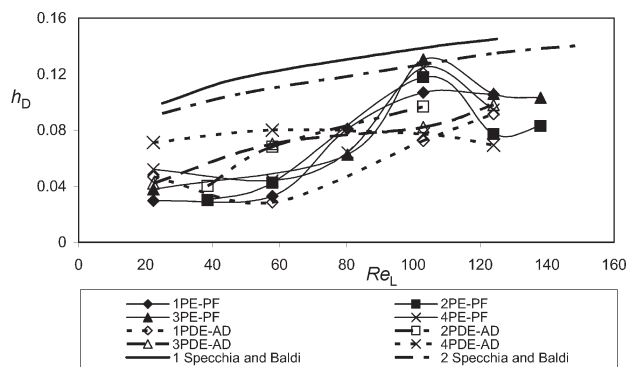

F i g . 4 – *The dependence of the dynamic liquid holdup, h_D on Re_L for the PE-PF1 and PDE-AD models and corelation[4] with Re_G as a parameter: Curve 1) Re_G = 81.9; 2) Re_G = 103; 3) Re_G = 124; 4) Re_G = 164*

Our results are approximately 30–50 % lower than those of *Rao* et al.[11]. The data of *Rao* et.al.[11] were measured on a substantially shorter column with smaller packing particles. A comparison with the work of *Moravec*[8] under the counter-current flow arrangement indicates that total holdups for co-current flow are lower.

Figure 4 plots the dependence of the dynamic liquid holdup, h_D, on the Reynolds number for liq-

uid evaluated from two models. In the calculations we have forced the optimization routine to obey the constraint that the sum of the dynamic and static holdup equals the total holdup:

$$h_D + h_S = h_L$$

From comparison of the PE-PF and PDE-AD models, it follows that at lower liquid rate end (Re_L = 22.4 and 38.6) the model of greater complexity (PDE-AD) yields higher values of dynamic liquid holdup suggesting perhaps the importance of using more faithful models. At higher liquid Reynolds numbers the scatter of results is larger and does not permit preference to be made between the two models. Also plotted results of *Specchia* and *Baldi*[12] are generally higher.

Figure 5 plots the Peclet number in the gas phase, defined as $Pe_G = v_G d_p / h_G E_G$, as a function of the gas phase Reynolds number for the AD–AD1 and the PE-AD model. We note that in the evaluation of axial dispersion in the gas phase the major role is that of the X_{GZ}/X_{G0} transfer function that is affected virtually only by gas phase related parameters. For individual models the values of Pe_G in Figure 5 are seen to differ relatively little. The values of gas phase Peclet number indicate that at low liquid flow rates axial dispersion plays only a minor role. Only at higher Re_L values do the Pe_G drop enough so that axial dispersion becomes important.
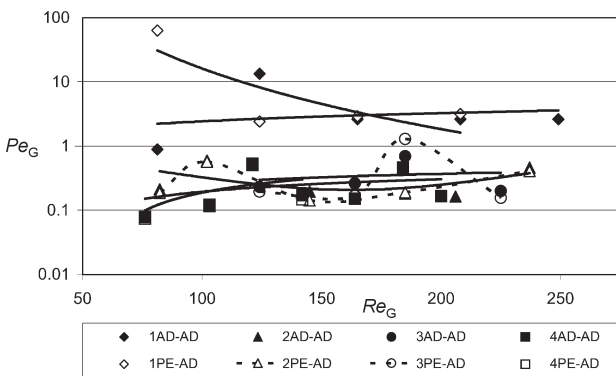

F i g . 5 – *The dependence of Peclet number in gas phase, Pe_G, on Re_G for the AD-AD1 and PE-AD models with Re_L as a parameter: Curve 1) Re_L = 22.4; 2) Re_L = 57.9; 3) Re_L = 80.3; 4) Re_L = 103*

The sensitivity of the gas-to-liquid X_{LZ}/X_{G0} transfer function to axial dispersion coefficient in the liquid phase, E_L or E_D, is substantially lower than that of X_{GZ}/X_{G0} transfer function to the coefficient E_G. Also the fact, that the transfer function X_{LZ}/X_{G0} is affected generally by the flow dynamics of both phases as well as interfacial mass transfer, plays a negative role in affecting the accuracy of parameter evaluation.

The Figure 6 plots the dependence of the Peclet number for the dynamic liquid $Pe_D = v_G d_p / h_D E_D$ and for the total liquid $Pe_L = v_L d_p / h_L E_L$ on the liquid Reynolds number for the AD-AD1 and the PDE-AD models. With increasing liquid rate the values of Pe_D and Pe_L increase while they decrease with increasing gas rate. At gas Reynolds number Re_G = 143 the values of Pe_L and Pe_D become very small and almost independent of liquid velocity; only at higher Re_G display an also increasing trend. With a further increase of gas velocity the results become scattered possibly due to sudden departure from the steady operation observed at high gas and liquid flow rates. These velocities, however, were at the upper extreme limit of operation of our equipment.
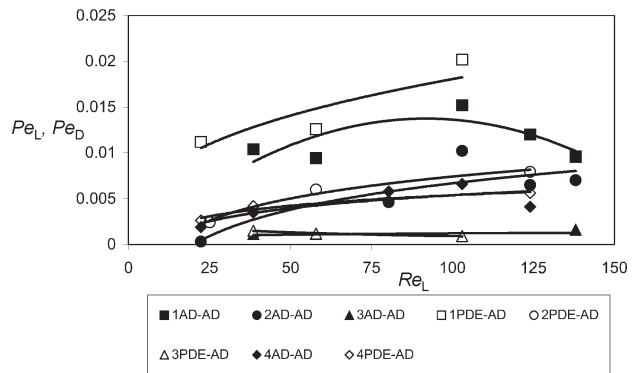

F i g . 6 – *The dependence of the Peclet number in the liquid phase, Pe_L, and in the dynamic liquid, Pe_D, on Re_L for the AD-AD1 and PDE-AD models with Re_G as a parameter: Curve 1) Re_G = 103; 2) Re_G = 124; 3) Re_G = 143; 4) Re_G = 164*

From the definition of the Peclet number it follows that the lower its values the stronger the effect of axial dispersion. The values of Pe_L for the AD-AD1 model are somewhat higher than those for the PDE-AD model and the axial dispersion is therefore felt more strongly. This appears to be in line with the construction of the two models.

An important evaluated parameters are the interfacial volume mass transfer coefficients between the gas and liquid phase, $k_L a$, between the gas and the dynamic liquid phase, $k_{LD} a_D$, between the stagnant and the dynamic liquid phase, q, and between gas and the stagnant liquid phase, $k_{LS} a_S$.

Figure 7 plots the dependence of the volume mass transfer coefficient, $k_L a$, on the gas Reynolds number for the PF-PF and AD-AD1 models. The values of $k_L a$ grow with increasing gas Reynolds number, particularly, at low liquid Reynolds numbers. Increasing trend is also seen with increasing liquid velocity excepting the lowest value at Re_L = 22.4.
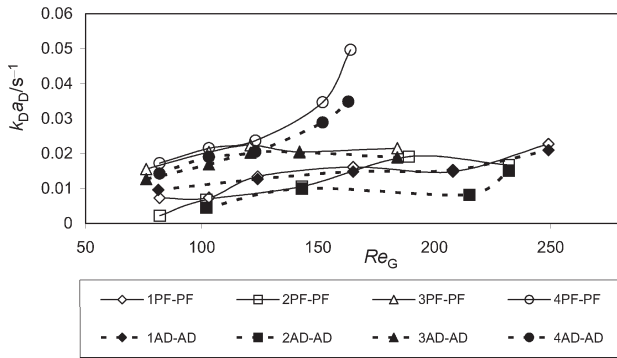
F i g . 7 – *The dependence of the interfacial mass transfer coefficient, $k_L a$ on Re_G for the AD-AD1 and PF-PF models with Re_L as a parameter: Curve 1) Re_L = 22.4; 2) Re_L = 38.6; 3) Re_L = 103; 4) Re_L = 124*
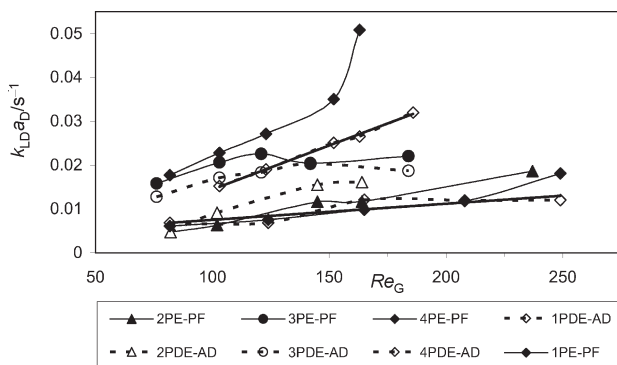

F i g . 9 – *The dependence of the interfacial mass transfer coefficient in the dynamic liquid, $k_{LD} a_D$ on Re_L for the PE-PF1 and PDE-AD models with Re_G as a parameter: Curve 1) Re_G = 81.9; 2) Re_G = 103; 3) Re_G = 124; 4) Re_G = 164*

The values of the mass transfer coefficient, $k_L a$, obtained in this work agree in the order of magnitude with the results of *Moravec*[8] measured on the same equipment at counter-current flow operation. The literature, however, has $k_L a$ correlations[1] that are as much as ten times greater. The mass transfer coefficient, however, depends strongly on the geometry of the packing and is found to be large particularly for small packing dimension. The results presented here appear therefore more realistic for large diameter, large packing columns.

Figures 8 and 9 show the dependences of the mass transfer coefficient in the dynamic liquid, $k_{LD} a_D$, on the gas and liquid Reynolds numbers respectively. The models used for evaluation were PE-PF1 and PDE-AD models. The obtained values of this coefficient are in all cases smaller than $k_L a$ for the AD-AD1 and PF-PF models and exhibit a similar trend as $k_L a$. This means that they strongly increase with increasing gas and liquid velocity. This trend is characteristic for Re_L = 22.4 and 124 and for Re_G = 81.9 and 186. For Re_L = 22.4 are $k_{LD} a_D$ for low gas rates is higher than those for Re_L = 38.6 and 57.9.

The found values of $k_{LS} a_S$ were almost always lower than those of $k_{LD} a$. The coefficient of mass transfer between the dynamic and stagnant liquid, q, was evaluated only for the PE-PF and PE-AD models. In all cases the optimization routine yielded value 0.003 s^{-1} is set as a minimum. Low values of q indicate poor communication between stagnant and dynamic liquid holdup.

Finally, we have developed correlations for the total liquid holdup, h_L, for the dynamic holdup, h_D, for the mass transfer coefficient, $k_L a$, and the gas- and liquid-phase Peclet numbers. The selected forms of the correlations are similar to those existing in papers by *Specchia* at al.[12] and *Hirose* et al.[13] Below are the obtained forms of the correlations with corresponding correlation coefficients.

$$h_L = 0.0485 \left(\frac{Re_L}{Re_G} \right)^{0.610} \qquad \rho = 0.549$$

$$h_D = 0.0322 \left(\frac{Re_L}{Re_G} \right)^{0.699} \qquad \rho = 0.761$$

$$k_L a = 0.0161 \left(\frac{Re_L}{Re_G} \right)^{0.113} \qquad \text{very low } \rho = 0.07$$

$$Pe_G = 2.68 \left(\frac{Re_L}{Re_G} \right)^{-1.73} \qquad \text{low } \rho = 0.29$$

$$Pe_L = 0.0012 \left(\frac{Re_L}{Re_G} \right)^{2.09} \qquad \rho = 0.586$$

In view of the very low correlation coefficient the correlation of the volume mass transfer coefficient may be approximated by:
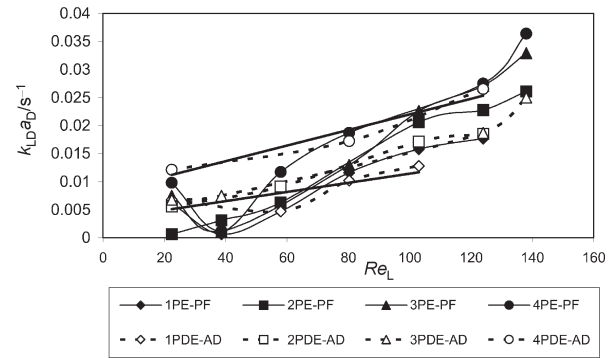
$$k_L a = 0.019 \text{ s}^{-1}$$

F i g . 8 – *The dependence of the interfacial mass transfer coefficient in the dynamic liquid, $k_{LD} a_D$ on Re_G for the PE-PF1 and PDE-AD models with Re_L as a parameter: Curve 1) Re_L = 22.4; 2) Re_L = 57.9; 3) Re_L = 103; 4) Re_L = 124*

Judging from the similar exponent of the ratio of the liquid and gas Reynolds numbers for the total and dynamic holdup one says that the dynamic holdup amounts approximately to:

$$\frac{h_D}{h_L} = \frac{0.0322}{0.0485}\left(\frac{Re_L}{Re_G}\right)^{-0.089} \frac{0.0322}{0.0485} = 0.66$$

that is about 66 % of the total holdup regardless of the flow rates of phases.

Conclusions

The conducted experimental study of absorption of oxygen in water in a co-current column packed with glass spheres has advantages as well as limits of using the frequency characteristic to evaluate parameters of the models presented in an earlier paper.[5]

It turns out that the dynamics of the flow and accompanied interfacial mass transfer may be approximated by the axially dispersed plug flow. The experiments show that axial dispersion is more important in the liquid phase which is in accord with the findings in the literature.

The values of the volume mass transfer coefficient, $k_L a$, found in this study appear by about 10 % lower than those found by *Moravec*[8] on the same column with the same packing but under counter-current flow arrangement. Similar conclusion is valid also for the coefficient $k_{LD}a_D$ characterizing mass transfer between gas and dynamic liquid.

The found values of holdups were determined primarily from the character of the gas-inlet-to-gas-outlet transfer function (X_{GZ}/X_{GL}). Evaluation of some of the liquid phase related parameters proved to be more difficult due to the low sensitivity of the gas-inlet-to-liquid-outlet transfer function to those parameters and due to the limited accuracy of the oxygen electrodes employed.

This is particularly true about the dynamic-to-stagnant liquid mass transfer coefficient, q. This problem could be resolved best by additional dynamic experiments taking place largely in the liquid phase, such as using a tracer in the liquid phase that does not undergo interfacial mass transfer. In view of the low solubility of oxygen in water oxygen functions in the gas phase effectively as a tracer.

The stagnant as well a dynamic liquid holdup were found both to increase with the liquid rate and their ratio was found to be virtually independent of the ratio of liquid and gas Reynolds numbers. The increase of both liquid holdups with the liquid rate agrees with the literature, but direct comparison is difficult, because the data in the literature were obtained on substantially shorter columns and smaller packings.

Major problem for the employed technique of parameter evaluation appears the number of quantities increasing with increasing complexity of the model. Physically sound conditions constraining the optimization routine in an effort to retain maximal physical meaning of the evaluated parameters were found useful. When evaluating parameters of more sophisticated models it also proved effective using values of parameters evaluated on the basis of simpler models.

Also the technical limitations of the employed oxygen polarographic electrode posed a problem. Oxygen electrodes of this type and their development are currently not in the main stream of interest because the probes used in expanding biotechnologies actually measure voltage. Potential electrodes, however, are rather slow for the purposes of a study such as this. In general one can say that the availability of a "fast and accurate" probe is key to the success of transient technique studies.

This work also provided a comparison of counter and co-current flow study by transient technique and the co-current arrangement proved to be more difficult to study.

List of symbols and abbreviations

a – specific interfacial surface of the packing, m^{-1}

d_p – diameter of packing element, m

v_G – superficial velocity of gas, ms^{-1}

E_L, E_D, E_G – axial dispersion coefficient in liquid, dynamic liquid hold up and gas, m^2 s^{-1}

h_D, h_G, h_L, h_S – hold up of dynamic liquid, gas, total liquid and stagnant liquid

k_L – mass transfer coefficient, m s^{-1}

k_{LD}, k_{LS} – mass transfer coefficient related to dynamic (LD) and static (LS) liquid hold up, m s^{-1}

v_L – superficial liquid velocity, m s^{-1}

P – amplitude ratio

Pe_D – Peclet number for dynamic liquid phase, $v_L d_p/h_D E_D$

Pe_L – Peclet number for liquid phase, $v_L d_p/h_L E_D$

Pe_G – Peclet number for gas phase, $v_L d_p/h_G E_D$

q – coefficient of exchange of liquid between dynamic and static liquid, s^{-1}

X – Laplace transform of concentration x

X_{GZ}/X_{G0} – gas outlet to gas inlet transfer function

X_{LZ}/X_{G0} – liquid outlet to gas inlet transfer function

ε – void fraction

ρ – correlation coefficient

AD   – axially dispersed flow

PDE – axially dispersed flow with stagnant zone

PE   – plug flow with stagnant zone

PF   – plug flow

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