

PRESSURE DROP IN ELECTROCHEMICAL CELL WITH FIXED BED

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Experiments were performed to study hydrodynamic characteristics of three-phase system (Solid-Liquid-Gas) in a cylindrical cell, where a gas phase (hydrogen) was electrochemically generated. Two-phase fluid (gas-liquid), in which an electrolyte was a continuous one, flowed through the fixed bed, packed of glass particles ($d=3$ mm, $d=2$ mm, $d=1.5$ mm). What was examined was the influence that electrolyte velocity, current density and particles diameter had on the relative pressure drop ($\Delta P/\Delta P_0$) through fixed bed. It was found that with velocity increase of electrolyte flow ($w=0.00587-0.03$ m/s), the relative pressure drop decreased through the fixed bed. At higher electrolyte velocity (0.03 m/s), fluidisation appeared, whereas at a little lower electrolyte velocity of 0.03 m/s, gas channels, which form through the bed, enable quicker gas flow and also a lesser pressure drop. With the current density increase, pressure drop increased, since greater gas quantities stayed behind in the fixed bed. Besides, it was also found, that relative pressure drop decreased with diameter decrease of glass particles, which formed the bed.

Keywords: pressure drop, electrolyte velocity, gas, electrochemical cell, gas-liquid-solid, fixed bed, fluidisation

Introduction

Electrochemical reactors with a fixed bed (gas-liquid-solid particles) are used very often in industry, in chemical and biochemical processes, in electrometallurgy as well as in processes of environment protection [1,2,3]. Besides, characteristics of the three-phase system were examined in reactor engineering, especially in the part which refers to hydrogenation [4-8]. Because of that it is good to know the hydrodynamic characteristics of two-phase gas-electrolyte fluid through a fixed bed. Hydrodynamic characteristics of two-phase flow were examined through the fixed bed, in which electrolyte is a continuous phase [9-12]. The following can influence hydrodynamic characteristics: electrolyte velocity, particle size constituting the bed, gas hold-up, geometrical characteristics of the column and so on. What was examined in this work were hydrodynamic characteristics of two-phase gas-electrolyte flow through a fixed bed of spherical particles, where gas phase had been generated electrochemically. During electrolysis of water, oxygen evolved on the anode, whereas hydrogen evolved on the cathode. At the same time, when the cathode surface developed, formation of the nucleus for hydrogen bubbles rising occurred all over the surface. In such a way, the obtained bubbles are

smaller than in the case when gas was introduced into electrolyte through a distributor.

Experimental

The experiments were carried out on the experimental set-up, which is schematically presented in *Fig.1*. Vertical cylindrical column with the inner diameter of 45 mm and 320 mm in height, was made of Plexiglas. Platinum net was built in the column, being at the same time a current feeder, the bed bearer and the electrolyte distributor as well. The wire, which made the net, was 0.16 mm thick, and the number of meshes per square centimeter is 378. The total surface of the cathode was $3.154 \cdot 10^{-2}$ m².

The height of the packed bed of particles through which pressure drop was measured was 50 mm. The anode was placed above the bed, as it is marked in *Fig.1*. It is made of lead sheet having cylindrical form with an outer diameter of 45 mm, height of 30 mm and thickness of 1 mm. The distance between the cathode and the anode was 63 mm. Because of the considerable distance between the cathode and the anode, a solution of sulphuric acid was used with the concentration of $3 \cdot 10^3$ mol/m³ H₂SO₄ and it had the greatest electric conductivity. Electric conductivity of this solution is

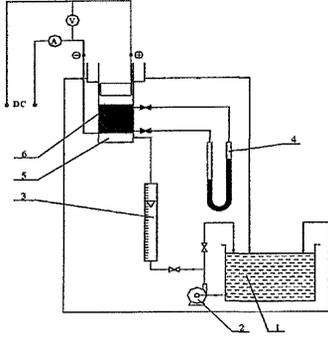


Fig.1 Experimental set-up: 1 - reservoir; 2 - centrifugal pump; 3 - flowmeter; 4 - differential manometer; 5 - column; 6 - fixed bed

73.88 S/m [13]. The power supply of direct current is a rectifier of alternating current equipped with voltmeter and ammeter.

Three fractions of glass particles 1.5 mm, 2 mm and 3 mm in diameter, were used as a dispersed phase.

All experiments were carried out at ambient temperature. The total volume of liquid phase for all the experiments was the same, $5 \cdot 10^{-3} \text{ m}^3$. The electrolyte was transported by a centrifugal pump, from the reservoir through a flowmeter into the column from the bottom upwards, so that the electrolyte passed through the platinum net cathode and carried gas bubbles evolving on the cathode. From the top of the column, the electrolyte returned to the reservoir overflow. Both the column and the reservoir were open to the atmosphere so that the gas phase, which was hold-up in the electrolyte, went freely to the atmosphere.

A differential "U-tube" manometer filled with carbon tetrachloride measured pressure drop through the bed whose density was 1586.7 kg/m^3 .

The procedure of performing the experiment went this way: firstly, in the column above the platinum grid electrode - bed support, an amount of $0.158 \cdot 10^{-3} \text{ m}^3$ of glass particles was introduced. An electrolyte of the concentration $3 \cdot 10^3 \text{ mol/m}^3 \text{ H}_2\text{SO}_4$ was put into the reservoir. After that, centrifugal pump was switched on, so when a constant flow was achieved, then pressure drop (ΔP_0) through the bed was measured. Then both the power supplier and a chronometer were switched on. On the platinum cathode, the evolution of hydrogen bubbles started up influencing pressure drop (ΔP) increase through the bed. This change was monitored every two minutes until it reached constant value. The centrifugal pump was still on, pumping the electrolyte, in order to remove the remained bubbles out of the bed. The system was then ready for the next experiment.

Results and Discussion

The influence of electrolyte velocity

When current is on at the platinum cathode, then there is electrolysis of water and also evolution of gas bubbles

(hydrogen), which are carried by liquid flow upwards through fixed bed, in which case hold-up of each phase is:

$$\varepsilon_s + \varepsilon_l + \varepsilon_g = 1 \quad (1)$$

Solid hold-up in fixed bed is constant, so the voidage of bed ε , is equal to the sum of liquid and gas hold-up.

$$\varepsilon = \varepsilon_l + \varepsilon_g = 1 - \varepsilon_s \quad (2)$$

that is the liquid hold-up is:

$$\varepsilon_l = \varepsilon - \varepsilon_g \quad (3)$$

If Ergun's equation [14] is modified:

$$\frac{\Delta P_0}{L} = 150 \cdot \frac{(1 - \varepsilon)^2 w_l \mu_l^*}{\varepsilon^3 d^2} + 1,75 \cdot \frac{(1 - \varepsilon) w_l^2 \rho_l^*}{\varepsilon^3 d} \quad (4)$$

with physical characteristics of two phase flow of gas-liquid. Larachi et al. interpret two phase flow by the emulsion theory [3]. Thus, dynamic viscosity of gas-liquid emulsion is given by Einstein's equation:

$$\mu_l^* = \mu_l [1 + 2,5(1 - \varphi_g)] \quad (5)$$

According to the same principle two phase flow density is defined:

$$\rho_l^* = \varphi_g \cdot \rho_g + (1 - \varphi_g) \cdot \rho_l \quad (6)$$

where φ_g is a volume part of gas in two phase flow:

$$\varphi_g = \frac{V_{lg}}{w_l A_p} = \frac{IM}{Fz\rho_g w_l A_p} \quad (7)$$

Thus, modification of Ergun's equation is given for two phase flow of gas-liquid:

$$\frac{\Delta P}{L} = 150 \cdot \frac{\varepsilon_l^2 w_l \mu_l [1 + 2,5(1 - \varphi_g)]}{(\varepsilon - \varepsilon_g)^3 d^2} + 1,75 \cdot \frac{\varepsilon_l^2 w_l^2 [\rho_g \varphi_g + (1 - \varphi_g) \rho_l]}{(\varepsilon - \varepsilon_g)^3 d} \quad (8)$$

This equation shows that voidage is decreased for a part of the space filled with gas bubbles. If bed voidage is decreased for gas hold-up in bed ($\varepsilon - \varepsilon_g$), then pressure drop increases for two phase flow, which is experimentally proved.

If Eqs.(8) and (4) are divided, we get the influence of gas phase on relative pressure drop:

$$\frac{\Delta P}{\Delta P_0} = \frac{150 \cdot \frac{\varepsilon_l^2 w_l \mu_l [1 + 2,5(1 - \varphi_g)]}{(\varepsilon - \varepsilon_g)^3 d^2} + 1,75 \cdot \frac{\varepsilon_l^2 w_l^2 [\rho_g \varphi_g + (1 - \varphi_g) \rho_l]}{(\varepsilon - \varepsilon_g)^3 d}}{150 \cdot \frac{\varepsilon_l^2 w_l \mu_l}{\varepsilon^3 d^2} + 1,75 \cdot \frac{\varepsilon_l^2 w_l^2 \rho_l}{\varepsilon^3 d}} \quad (9)$$

The influence of electrolyte velocity was examined in such a way that the velocity varied in the interval of 0.003 m/s - 0.03 m/s, at the constant current density. Two series of experiments were performed, current density of 100 A/m^2 being used in the first series, whereas current density of 50 A/m^2 was used in the second series. Glass particles of 2 mm in diameter were used. The results are shown in Fig.2-4.

Fig.2 shows as the electrolyte velocity decreases, a relative pressure drop increases (for $w =$

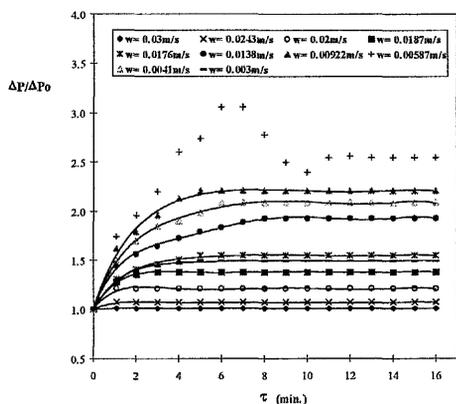


Fig.2 Dependence of relative pressure drop on time for different electrolyte velocities (current density $i=100 \text{ A/m}^2$ and diameter of glass particles $d=2 \text{ mm}$)

0.0058 - 0.03 m/s). The curves which show the change $\Delta P/\Delta P_0$ against time, have two parts: the first part matching unsteady state in which $\Delta P/\Delta P_0$ increases with time, and the second part matching steady state in which pressure drop changes slightly with time and can practically be considered as constant. Unsteady state only takes several minutes and lasts shorter when velocities of electrolyte are higher.

Fig.2 was obtained by measuring pressure drop for electrolyte velocity of 0.00587 m/s, has a characteristic form. A peak is observed on this curve, which can indicate that at a low electrolyte velocity the buoyancy force is low, so as a result, large quantities of gas phase are hold-up in the bed. Coalescence of bubbles also was observed. The bubbles, which become big in such a way, going through a bed of particles, disturb the structure of the bed, since they make channels in order to pass through. At the moment of taking those bubbles away, pressure drop decreases in the bed of particles, all of which lasts four minutes. The other fluctuations of the pressure are inconsiderable in the stationary part of two-phase fluid flow and take less time. The changes of pressure during two-phase fluid flow upward, where electrolyte was a continual phase through a fixed bed of glass particles, were noticed [11]. In their work, the fluctuations of the pressure are considerably higher and range within $\pm 20\%$ of some constant values for the liquid velocity of 0.0068 m/s and gas velocity which is 0.0132 m/s. The time change of pressure drop at parallel gas-liquid flow through a fixed bed was examined by Krieg et al [16]. For the same size of the glass particles, which made the bed, they noticed the similar changes of the pressure that was also observed in this work.

The obtained results can be explained in the following way: by switching the current circuit on, a constant current density of 100 A/m^2 is released through the electrolyte which leads to the electrolysis of water and evolution of hydrogen, forming a two-phase fluid which consists of a continual liquid phase and gas bubbles. The evolved hydrogen, carried by the electrolyte, go through a bed with a certain quantity, and the rest of the hydrogen is captured in the

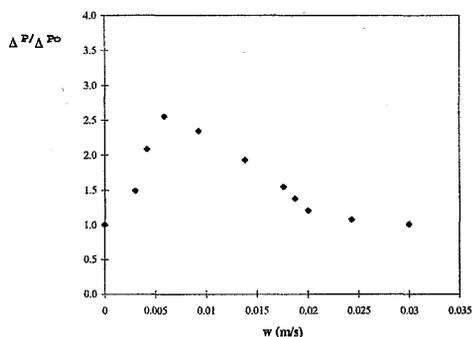


Fig.3 Relative pressure drop values vs. electrolyte velocity (diameter of glass particles $d=2 \text{ mm}$ and current density $i=100 \text{ A/m}^2$)

channels formed by glass particles. The hydrogen, which remains in the bed causes the decrease of the cross-sectional area through which electrolyte may flow, increasing the electrolyte flow resistance that is manifested by increased pressure drop. The more enlarged the quantity of the hydrogen, which was trapped in the bed, the bigger is the fluid flow resistance. At the same time this results in the increase of pressure drop through the bed, with the increase of the volume of hydrogen in the bed. In Fig.2 it is shown that only in the first few minutes the content of hydrogen in the bed increases (unsteady state). For each rate of electrolyte flow, there is a maximum quantity of hydrogen in the bed and that quantity defines the plateau value of the pressure drop. It was noticed that with the increase of electrolyte velocity, the change of relative pressure drop through the bed decreases. This means that at lower velocities of electrolyte in the bed, there is a higher hold-up of hydrogen bubbles, which brings a pressure drop increase. At higher electrolyte velocities buoyancy forces increase which gets hydrogen bubbles carried away from the bed. Higher velocities of electrolyte have a higher kinetic energy, and carrying away hydrogen bubbles from the bed more easily, the resistance to electrolyte flow decreases, i.e. at these conditions the change of the relative pressure drop is smaller. It can also be seen that at higher electrolyte velocities, relative pressure drop does not change significantly with time. At these velocities, the appearance of fluidisation has been remarked, which leads to a decrease of total resistance of electrolyte velocity through the bed, and thus the fluidisation state brings a decrease of pressure drop. This phenomenon was described by Stankovic et al. [15] and he has proved that at fluidisation state relative pressure drop is less than one.

In Fig.3 the dependence of a relative pressure drop against electrolyte velocity is shown. The presented values for $\Delta P/\Delta P_0$ are those ones corresponding to the steady state relative pressure drop obtained at different electrolyte velocities.

As one can see, the relative pressure drop increases with the increase of the velocity of two-phase fluid flow in the fixed bed (1st region). This indicates that, for low electrolyte velocity (0.003 and 0.0042 m/s), coalescence is reduced. At electrolyte velocity of 0.00587 m/s,

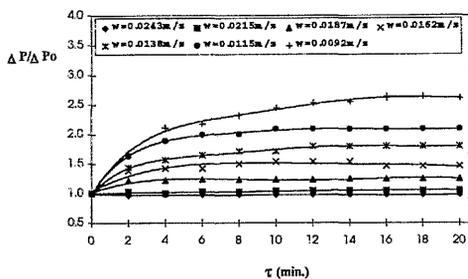


Fig.4 Dependence of relative pressure drop on time for various electrolyte velocities (diameter of glass particles $d=2$ mm and current density $i=50$ A/m²)

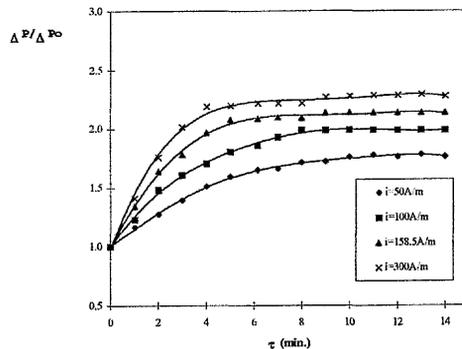


Fig.5 Dependence of relative pressure drop on time for different current densities (diameter of glass particles $d=2$ mm and electrolyte velocity $w=0.0138$ m/s)

pressure drop in the bed is the highest one. After this value, pressure drop in the bed decreases (2nd region). Namely, forming of channels through the bed occurs at the velocities higher than 0.00587 m/s. Through these channels two-phase fluid mainly flows, and so under these circumstances, pressure drop through the bed decreases. At electrolyte velocity of about 0.02 m/s there is not any change of pressure drop with the gas phase, indicating that electrolyte velocity is approached to minimum velocity fluidisation. In the third region, fluidisation in the bed exists, so the higher electrolyte velocity, the more intensive fluidisation of the particles is, and the relative pressure drop value is less than one. Similar behaviour of the pressure drop with the increase of liquid velocity was observed by Chern et al. [9]. Namely, in their work, pressure drop decreases to the liquid velocity of 0.02 m/s, when the bed transforms to a fluidisation state. A slightly lower minimum fluidisation velocity, in their work, is a consequence of less particle density than liquid density (beads made of polypropylene and polyethylene).

The above discussion has been related to the experimental data, which were obtained at current density of 100 A/m². In Fig.4, the dependence of $\Delta P/\Delta P_0$ against time is shown for a cylindrical cell, in which a bed of 2 mm in size is packed of glass particles, and current density used for gas phase generating was 50 A/m².

From Fig.4, it can be concluded that with the increase of electrolyte velocity a relative pressure drop through the bed decreases which points to a less hold-up of gas bubbles in the bed. At constant current density, gas evolution intensity will be constant, too. Increasing in liquid velocity will lead to the increase in kinetics energy of the liquid phase and its buoyancy force, causing a more efficient detachment of bubbles from the particles. Transforming the bed to the fluidised state, relative pressure drop reaches the value equal to one.

The influence of current density

Based on Faraday's laws, the connection between mass of evolved gas in water electrolysis and applied current, is given:

$$m = \frac{M}{z \cdot F} \cdot I \cdot \tau \quad (10)$$

If the mass of the evolved gas, obtained during electrolysis is calculated per unit of time, i.e. a second, then a mass flow rate of the gas is obtained, which, if it is expressed through continuity equation, is the following:

$$G = \frac{m}{\tau} = V_{ig} \cdot \rho_g = w_{ig} \cdot A_k \cdot \rho_g \quad (11)$$

Equating the Eqs.(1) and (2), the velocity of gas evolution may be expressed as follows:

$$w_{ig} = \frac{I \cdot M}{z \cdot F \cdot \rho_g \cdot A_k} = \frac{i \cdot M}{F \cdot z \cdot \rho_g} \quad (12)$$

The influence of current density on pressure drop was examined by performing the experiments in which a change of pressure drop with time was measured at different current densities (50 A/m² - 300 A/m²), and at constant electrolyte velocity. One series of experiments was carried out. In the series glass particles of 2 mm in diameter were used, and the velocity of electrolyte was kept at 0.0138 m/s. The obtained results are shown in Fig.5

In Fig.5 it can be seen that the curves representing a dependence of $\Delta P/\Delta P_0$ on time have similar form to those obtained during investigation of the influence of electrolyte velocity on the pressure drop. On these curves there are also recognizable steady and unsteady parts, where it can be observed that the parts of the curves which correspond to the steady state are almost horizontal and parallel to each other, and the plateau is a translator moved towards the vertical axis. The unsteady state parts of these curves have a higher slope for higher current densities.

With the increase of current density, the change of relative pressure drop also increases. That assumption can be explained in the following way: after switching on the power supplier a reduction of H⁺ - ions on the cathode occurs, and in the continual liquid phase there is a formation of two-phase gas-liquid fluid. As a result of gas evolution, which in the form of a bubble is captured in the bed, the resistance to electrolyte flow increases,

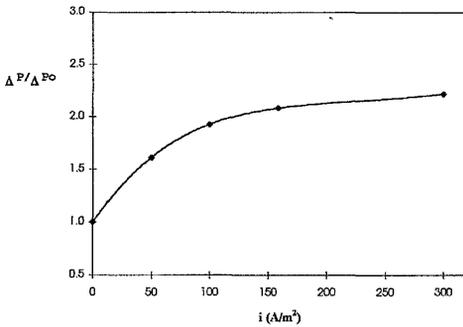


Fig. 6 Relative pressure drop vs. current density (diameter of glass particles $d=2$ mm and electrolyte velocity $w=0.0138$ m/s)

and at the same time there is an increase of the relative pressure drop. In other words, the content of gas in the bed increases with time, heading to the increase of pressure drop. When the maximum content of the gas in the bed is reached, i.e. when the steady state period passes, the change of relative pressure drop reaches maximum value which does not change with time any more. From the electrochemical principles it is well known that there will be more intensive hydrogen evolution on the cathode at higher current density. Higher quantities of evolved hydrogen lead to a faster saturation of the bed with gas bubbles in the initial period, leading to an increase in slopes of the curves $\Delta P/\Delta P_0$ - time. Increase of the relative pressure drop in the steady state can be attributed to an increased gas hold-up, due to a higher gas evolution rate occurring at higher current densities. This increase is observed in the whole range of applied current densities. Since bubble presence in the fixed bed obstructs electrolyte flow, the enlarged gas hold-up will directly influence the increase in the relative pressure drop.

In Fig. 6 the dependence of $\Delta P/\Delta P_0$ against current density for the steady state is shown more clearly.

Lower current densities correspond to a higher pressure drop change tending to reach a certain value, after which the relative pressure drop remains almost constant. Similar behaviour was observed by Chern et al. [9] for a semifluidised bed, for the same range of both gas and liquid.

The influence of particle size

The interspace and channels which are among the particles will depend on the size of the particles. The form of the space can greatly influence hydrodynamic characteristics of two-phase fluid so, because of that, the analysis was made of the influence of particle size on relative pressure drop through a bed. It can also be assumed that the gas phase will be distributed in a different way when passing through a fixed bed than when passing through the bed made up of larger particles. Three series of experiments were carried out,

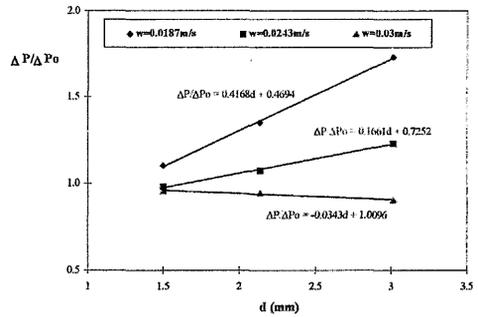


Fig. 7 Dependence of relative pressure drop on particle size (current density $i=100$ A/m²)

in which current density kept constant (100 A/m²), whereas electrolyte velocity in the first series was 0.0187 m/s, in the second series the velocity was 0.0243 m/s, and in the third it was 0.03 m/s. Glass particles of 1.5 mm, 2 mm and 3 mm in diameter were used.

It was found that the change of relative pressure drop increased with the increase of the diameter of particles. This indicated that, at electrolyte velocity of 0.0187 - 0.0243 m/s in a bed, which was formed from larger particles, the channels, were wider. The coalescence of smaller bubbles of hydrogen in such channels was more evident than in narrower channels, which were formed in the fixed bed of smaller particles. Besides, two-phase fluid was distributed in a better way when it passed through a bed of smaller particles, which decreased the total resistance to electrolyte flow. These are the reasons why the change of pressure drop was higher in the bed consisting from larger particles. The fluidisation of particles appeared, at velocities higher than 0.03 m/s, changing the image of the investigated phenomenon. The bed consisting from smaller particle transformed into a fluidised state at smaller velocities of electrolyte, so by the transformation of the bed, the total resistance of electrolyte flow through bed decreased.

The influence of particle size on relative pressure drop is presented Fig. 7 in which a dependence of $\Delta P/\Delta P_0$ (values at a steady state) on diameter of particles is shown.

In Fig. 7 one can see the relationship between the relative pressure drop and particle size for different velocities of electrolyte flow to be linear. It can be seen that the lines corresponding to velocities of 0.0187 m/s and 0.0243 m/s have positive slopes, indicating that at those velocities of electrolyte, the relative pressure drop increases with the increase of particle diameter. With a further increase of electrolyte velocity (0.03 m/s) the slope becomes negative, indicating that particle size at those velocities does not play a great part on the change of pressure drop. The negative slope value, which was found for the velocity of $w = 0.03$ m/s, is a consequence of fluidisation, where the gas phase does not increase the relative pressure drop through the bed. Stankovic [15] has also remarked similar effect.

Conclusions

Based on experimental results the following conclusions could be pointed out:

- The presence of gas phase is evaluated on the electrode in the electrolyte flowing through the fixed bed. This contributes to the pressure drop increase. Gas bubbles decrease the existing electrolyte hold-up, attaching to the particles and bridging the channels in the bed, obstructing electrolyte flow in such a way.
- Relative pressure drop increases with time reaching a constant value after certain time due to an increase of gas hold-up with time.
- Gas evolution intensity, i.e. operating current density, strongly influence the relative pressure drop in such a way that, with increasing of current density, the relative pressure drop increases too.
- Electrolyte velocity through the bed has an opposite effect - the relative pressure drop decreases with increasing of electrolyte velocity (for $w = 0.00587 - 0.03$ m/s). Such effect results in an increased buoyancy force, enabling bubbles to detach from the particles and to take them out of the bed.
- Achieving the fluidised state, relative pressure drop becomes equal to one or slightly less indicating that in fluidised bed, gas phase has no remarkable influence on the pressure drop.
- What has also been remarked is a linear dependence of the relative pressure drop increase with the particle size increasing.

SYMBOLS

A_K	total surface of the cathode, m^2
A_p	cross surface of cell, m^2
d	diameter of a particle, m
F	Faraday's constant, $96\,500\text{ As mol}^{-1}$
G	mass flow, $kg\,s^{-1}$
I	current, A
i	current density, $A\,m^{-2}$
L	height of a bed, m
M	molecular mass of the evolved gas, $kg\,mol^{-1}$
m	mass of gas obtained during water electrolysis, kg
V_{ig}	volume flow of gas evolution, $m^3\,s^{-1}$
w	electrolyte velocity, $m\,s^{-1}$
w_l	liquid velocity, $m\,s^{-1}$
w_{ig}	velocity of gas evolution, $m^3\,s^{-1}\,m^{-2}$
z	number of exchanged electrons

Greek letters

ϵ	bed voidage
ϵ_g	gas hold-up

ϵ_l	liquid hold-up
ϵ_s	solid hold-up
ΔP	pressure drop through the bed, kPa
ΔP_o	initial pressure drop through the bed without gas phase, $kPa\,m^{-1}$
$\Delta P/\Delta P_o$	relative pressure drop
Φ_g	volume part of gas
μ_l	liquid viscosity, Pa's
μ_l^*	dynamic viscosity of gas-liquid, Pa's
ρ_g	gas density, $kg\,m^{-3}$
ρ_l	liquid density, $kg\,m^{-3}$
ρ_l^*	density of gas-liquid, $kg\,m^{-3}$
τ	time realisation of the electrochemical process, s

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