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DIFFUSIONAL MASS TRANSFER THROUGH POROUS MEDIA

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Abstract—A theoretical model representing diffusional mass transfer in electrochemical systems through two successive porous media has been developed and experimentally verified.

There follows from the theory a simple and rapid method for the simultaneous determination of flow porosity and tortuosity, which was successfully applied to sintered glass and beds of glass beads. The extension of the theoretical model to three successive porous media was also experimentally verified.

NOMENCLATURE

A,	mass transfer area [cm^2];
c,	molar concentration of transferred species [mol/cm^3];
D_e,	effective diffusivity [cm^2/s];
f,	Faraday's constant [96 500 C/g equiv.];
i,	limiting current [A];
l,	porous medium thickness [cm];
N_A,	molar flow rate [mol/s];
t,	time [s];
x,	coordinate indicated in Fig. 1;
z,	valence change in electrochemical reaction;
ε,	porosity;
τ,	tortuosity.

Dimensionless groups

C,	dimensionless concentration for the first medium, $(c_1 - c_0)/c_0$;
F,	Fourier number, $D_{e1}t/l^2$;
W,	dimensionless concentration for the second medium, $(c_2 - c_0)/c_0$;
X,	dimensionless length, x/l;
α,	dimensionless parameter, equation (21);
β,	relation between porosities, $\varepsilon_1/\varepsilon_2$;
λ,	relation between effective diffusivities, D_{e1}/D_{e2}.

Subscripts

0,	bulk or initial condition;
1,	first medium;
2,	second medium;
s,	point of divergence between semi-infinite and present model.

INTRODUCTION

THE LITERATURE of studies on porous materials is very extensive. It is sufficient to mention for instance the review of Holdsworth [1], who discusses parameters for characterising porous media, or of Combarnous and Marle [2], who analyse most of the existing methods for determination of properties associated with the structure of porous media. Also several attempts, as reviewed by Petersen [3], have been made to relate the effective diffusion coefficient to the variables characterising porous materials.

In the present work a study of diffusion through porous media, using the well known method of electrochemical measurements of instantaneous mass transfer rates [4–6] was made.

Limiting currents for different electrochemical reactions on circular electrodes contiguous to the porous material were measured. With a suitable position of the electrodes—to avoid the production of free convection—and the addition of a supporting electrolyte—to render negligible the ionic migration—a pure diffusional mass transfer is obtained.

The formulation of a theoretical model representing the phenomenon yields a new method of measuring simultaneously the effective diffusion coefficient, the tortuosity and the flow porosity.* This last parameter is of special interest in, for example filtration problems, and although many methods of measuring open porosity are available, they are generally not suitable for the determination of flow porosity, only the methods based upon transient flow through porous media [7], sinusoidal flow measurements [8, 9] and the penetration of pores by a liquid which wets the medium [10], being reported in the literature.

RELATED THEORY

The mass transfer which takes place through a given porous surface, because of a concentration gradient, is given by:

$$|N_A| = AD_e\varepsilon \frac{\partial c}{\partial x}. \qquad (1)$$

During an electrochemical process, which occurs on an electrode contiguous to a porous medium, the electrical current intensity generated will be given by:

$$i = zf|N_A|_{\text{sup}}. \qquad (2)$$

A representative model of an experimental arrangement (Fig. 1) is that one which takes into consideration the existence of two successive porous media; the former, of finite thickness, rests on the electrode and the latter, infinite, may be another porous medium or else stagnant electrolytic solution.

When pure diffusion is encountered, the following differential equations apply:

$$D_{e_1}\frac{\partial^2 c_1}{\partial x^2} = \frac{\partial c_1}{\partial t} \qquad -l < x < 0; \qquad t > 0 \qquad (3)$$

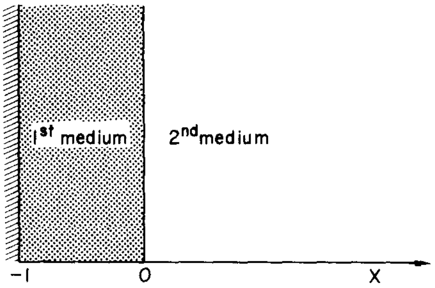

FIG. 1. Schematic diagram of physical model.

$$D_{e_2}\frac{\partial^2 c_2}{\partial x^2} = \frac{\partial c_2}{\partial t} \qquad 0 < x < \infty; \qquad t > 0 \qquad (4)$$

with the corresponding initial and boundary conditions:

$$c_1 = c_0 \qquad -l < x < 0; \qquad t = 0 \qquad (5)$$

$$c_2 = c_0 \qquad 0 < x < \infty; \qquad t = 0 \qquad (6)$$

$$c_1 = 0 \qquad x = -l; \qquad t > 0 \qquad (7)$$

(when working with limiting current)

$$c_1 = c_2 \qquad x = 0; \qquad t \geqslant 0 \qquad (8)$$

$$D_{e_1}\varepsilon_1\frac{\partial c_1}{\partial x} = D_{e_2}\varepsilon_2\frac{\partial c_2}{\partial x} \qquad x = 0; \qquad t \geqslant 0 \qquad (9)$$

$$c_2 = c_0 \qquad x \to \infty; \qquad t \geqslant 0. \qquad (10)$$

By using the dimensionless variables defined in the Nomenclature, equations (3)–(10) respectively may be written

$$\frac{\partial^2 C}{\partial X^2} = \frac{\partial C}{\partial F} \qquad -1 < X < 0; \qquad F > 0 \qquad (11)$$

$$\frac{\partial^2 W}{\partial X^2} = \lambda\frac{\partial W}{\partial F} \qquad 0 < X < \infty; \qquad F > 0 \qquad (12)$$

$$C = 0 \qquad -1 < X < 0; \qquad F = 0 \qquad (13)$$

$$W = 0 \qquad 0 < X < \infty; \qquad F = 0 \qquad (14)$$

$$C = -1 \qquad X = -1; \qquad F > 0 \qquad (15)$$

$$C = W \qquad X = 0; \qquad F \geqslant 0 \qquad (16)$$

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* Referred to, in this paper, simply as porosity.

$$\lambda\beta\frac{\partial C}{\partial X} = \frac{\partial W}{\partial X} \qquad X = 0; \qquad F \geqslant 0 \qquad (17)$$

$$W = 0 \qquad X \to \infty; \qquad F \geqslant 0. \qquad (18)$$

Solving the differential equations by means of Laplace transform methods [11] the following solutions are obtained:

$$C = -\sum_{n=0}^{\infty} \alpha^n \left(\operatorname{erfc}\left\{\frac{2n+1+X}{2\sqrt{F}}\right\} - \alpha \operatorname{erfc}\left\{\frac{2n+1-X}{2\sqrt{F}}\right\}\right) \qquad (19)$$

$$W = -\frac{2\beta\sqrt{\lambda}}{(1+\beta\sqrt{\lambda})}\sum_{n=0}^{\infty} \alpha^n \operatorname{erfc}\left\{\frac{2n+1+X\sqrt{\lambda}}{2\sqrt{F}}\right\} \qquad (20)$$

where

$$\alpha = \frac{\varepsilon_2\sqrt{D_{e_2}} - \varepsilon_1\sqrt{D_{e_1}}}{\varepsilon_2\sqrt{D_{e_2}} + \varepsilon_1\sqrt{D_{e_1}}}. \qquad (21)$$

Equations (19) and (20) permit the evaluation of dimensionless concentration profiles along the first and second medium respectively for given values of F and α.

Intensity of electrical current

According to expressions (1) and (2), and in order to know the intensity of electrical current, it is necessary to find the concentration gradient at the surface of the electrode.

Applying the Leibnitz differentiation formula to (19) one obtains:

$$\left(\frac{\partial C}{\partial X}\right)_{X=-1} = \frac{1}{\sqrt{\pi F}}\left\{1 + 2\sum_{n=1}^{\infty} \alpha^n e^{-n^2/F}\right\} \qquad (22)$$

which combined with (1) and (2) gives:

$$i = \frac{zfAD_{e_1}\varepsilon_1 c_0}{l\sqrt{\pi F}}\{1 + 2\sum_{n=1}^{\infty} \alpha^n e^{-n^2/F}\}. \qquad (23)$$

If i_∞ is defined as:

$$i_\infty = \frac{zfAD_{e_1}\varepsilon_1 c_0}{l} \qquad (24)$$

then:

$$\frac{i}{i_\infty} = \frac{1}{\sqrt{\pi F}}\{1 + 2\sum_{n=1}^{\infty} \alpha^n e^{-n^2/F}\}. \qquad (25)$$

This expression provides the dimensionless electrical current intensity as a function of F and α (Fig. 2).

From the definition of α, equation (21), three distinct systems are worth considering, namely:

1. If $\varepsilon_2\sqrt{D_{e_2}} = \varepsilon_1\sqrt{D_{e_1}}$, then $\alpha = 0$, that is to say that both media are equal. In other words, the system becomes semi-infinite. For this case:

$$\frac{i}{i_\infty} = \frac{1}{\sqrt{\pi F}}. \qquad (26)$$

2. If ε_1 tends to zero, α tends to 1; the parameters of the second medium remain practically constant during the process. An identical solution would be obtained if ideal mixing is postulated for the second medium, in which case D_{e_2} tends to infinity and $\lim_{D_{e_2}\to\infty} \alpha = 1$.

3. If the second medium does not exist ($\varepsilon_2 = 0$), then $\alpha = -1$. This case corresponds to an electrolysis with a porous medium "sandwiched" between two electrodes.

From these observations it can be said that the above three models are all special cases of the more general herein proposed model.

Method for the determination of ε, τ *and* D_e

When comparing expression (25) with the classical solution for a semi-infinite medium, equation (26) it can be inferred that the term

$$2\sum_{n=1}^{\infty} \alpha^n e^{-n^2/F} \qquad (27)$$

is responsible for the deviation existing between both models. Since this deviation occurs gradually it is convenient to define a certain arbitrary "F_s", so that the deviation with respect to the semi-infinite model will be 0·5 per cent for instance; this means that the term (27) should be equal to 0·005.

Therefore, considering only the first element of the summation in (27), since for all practical

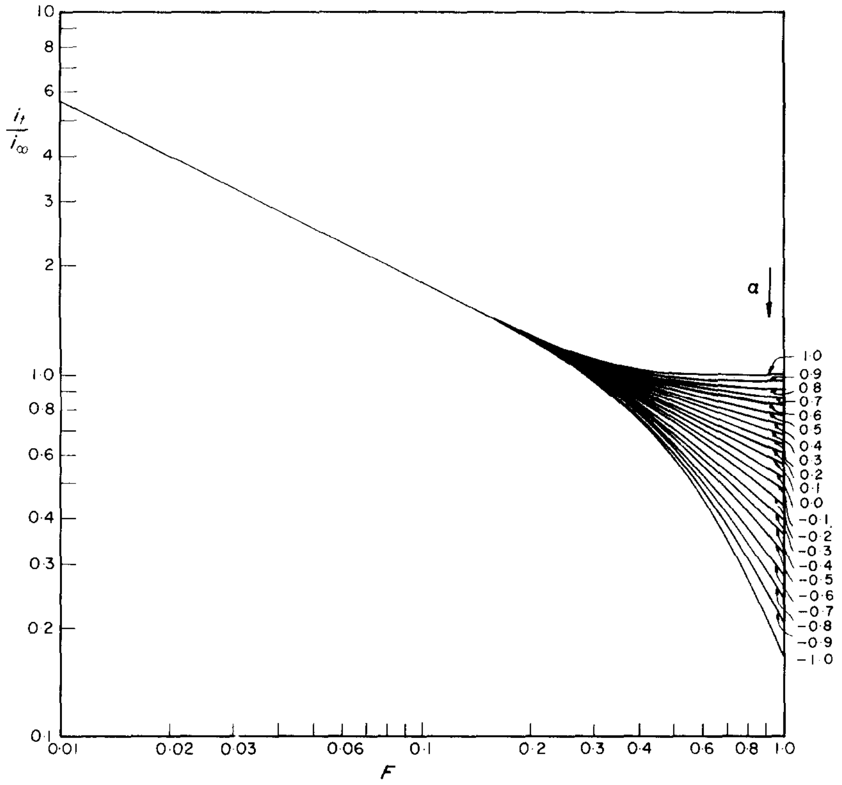


FIG. 2. Theoretical dimensionless curves of the diffusional process.

purposes the remaining terms are negligible for $F > 0{\cdot}17$, it can be written:

$$0{\cdot}005 = 2|\alpha|\,e^{-1/F_s} \tag{28}$$

$$\therefore F_s = \frac{1}{\ln(400|\alpha|)}. \tag{29}$$

When the second medium is an electrolytic solution:

$$\alpha = \frac{\sqrt{D_0} - \varepsilon_1\sqrt{D_{e_1}}}{\sqrt{D_0} + \varepsilon_1\sqrt{D_{e_1}}}. \tag{30}$$

For $F < F_s$ and from equation (23):

$$\varepsilon_1\sqrt{D_{e_1}} = \frac{i\sqrt{t\pi}}{zfAc_0} = k. \tag{31}$$

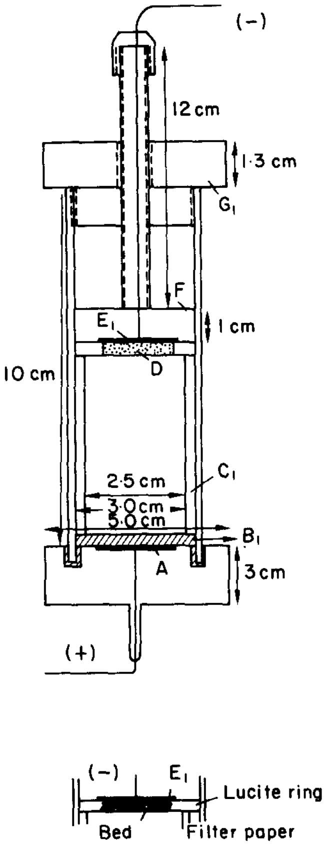
Based on experimental measurements [2nd member of equation (31)] it is then possible to determine $\varepsilon_1\sqrt{D_{e_1}}$, and with a known value of D_0 the value of α corresponding to the system being analysed, is found, equation (30), and therefrom the value of F_s, equation (29).

If tests are made up to and slightly beyond the change of slope, observed when values of log i are plotted vs. log t, $-\varepsilon$, τ and D_{e_1} of packed beds and porous materials may be obtained according to the following relations:

Effective Diffusivity: $$D_{e_1} = \frac{F_s l^2}{t_s} \tag{32}$$

Tortuosity: $$\tau = \frac{D_0}{D_{e_1}} \tag{33}$$

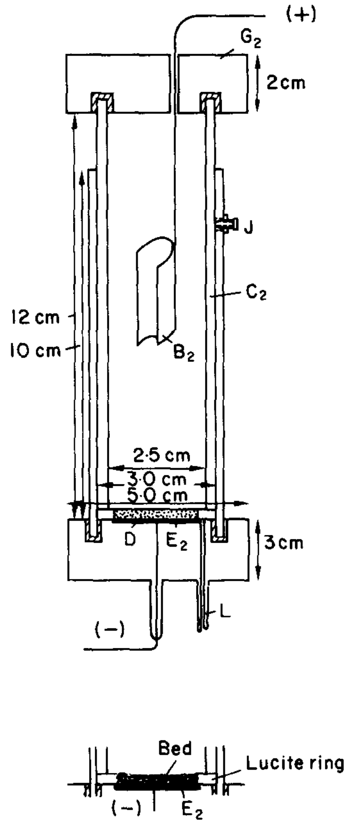
Porosity: $$\varepsilon_1 = \frac{k}{\sqrt{D_{e_1}}}. \tag{34}$$



Porous bed in cell 1

FIG. 3. Cell 1 used with the systems $CuSO_4/H_2SO_4$ and glycerine/$CuSO_4/H_2SO_4$

B_1: copper anode resting on the nickel electrode A
C_1: lucite support cylinder for porous plate D
E_1: copper plated nickel cathode embedded in the piston F
G_1: cell cover.



Porous bed in cell 2

FIG. 4. Cell 2 used with the system $K_3Fe(CN)_6/K_4Fe(CN)_6/$ NaOH.

B_2: nickel anode
C_2: lucite cylinder joined to the cell cover G_2
D: porous plate resting on the nickel cathode E_2
J: screw which sets the cylinder C_2
L: Back-Side-Luggin capillary for measuring the cathodic potential.

Note: The experimental datum t_s must be obtained as follows:

1. The $k' = i\sqrt{t}$ values of the experimental points are calculated. These values remain practically constant while the process continues along the straight line of slope $-0{\cdot}5$, obtained when plotting log i vs. log t. For the straight portion of the line the average value k'_0 is calculated.

2. From all the experimental points the relation $K = k'/k'_0$ is found.

3. K is plotted as a function of t, and t_s is obtained when $K = 1{\cdot}005$.

EXPERIMENT

The following electrochemical systems were tested: electrodeposition of copper from solutions of $CuSO_4/H_2SO_4$ 1·5 M and $CuSO_4/H_2SO_4$

Table 1. Characteristics of model plates, porous plates and porous beds and comparison of ε and τ determined by the electrochemical method with those obtained by conventional methods

Model plates of lucite

No	Density (g/cm^3)	Thickness (mm)	Pore diameter (mm)	Number of pores	$\varepsilon^{(1)}$	ε elect	τ	τ elect
1	1·228	1·110	1·0	40	0·181	0·181	1·0	1·003
2	1·228	1·114	0·8	51	0·148	0·148	1·0	1·002
3	1·228	1·126	0·5	60	0·067	0·068	1·0	1·002
4	1·212	2·364	1·0	40	0·180	0·179	1·0	1·003
5	1·212	2·373	0·8	51	0·159	0·160	1·0	1·001
6	1·212	2·359	0·5	60	0·078	0·079	1·0	1·004
7	1·203	3·278	1·0	40	0·185	0·187	1·0	1·004
8	1·203	3·223	0·8	49	0·167	0·169	1·0	1·001
9	1·203	3·262	0·5	60	0·085	0·086	1·0	1·001

Porous plates of sintered glass

No	Density (g/cm^3)	Thickness (mm)	Pore diameter (μ)	Surface area (cm^2)	$\varepsilon^{(2)}$	ε elect	$\tau^{(3)}$	τ elect
G_0	3·03	2·643	150–200	3·188	0·256	0·249	1·54	1·56
$G_0^{(4)}$	3·03	2·643	150–200	3·188	0·256	0·248	1·54	1·55
G_1	3·03	2·108	90–150	2·931	0·265	0·252	1·52	1·55
G_2	3·03	2·287	40– 90	2·877	0·293	0·291	1·72	1·83
G_3	3·03	1·829	15– 40	2·953	0·365	0·346	1·84	1·84
$G_3^{(5)}$	3·03	1·829	15– 40	2·953	0·365	0·349	1·84	1·89
G_4	3·03	2·031	3– 15	2·673	0·246	0·241	1·80	2·01
F_1	3·03	2·623	—	2·764	0·291	0·289	1·70	1·77
F_2	3·03	2·632	—	3·278	0·280	0·278	1·95	1·93

Porous beds of glass spheres

No	Density (g/cm^3)	Thickness (mm)	Diameter of spheres (μ)	Surface area (cm^2)	$\varepsilon^{(6)}$	ε elect	τ	τ elect
L_1	3·03	1·880	230	3·180	0·355	0·345	—	1·35
L_2	3·03	1·880	300	3·180	0·381	0·379	—	1·31
L_3	3·03	1·880	500	3·180	0·450	0·442	—	1·20

(1) By weight difference with and without pores.
(2) By imbibition of water.
(3) By conductivity [14].
(4) Test E_3.
(5) Test E_4.
(6) From bulk density.

1·5 M/glycerine and the cathodic reduction of ferricyanide in solutions of $K_3Fe(CN)_6/K_4Fe(CN)_6/NaOH$ 2 M.

The cylindrical cells used for these systems, totally built in lucite, are shown in Figs. 3 and 4. Both cells differ basically on the orientation of the cathode in order to avoid natural convection currents.*

Preliminary tests showed that the presence of the porous medium practically does not alter

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* The layers of electrolyte of lower density remain distributed horizontally over the layers of higher density.

the known polarization curves for both systems [12]. Therefore the intensity was recorded as a function of time, applying directly a constant e.m.f. of −400 mV in the case of the electrodeposition and of −600 mV in the case of the redox system, and using the conventional electrical circuit.

Porous media tested

1. *Model plates.* An experimental study was made with 9 model plates, made of lucite, of different thicknesses, with rectilinear pores ($\tau = 1$) of known diameter and number, the porosity of which could be determined with precision. In Table 1 a summary of the characteristics of each plate is given.

In order to assess the validity of the theoretical model developed, the most relevant variables of the diffusional process were analysed: system, concentration, thickness of plate and pore diameter. Three levels were assigned to these variables, which are identified with the numbers 0, 1 and 2 (Table 2).

The study was based on a statistical program, 27 tests being selected as representative from the 81 possible. These were represented schematically with four digital numbers; the first indicates the concentration, the second the system, the third the thickness of the plate and the fourth the pore diameter.* The analysis of the variance of the experimental results, obtained according to an experimental design of the greco-latin square type [13], indicated that none of the variables tested has any influence of relative importance on the diffusional process.

2. *Porous plates and beds.* The study made on the diffusion and the method developed for the determination of ε, τ and D_e was applied to sintered glass plates and beds of glass spheres, the characteristics of which are shown in Table 1.

Since the statistical analysis does not select any one level of the variables, it was convenient to work with $CuSO_4$ 0·1 M/H_2SO_4 1·5 M, based on the following: higher intensities are obtained, the solutions are not altered by time and the

Table 2. Variables and their levels

Assigned position → / Assigned number ↓	Concentration ×	System	Plate thickness (mm)	Pore diameter (mm)
0	0·1 M *(1 M glyc.)	× $K_3Fe(CN)_6$/ × $K_4Fe(CN)^6$/ 2 M NaOH	1	1
1	0·05 M *(2 M glyc.)	× $CuSO_4$/ 1·5 M H_2SO_4	2	0·8
2	0·075 M *(3 M glyc.)	× glycerine/ 0·1 M $CuSO_4$/ 1·5 M H_2SO_4	3	0·5

* When using system 2.

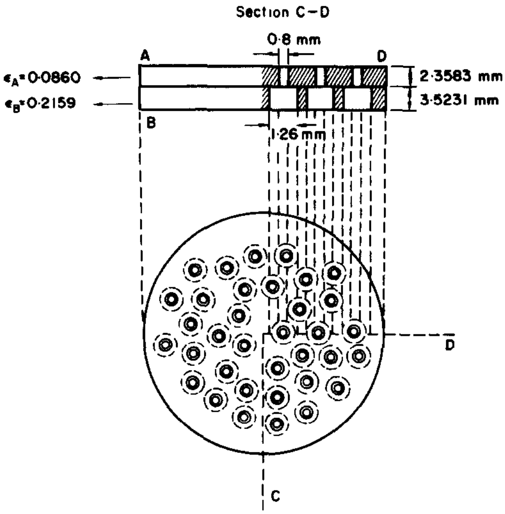

FIG. 5. Combined plate with equal number of concentric pores of different diameter.

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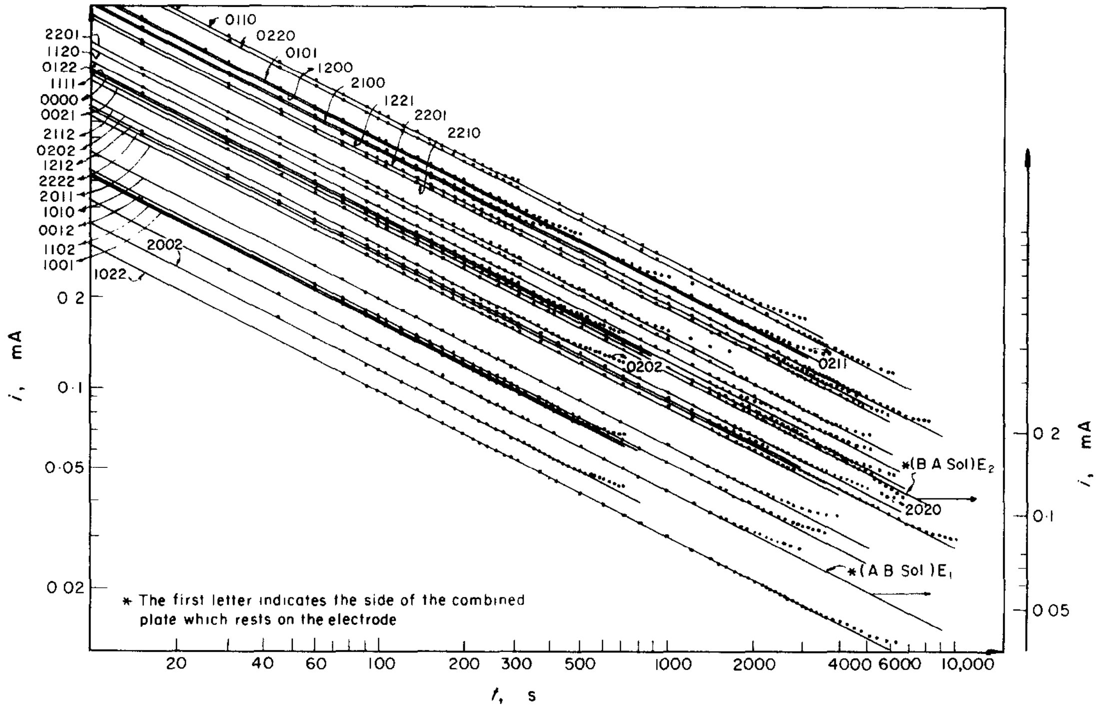
* For example test 1102 corresponds, according to Table 2, to the following conditions: concentration 0·05 M; system $CuSO_4$/H_2SO_4; plate thickness 1 mm; pore dia. 0·5 mm.



* The first letter indicates the side of the combined plate which rests on the electrode

FIG. 6. Experimental curves of limiting current as function of time obtained with model plates; two and three successive media.

corresponding cell can be handled easily. However, the concentration and/or the system were varied in some of the tests, as specified in Fig. 7.

3. *Three successive media.* In order to follow the behavior of the diffusion phenomenon when the second medium is not an electrolytic solution, but a medium whose porosity differs from that of the first medium, a model was built in lucite (Fig. 5) by joining two plates with an equal number of rectilinear pores but of different diameters, which forms, with the electrolytic solution, a system of three successive media. The tests E_1 and E_2 were made with this combined plate. Also the sintered glass plates G_0 and G_3 placed in series were used (tests E_3 and E_4).

RESULTS

The experimental data of the limiting current as a function of time, for all the tests made, are represented on a log–log scale in Figs. 6 and 7. As expected from the theoretical analysis, a straight line of slope $-0{\cdot}5$ and the ascending or descending deviations, in accordance with the value of α for the given system, is obtained.* The times at which the deviations occur depend on the thickness of the porous medium used in the experiment. The values of t_s were obtained as indicated. (Figure 8 shows the curves corresponding to some of the tests.) The values of ε, τ and D_e were calculated as described.

The calculated values of ε and τ, as well as the data of porosity and tortuosity obtained by other known methods, appear in Table 1. A satisfactory concordance can be appreciated.

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* In the case of porous beds a slightly higher intensity than that corresponding to the straight line of slope $-0{\cdot}5$ is recorded at the beginning. After 200–250 s the data behave as expected theoretically. This behavior is probably due to a greater surface porosity with respect to volume porosity.

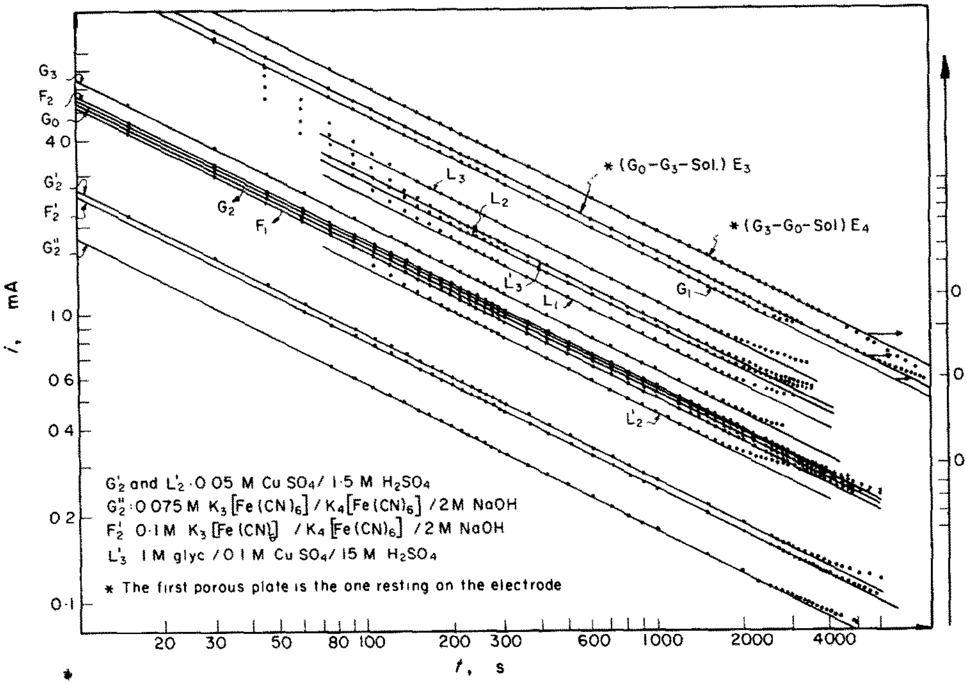

FIG. 7. Experimental curves of limiting current as function of time for porous plates and beds; two and three successive media.

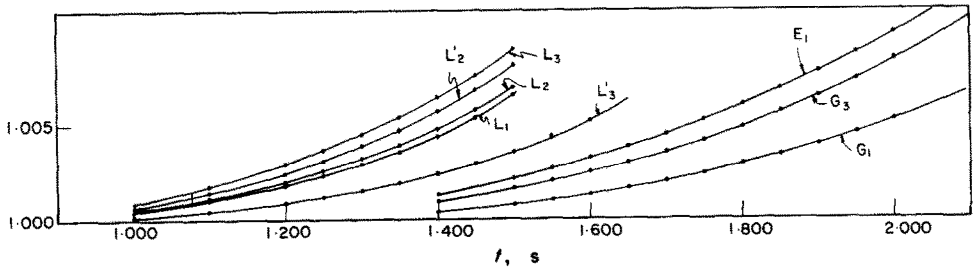

FIG. 8. Determination of t_s for porous plates and beds.

Values of i_∞ [equation (24)] for each run were obtained using the data of ε and D_e. Also, the values of F corresponding to the times recorded in the different tests were calculated, making it possible to represent i/i_∞ vs. F. These dimensionless curves of the diffusional process permit the quantitative verification of the theoretical model. Figure 9 shows the dimensionless curves of some of the tests simultaneously with the theoretical curves. The concordance obtained shows that the model is representative of the diffusional process in porous media and may be extended to three successive media up to relative large values of F.

CONCLUSIONS

1. The theoretical model represents adequately the diffusional mass transport in porous media.
2. The porosity, tortuosity and effective diffusivity of porous plates and beds may be determined simultaneously in a short time by means of the electrochemical technique in combination with the method developed in the theoretical analysis.
3. None of the variables which affect the diffusional process have any influence over the optimal conditions to be used in the method herein presented.
4. Restrictions:

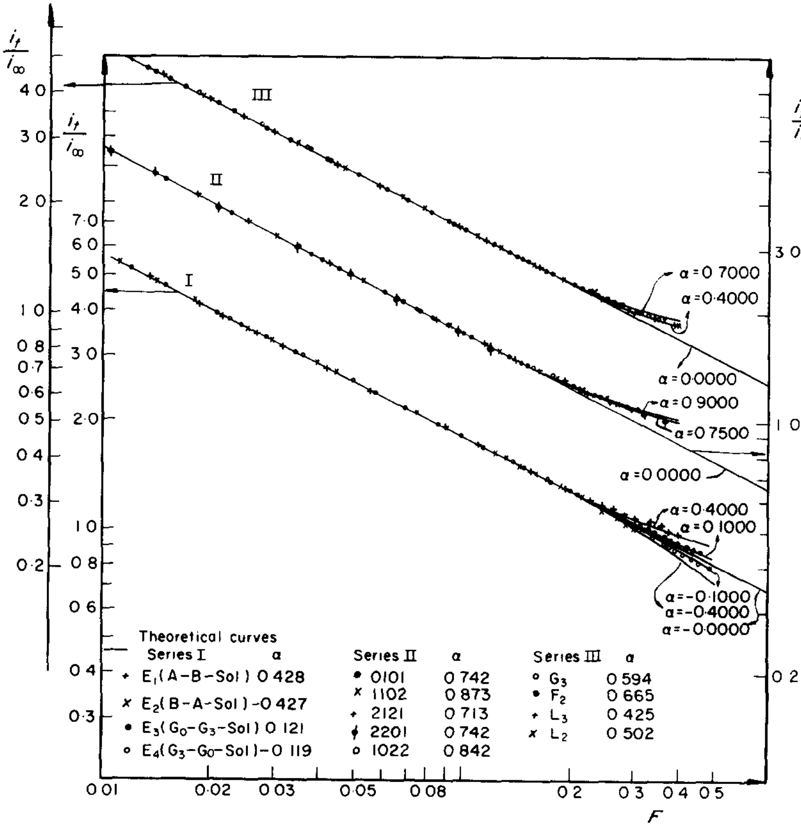

FIG. 9. Comparison of theoretical curves with experimental data.

(a) The electrochemical method is applicable only to materials which are non-conductive.

(b) The porous plates must rest evenly on the electrode.

(c) The pore diameter must not be so small as to permit measurable film diffusivities.

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TRANSFERT DIFFUSIONNEL MASSIQUE À TRAVERS UN MILIEU POREUX

Résumé— On a développé et vérifié expérimentalement un modèle théorique représentant un transfert diffusionnel massique dans des systèmes électrochimiques à travers deux milieux poreux successifs. Il découle de cette théorie une méthode simple et rapide permettant la détermination simultanée de la porosité et de la tortuosité, méthode qui est appliquée avec succès à du verre brisé et des lits de billes de verre.

L'extension du modèle théorique à trois milieux successifs poreux est aussi vérifiée expérimentalement.

STOFFÜBERGANG MIT DIFFUSION DURCH PORÖSE MEDIEN

Zusammenfassung—Ein theoretisches Modell wurde entwickelt und experimentell bestätigt, das den Stoffübergang mit Diffusion in elektrochemischen Systemen durch zwei aufeinanderfolgende poröse Medien beschreibt.

Aus der Theorie folgt eine einfache und schnelle Methode zur gleichzeitigen Bestimmung von Strömungsdurchlässigkeit und Strömungsverwindung. Diese Methode wurde nacheinander auf gesintertes Glas und Schüttungen von Glaskügelchen erfolgreich angewandt. Die Erweiterung des theoretischen Modells auf drei aufeinanderfolgende poröse Medien wurde ebenfalls experimentell bestätigt.

ДИФФУЗИОННЫЙ ПЕРЕНОС МАССЫ ЧЕРЕЗ ПОРИСТЫЕ СРЕДЫ

Аннотация—Разработана и экспериментально проверена теоретическая модель диффузионного переноса массы в электрохимических системах через две последовательные пористые среды.

На основании теории выводится простой и быстрый метод для одновременного определения порисости и извилмстости течения, который успешно применялся для исследования пористого стекла и слоев стеклянных шариков. Применение теоретической модели для трех последовательных пористых сред проверено экспериментально.