WASH-OUT OF SOLID PHASE
FROM THE MECHANICALLY STIRRED CHARGE

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Experimental measurements were performed of the time dependence of solid phase concentration in suspension at the outlet from the apparatus with the mechanically stirred charge. Suspension was being withdrawn continuously with fresh water supplied into the charge. Validity of the theoretical approach proposed earlier has been verified by the performed experiments. It has been demonstrated that the studied effect can be under certain conditions described with sufficient accuracy by a simple material balance of the solid phase in the system.

A simple stochastic model has been proposed recently[1] enabling description of the solid phase concentration distribution in the liquid with respect to the axial coordinate in time, in the flow and mechanically stirred system. The model has been developed first of all for descriptions of the unsteady operation: withdrawal of suspension of solid phase from the in advance fixed point of the charge.

Here an attempt has been made to describe such operation on basis of a simple material balance of the solid phase in the system. In the discussion it is demonstrated that this relation can be reached from the earlier considered stochastic model at certain simplifications and thus that it has a limited validity.

Experimental studies of the flow system formed by the mechanically mixed solid and liquid phases have been performed both in the steady state, in which the mean concentration of suspension has not changed and in unsteady state where the change in concentration of suspension in the vessel with time took place. The wash-out experiments were performed in which pure liquid was introduced into the stirred suspension and the concentration of suspension in the outlet stream was measured in dependence on time.

THEORETICAL

Material Balance of the Two-Phase System

Let us define the separation coefficient by the relation

$$\varrho(P, t) = c(P, t)/\bar{c}(t) . \tag{1}$$

The separation coefficient ϱ at the withdrawal from the point P in the vessel is the ratio of bulk concentration in the outlet pipe situated into the point P in time t, $c(P, t)$ and the mean bulk concentration of suspension in the vessel in the same time $\bar{c}(t)$. In general, the separation coefficient is a function of P, T, speed of the stirrer, particle size, volumetric flow rate of suspension through the vessel, physical properties of compounds and geometry of the vessel. At the use of the same apparatus with the same speed of the stirrer, the same liquid and solid phases, for the case of monodispersion, with the narrow range of particle sizes and for constant flow rate of suspension through the device the separation coefficient is the function of P and t, which can be written

$$\varrho = \varrho(P, t) . \tag{2}$$

For derivation of the balance model is assumed that the value of the separation coefficient is independent of the time

$$\varrho = \varrho(P) \tag{3}$$

and thus, that the separation coefficient in the given point is a constant. Let us assume that the ratio of concentration in the outlet pipe and the mean concentration in the whole vessel is independent of the mean concentration which is changing with time. The balance of the solid phase at its withdrawal from the vessel of volume V can be written in the differential form

$$V \bar{c}(t) - V[\bar{c}(t) + \mathrm{d}\bar{c}(t)] = \dot{V} c(t) \, \mathrm{d}t , \tag{4}$$

where $c(t)$ is the concentration of suspension in the outlet piping in time t. According to Eq. (1) is $c(t) = \varrho \, \bar{c}(t)$ and by substitution into Eq. (4) the relation is obtained

$$-V \mathrm{d}\bar{c}(t) = \dot{V}\varrho \, \bar{c}(t) \, \mathrm{d}t \tag{5}$$

and moreover

$$\frac{\mathrm{d}\bar{c}(t)}{\bar{c}(t)} = - \frac{\dot{V}}{V} \varrho \, \mathrm{d}t . \tag{6}$$

The initial condition is

$$t = 0, \quad \bar{c} = \bar{c}_0, \tag{7}$$

where $\bar{c}_0$ is the mean concentration of suspension in the vessel at the beginning of the experiment in time 0. By use of the mean residence time $\bar{t}$ defined as
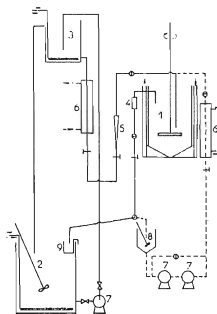
$$\bar{t} = V/\dot{V} \tag{8}$$

Fig. 1

Experimental Apparatus

1 Flow vessel with mechanical mixer, 2 storage tank, 3 overflow vessel, 4 conductivity cell (probe), 5 rotameter, 6 coolers, 7 centrifugal pump, 8 storage tank, 9 sieve. ——— measuring loop, – – – – calibration circuit.
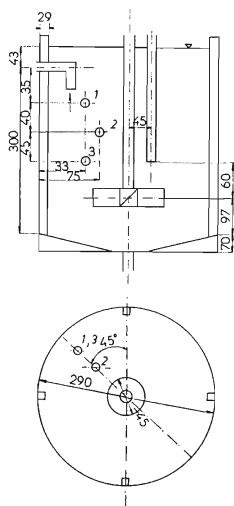
Fig. 2

Flow Vessel with Points of Sample Withdrawal 1—3

the relation is obtained for the change of mean concentration with time in the form

$$\frac{\bar{c}(t)}{\bar{c}_0} = \exp\left(-\varrho t/\bar{t}\right). \tag{9}$$

The dimensionless time τ and dimensionless concentration $C(t)$ are introduced by relations

$$\tau = t/\bar{t}\,; \quad C(t) = c(t)/\bar{c}_0 \tag{10}$$

According to definition of ϱ, at the given assumptions the relation holds

$$\bar{c}(t) = c(t)/\varrho\,. \tag{11}$$

By combination of Eqs (9) to (11) the relation is obtained

$$C(t) = \varrho \exp\left(-\varrho\tau\right). \tag{12}$$

This relation includes only one parameter and is thus especially sensitive to experimental verification.

EXPERIMENTAL

Apparatus. Schematical drawing of the experimental unit is given in Fig. 1. The storage liquid tank 2 of the volume 0.15 m^3 was situated in the lowest part of the apparatus. From it, the liquid was pumped by the centrifugal pump 7 into the overflow vessel 3 of the volume 0.03 m^3 situated in the upper part of the apparatus. By use of this overflow vessel the liquid level was kept constant and through it the constant liquid flow rate was obtained. From the overflow vessel the liquid was delivered through the thermostated cooler 6 and floating flowmeter 5 into the mechanically stirred cylindrical flow vessel 1 with suspension of particles in the liquid. From the flow vessel the suspension was flowing through the outlet piping over the conductivity cell 4 recording its instantaneous concentration, back into the storage tank 2 or 8. At the end of the outlet piping was situated into the liquid tank the sieve 9 separating the solid phase from the liquid. The flow rate of liquid was fixed according to the reading on the rotameter by a valve. The flow rate of the outlet suspension was controlled by the outlet valve so that the constant height of the liquid was kept in the vessel. The liquid tank, overflow vessel and flow vessel were kept at constant temperature. The interconnecting piping was made of glass or polyethylene with the inside diameter 0.015 m.

The flow vessel of diameter 0.29 m was made of polymethyl metacrylate. It was equipped with four baffles equal to $1/10$ of the vessel with conical bottom. The liquid inlet was situated in the vessel closely to the stirrer so that the fresh liquid was quickly spread over the whole bulk of the charge. The outlet piping was situated on the opposite side of the stirrer toward the liquid surface. The six-blade mixer with inclined paddle blades of diameter $d = D/2$ was used for mixing of the suspension. The mixer was situated in the axis of symmetry in one third of the height of the charge above the conical bottom and was driven by a direct-current motor with continuous control

of speed of rotation (Fig. 2). Conductivity probe was formed by a flow vessel of square cross section $1\cdot8 \cdot 10^{-4}$ m^2 made of polymethyl metacrylate equipped with two plane Pt electrodes having the dimensions $1\cdot2 \cdot 10^{-2} \times 3 \cdot 10^{-2}$ m in the distance $1\cdot5 \cdot 10^{-2}$ m apart. The conductivity cell formed the measuring branch of the measuring instrument with the transformer bridge and with the phase sensitive detector. Detailed description of the measuring instrument and of its function is given in another article[2]. To the outlet of the conductivity probe is connected the line recorder which recorded the instantaneous value of concentration of suspension. The conductivity probe had to be calibrated for each fraction of particles. For this purpose the measuring circuit was used with simple rearrangements. The calibration loop is given in Fig. 1 by a dashed line. Suspension leaving the vessel 1 (at the rotating mixer) has passed through the conductivity probe 4, recording its concentration through the three-way cock enabling sampling into a small polyethylene vessel 8, separating suction of pumps 7 of the calibration loop from the conductivity probe. This vessel served as the storage tank for suspension in the moment of sample withdrawal. The suspension was pumped from the vessel by two in a series connected centrifugal pumps 7 back into the flow vessel.

Measuring procedure. The recorder was calibrated so that the resistance decade was connected into the measuring branch of the transformer bridge of the conductivity cell instead of the probe. The value of the resistance 300 Ω was fixed on the compensation resistance decade and the bridge was reset by fixing the resistance of the measuring decade. The deflection of the recorder V^+ has changed by increasing the resistance on the measuring decade. The result of calibration of the recorder was the relation between the relative conductivity G_p (which is the ratio of resistances fixed on the measuring decade to the resistance fixed on the compensation decade) and the deflection of the recorder V^+ in the form

$$G_p = 1 + k\,V^+ . \tag{13}$$

Calibration of the probe means determination of constants of the dependence between the relative conductivity G_p and concentration of suspension c as given by the relation

$$G_p = a + bc . \tag{14}$$

The calibration loop was filled with conductive liquid (solution of NaCl in water) and its temperature was kept at 25°C by the thermostate. As the used conductivity measuring instrument includes the transformed bridge, it is linear only in a narrow range of resistances of the probe. It was necessary at the given temperature to alter the conductivity of the electrolyte so that the bridge was always equalized at the same value of the resistance of the compensation resistance decade. The value of the resistance 300 Ω ± 10% was selected which, at the used measuring instrument, represents conductivity of solution 0·15 kgm^{-3} Na Cl at 25°C.

Solid particles were then added into the electrolyte and suspension was stirred. At the outlet from the flow vessel was situated a sieve and the calibration loop had been switched on so that pure electrolyte was flowing through the loop. The bridge was set to zero and the zero was set on the recorder. After removal of the sieve, suspension started to flow through the loop and on the recorder a deflection appeared proportional to the concentration of the flowing suspension. After steadying of the records a sample of suspension has been taken in which the concentration of the solid phase in the liquid was determined by weighing. Then the liquid was again returned back into the loop which decreased concentration of suspension in the vessel and the whole process was repeated. For determination of one calibration dependence 10 measurements were always performed with the range of concentrations of suspension being in the range from 1 to 10% by mass.

For measurement of separation coefficients the calibration loop was used. The known amount of liquid was poured into the calibration loop and then the weighed amount of solid particles has been added. The measuring procedure has been the same as with the calibration only no samples of suspension were withdrawn. From deflection of the recorder, proportional to the solid phase concentration in the liquid the corresponding concentration was determined by use of the callibration relation. The separation coefficients were measured for all experimental conditions at which the individual wash-out curves were measured.

Before the measurements of wash-out curves it was necessary to prepare the measuring loop. The liquid tank was filled with water and its conductivity has been approximately fixed by addition of sodium chloride. The electrolyte has been brought to 25°C by a thermostate. The measuring loop was switched on. At the outlet from the flow vessel was fixed the sieve, solid particles were added into the vessel and the mixer was switched on. Pure electrolyte was flowing through the loop. Conductivity of the electrolyte was arranged so that bridge was set to zero while on the compensation resistance decade was fixed resistance of about 300 Ω. The zero on the bridge has been recorded. Experimental conditions were set and by removal of the sieve started the proper wash-out experiment which lasted for about 10 min. The recorder recorded value proportional to the concentration of suspension flowing through the probe. After termination of the experiment the sieve was again fixed at the outlet from the flow vessel and the zero position of the bridge has been recorded. The washed-out solid particles were returned back into the flow vessel and the loop was ready for another measurement.

Experimental. For measurement of wash-out curves solid spherical particles of two materials differing by densities were used: particles of Koplen 114 with the density 1043 $kg\ m^{-3}$ and particles of glass ballotine with the density 2636 $kg\ m^{-3}$. With both materials two fractions

TABLE I
Results of Selected Experiments

d_p, mm	Material	n, s^{-1}	$\dot{V} \cdot 10^4$ $m^3\ s^{-1}$	Location of sample withdrawal	ϱ_1	ϱ_2	ϱ_3	ϱ_4
2·00—2·05	polystyrene	3·33	1·667	1	0·82	0·84	0·83	0·91
2·00—2·05	polystyrene	3·33	1·167	1	0·73	0·74	0·76	0·87
2·00—2·05	polystyrene	1·83	1·167	3	1·01	0·91	0·98	0·98
1·05—1·20	polystyrene	1·83	1·167	1	0·75	0·68	0·81	0·81
1·05—1·20	polystyrene	3·33	1·667	2	0·84	0·82	0·70	0·87
1·05—1·20	polystyrene	3·33	1·167	2	0·82	0·71	0·78	0·85
0·16—0·18	ballotine	5·00	1·667	2	0·69	0·68	0·53	0·66
0·16—0·18	ballotine	3·33	1·667	1	0·18	0·16	0·22	0·14
0·16—0·18	ballotine	3·33	1·167	3	0·73	0·70	0·59	0·76
0·16—0·18	ballotine	5·00	1·167	2	0·67	0·65	0·52	0·71
0·10—0·125	ballotine	3·33	1·167	1	0·39	0·36	0·33	0·41
0·10—0·125	ballotine	5·00	1·667	3	0·98	0·94	0·70	0·90
0·10—0·125	ballotine	3·33	1·667	1	0·38	0·32	0·27	0·37

of particles sizes were used. Individual experimental conditions at which the experiments were performed were: Particle density $1\,043$ kg m^{-3}; $d_p = 1.05—1.2 \cdot 10^{-3}$ m and $2.0—2.05 \cdot 10^{-3}$ m; $\dot{V} = 1.167 \cdot 10^{-4}$ and $1.667 \cdot 10^{-4}$ m^3 s^{-1}; $n = 1.83$ and 3.33 s^{-1}. Particle density $2\,636$ kg m^{-3}; $d_p = 0.1—0.125 \cdot 10^{-3}$ m and $0.16—0.18 \cdot 10^{-3}$ m; $\dot{V} = 1.167 \cdot 10^{-4}$ and $1.667 \cdot 10^{-4}$ m^3 s^{-1}; $n = 3.33$ and 5.0 s^{-1}. Location of points of sample withdrawal is given in Fig. 2. Initial concentration of solid phase in the liquid was about 4 vol.%. Temperature during the experiments was in the range from 24.5 to $25.5°$C.

In measurements with particles of ballotine small improvements of the apparatus have been made in comparison with those performed with particles of polystyrene. In measurements with the polystyrene particles, the probe has been situated in the vertical position as given in Fig. 1. In measurements with ballotine the probe has been situated in the horizontal position, while between it and the outlet hole for the sample withdrawal from the vessel the static stirrer of the KENICS type was situated. So the homogeneity of suspension at the passage through the probe has been secured.

RESULTS

The detailed procedure of evaluation of experimental data is given in another article[3]. In Table I some final results are given of experiments at selected experimental conditions. As the most illustrative we consider comparison of separation coefficients obtained from the stochastic model, from the balance model with the independent values directly measured. The value ϱ_1 in Table I is the separation coefficient obtained from the stochastic model according to Eq. $(I-35)$.* In the next column the values ϱ_2 are the separation coefficients obtained from Eq. (12) of the balance model from the intercept and ϱ_3 are obtained from Eq. (12) from the slope of straight line. The values were obtained by linear regression of the logarithmic form of equation. The values ϱ_4 are the separation coefficients obtained experimentally by the procedure described in the experimental part of this study.

Typical examples of the wash-out curves are given in Figs 3 and 4 *i.e.* the dependence of the dimensionless concentration C on dimensionless time θ in log coordinates.

DISCUSSION

Earlier, than the proposed model assumptions are analyzed on basis of experimental data it is demonstrated that Eq. (12) which has been derived on basis of a simple material balance is the partial result of a more general model, proposed in our last study[1]. It resulted, that the dependence of concentration of the solid phase in the point of withdrawal in dependence on time can be described by Eq. $(I-33)$ which is the infinite sum of exponentials with the dimensionless time θ as the independent variable. It also resulted, that for sufficiently small liquid flow in rates the experimental device and for sufficiently high speeds of stirrer it has been possible to take

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* To differentiate the numbering relations from the last study[1] to which we refer here, the symbol I is added.

the separation coefficient, defined by Eq. $(I-35)$, as the constant and to neglect all terms of the sum with the exception of the first one (Eq. $(I-36)$).

It is obvious that the definition of the separation coefficient (I) and definition $(I-35)$ in dimensionless coordinates are equal as long the made assumptions are accepted[1], first of all the assumption that the solid phase concentration in the vessel is not changing in the radial direction.

It remains to prove identity of relations $(I-36)$ and (12). It is necessary to realize that the dimensionless time θ in study[1] is related to the time of particle fall, while the dimensionless time τ to the mean residence time in the stirrer. By arrangement of Eq. $(I-5a)$ the relation is obtained for the dimensionless time θ in the form

$$\theta = \frac{t\omega}{D} \cdot \frac{S_n}{S_n} \cdot \frac{S_t w}{S_t w}, \tag{15}$$

Fig. 3

Outlet Concentration in Dependence on Dimensionless Time

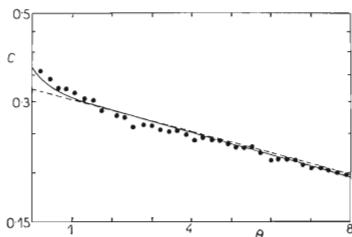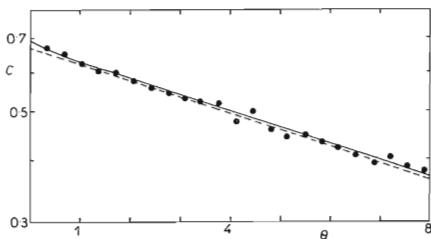——— Stochastic model; – – – – balance model; ● experimental values.

Fig. 4

Outlet Concentration in Dependence on Dimensionless Time

——— Stochastic model; – – – – balance model; ● experimental values.

where ω is the terminal particle velocity, D liquid height in the unit, S_n cross-sectional area of the unit, S_t cross-sectional area of the pipe and w suspension outlet velocity. The product $D . S_n = V$ obviously represents the volume of the vessel and the product $S_t w$ volumetric flow rate through the vessel. With regard to Eqs $(I-10)$ and (8) the relation is obtained

$$\theta = t \frac{\dot{V}}{V} . \frac{1}{M} = \frac{\tau}{M} . \qquad (16)$$

By substitution of this relation Eq. $(I-36)$, the relation (12) is obtained.

The assumption on independence of the separation coefficient of time is thus justified only at sufficiently low liquid flow rates and sufficiently high speeds of rotation of the mixer. This is the case when stirring in the vessel is so intensive that it suffices to compensate practically immediately disturbances (relative) in concentration distribution of the solid phase in the vessel which is the result of its steady withdrawal.

From Fig. 3 is clearly visible that the wash-out curve has not the exponential form at the beginning, thus the simplified model model cannot be used. In the sum $(I-33)$ also other terms than the first one, are obviously significant, while with increasing time the effect of these additional terms decreases. This is also in agreement with the proposed equation since, as is obvious from Fig. $(I-2)$, the roots a_1 in Eq. $(I-33)$ are increasing with the increasing index, thus the value of decrement in the exponential is also increasing.

Under different conditions (Fig. 4) the effect of other terms is so small that it can be neglected practically from the beginning. In this case it is obviously possible to use for description of such a dependence Eq. (12) resulting from the material balance. This fact is confirmed from Table 1 in which the separation coefficients ϱ_1 and ϱ_2 in some cases significantly differ *i.e.* the extrapolated initial concentrations at the beginning of withdrawal by use of Eq. $(I-33)$ or from the material balance (Eq. (12)). The statistical tests have demonstrated that on the 95% level of significance, the values ϱ_1 and ϱ_2 have differed in 13 cases out of 41 testing experiments. This means that approximately only in two thirds of cases it has been possible to describe the whole dependence of the wash-out curve with the sufficient accuracy of the exponential dependence.

It is also significant that the values ϱ_4 of separation coefficients obtained by direct measurements were in 11 out of the above given cases, closer to values ϱ_1, so the more complicated model describes better the actual situation.

Thus there exist the experimental conditions at which it is not possible to consider the simple exponential shape of the wash-out curve which is in contradiction with the results of Baldi and Conti[5]. The authors even accept this dependence as the basic postulate in their theoretical considerations. It is obvious that this postulate is equi-

valent to the assumption of independence of the separation coefficient on time made in this study. Thus their results obviously hold only with regard to the above made restrictions.

The symbol K in the quoted study[5] is identical with the separation coefficient ϱ which we have defined. But similarly as the quoted authors, we have found a relatively good agreement of the separation coefficient ϱ_2 and the values ϱ_3 calculated from the slope of the wash-out curve in semilog coordinates. But the objective statistical tests have demonstrated that in about 80% of cases these values significantly differ on the 95% level of significance. From this fact the conclusion can be made that the over-all material balance gives only an approximate description of the considered operation.

But as long as we have considered the quantities ϱ_1 and ϱ_2 as two parameters, the description of the wash-out curves obtained by use of the exponential dependence was sufficiently accurate (i.e. on the 95% level of significance) in about 67% of cases; by use of Eq. $(I-33)$ in 42 out of 43 studied cases.

The material balance of the solid phase in the system has been checked several times similarly as by Baldi and Conti[5], and no residual trapped mass has been found, which could not be removed from the system. At conditions of our experiments the conclusion by Bourne and Sharma[6] has thus not been confirmed. Though, it is possible that at low speeds of rotation i.e. lower than critical[7] such phenomenon takes place, but according to our opinion, Bourne and Sharma have uncorrectly explained deviations of experimental results from perfect mixing (i.e. differences of the mean residence time of liquid and mean residence time of the solid phase in the vessel in the terminology of Baldi and Conti[5]). The value of separation coefficient ϱ in Eq. (12) for perfect mixing must be obviously equal to one. Bourne and Sharma have thus compared the wash-out curves with the unit value of ϱ with the experimentally obtained dependence for separation coefficients smaller than one.

Fig. 5

Separation Coefficient in Dependence on Vertical Coordinate y

- - - - Dependence given by Baldi and Conti; ○ our values, ballotine, $d_p = 0.10$ to 0.125 mm, $n = 3.33$ s^{-1}, ● our values, ballotine, $d_p = 0.10$—0.125 mm, $n = 5.00$ s^{-1}, ◓ our values, ballotine, $d_p = 0.16$—0.18 mm, $n = 3.33$ s^{-1}, ◒ our values, ballotine, $d_p = 0.16$—0.18 mm, $n = 5.00$ s^{-1}.
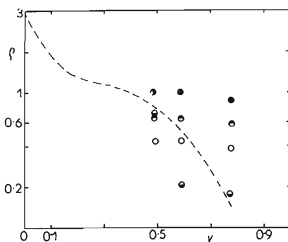
A considerably worse agreement with the experiments has been obtained with the model in the case of the spacial concentration distribution of the solid phase in the charge. Though we do not have at present a sufficient evidence in this respect, it is possible to consider — also with regard to the experiments by Baldi and Conti[5] — that the proposed model is not expressing the situation with sufficient accuracy (Fig. 5). According to relations $(I-14)$, the dependence of the separation coefficient on the dimensionless height coordinate should be linear with the negative slope. Even if it were necessary to study experimentally the whole space of the charge it is possible to state that the postulates concerning the uniform concentration distribution of the solid phase in the horizontal plane and the homogeneous turbulent intensity in the whole charge made in the last study[1] are oversimplifying the whole situation and need to be improved. In this respect there obviously remains open the question of the effect of operating conditions on parameters of the model or on the separation coefficients. With the exception of variables whose effect has been studied by Baldi and Conti it would be possible to except also a significant effect of the speed of rotation of the mixer. This assumption has been confirmed as is obvious from Table I or Fig. 5.

CONCLUSIONS

Experimental results have been compared with the conclusions made on the basis of a model describing the random motion of particles in the mixed charge[1]. The proposed relation enables a very good description of the solid phase wash-out from the charge in the whole range of the time variable. Description of spacial distribution of the solid phase is imperfect and the proposed model needs additional improvements.

Conditions were clarified at which this procedure could be described by an exponential relation and it was demonstrated that this relation resulted from a simple material balance of the system.

LIST OF SYMBOLS

a constant in Eq. (14)
b constant in Eq. (14), $(m^3 \, kg^{-1})$
C dimensionless concentration
c volume concentration, $(kg \, m^{-3})$
c_0 volume concentration in time $t = 0$, $(kg \, m^{-3})$
$\bar{c}$ mean volume concentration in the vessel, $(kg \, m^{-3})$
$\bar{c}_0$ mean volume concentration in the vessel, in time $t = 0$ $(kg \, m^{-3})$
D height of liquid in the vessel, (m)
d_p diameter of particles, (m)
G_p relative conductivity
k constant in Eq. (13), (m^{-1})
M dimensionless parameter in Eq. (16)
n speed of rotation of the stirrer, (s^{-1})

P location of the point of suspension withdrawal in the vessel
S_n cross sectional area of vessel,   (m^2)
S_t cross sectional area of the outlet pipe,   (m^2)
t time,   (s)
$\bar{t}$ mean residence time,   (s)
V volume of suspension in the vessel,   (m^3)
$\dot{V}$ volumetric flow rate of suspension,   (m^3 s^{-1})
V^+ deflection of the recorder,   (m)
w flow rate of suspension from the vessel,   (ms^{-1})
ϱ separation coefficient
ϱ_1 separation coefficient calculated from the stochastic model
ϱ_2 separation coefficient calculated from the intercept on basis of the balance model
ϱ_3 separation coefficient calculated from the slope on basis of the balance model
ϱ_4 separation coefficient obtained experimentally
θ dimensionless time related to the mean time of particle fall in the equipment
τ dimensionless time related to the mean residence time of suspension in the vessel
ω terminal particle velocity,   (m s^{-1})

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