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ORIGINAL PAPER

Electrodiffusion diagnostics of laminar flows using the delay-time method

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Abstract Electrodiffusion diagnostics are widely used for measurement of local wall shear stresses in liquid flows. At present, this method requires special electrolytes, so the applications are limited to laboratory conditions. Another problem concerns the dependence of the limiting diffusion current on the electrode surface state and on the bulk concentration of the electroactive species. In this paper, it is shown that the delay time between generation of the electroactive species and their detection on the downstream electrode, is directly related to the local wall shear stress value. Thus, the measurements of the delay-time open a new way for the study of near-wall hydrodynamics. This new method has been confirmed experimentally using an electrolyte containing the conventional hexacyanoferrate(III/II) redox couple, as well as with the chlorine (chloric(I))/chloride couple in an electrolyte similar to seawater.

Keywords Electrodiffusion diagnostics · Mass transfer · Flow measurements · Delay-time

Nomenclature

Symbols

c	Electroactive species concentration, mol m^{-3}
c_0	Bulk concentration of electroactive species, mol m^{-3}
D	molecular diffusion coefficient, m^2 s^{-1}
E_G, E	Electric potentials of electrode, V
I	Electric current, A
S	Microelectrode surface, m^2
T	Electrolyte temperature, °C
V	Flow velocity in longitudinal (×) direction, m s^{-1}
x	Flow direction, m
y	Normal to the wall direction, m
t	Time, s
d	Electrode length in flow direction, m
h	Electrode width in transversal (z) direction, m
l	Distance between generator and detector electrodes, m
j_0	Mass flux density on the surface of generator electrode, mol m^{-2} s^{-1}
$y_{\mathrm{eff}} = (Dl\mu/\tau_{\mathrm{w}})^{1/3}$	Effective diffusion length in normal to the wall direction, m

Greek symbols

μ	Dynamic viscosity, kg m^{-1} s^{-1}
ν	Kinematic viscosity, m^2 s^{-1}
ρ	Electrolyte density, kg m^{-3}
τ_{d}	Delay time, s
τ_x	Convective time, s
τ_y	Diffusion time, s
$\tau_* = y_{\mathrm{eff}}^2/D$	Characteristic time, s
τ_{w}	Wall shear stress, Pa
$\zeta = d/l$	Dimensionless geometrical parameter of the electrode system

Superscript

w	Wall or electrode surface

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1 Introduction

The electrochemical (electrodiffusion) diagnostics of flows are widely used for the measurements of the wall velocity gradient and the local wall shear stress τ_w in electrolyte solutions [1–14]. This method is based on the measurement of the limiting diffusion current I on small flush-mounted electrodes. According to the Leveque analogy [15] with heat transfer, the current I depends on the local wall shear stress value τ_w on the electrode surface

$$I = kSc_0\tau_w^{1/3}, \tag{1}$$

where c_0 is the bulk concentration of the electroactive species, S is the electrode surface area, and k is a constant, which depends on the electrode geometry, molecular diffusion coefficient and the solution viscosity.

Limiting diffusion current measurements are possible only if the electrochemical reaction kinetics are diffusion controlled. Fast redox couples, such as hexacyanoferrate(III/II), should be used to ensure this condition:

$$\text{anode reaction:}\quad [\mathrm{Fe(CN)_6}]^{-4} - \mathrm{e} \rightarrow [\mathrm{Fe(CN)_6}]^{-3}; \tag{2a}$$

$$\text{cathode reaction:}\quad [\mathrm{Fe(CN)_6}]^{-3} + \mathrm{e} \rightarrow [\mathrm{Fe(CN)_6}]^{-4}. \tag{2b}$$

Electrochemical flow diagnostics based on limiting diffusion current measurements and Eq. (1), can be called *the amplitude electrodiffusion method.* The main disadvantage of the amplitude method is a high sensitivity of the limiting diffusion current I to the electrode surface state and to the electrolyte purity. For example, non-controlled changes of the microelectrode surface state caused by the adsorption of surfactants or non-soluble products of the auxiliary reactions can influence the limiting diffusion current value significantly. In natural (water, seawater) and industrial conditions, the amplitude method has not been used hitherto, due to the lack of suitable fast redox couples.

The electrodiffusion amplitude method has proved its efficiency for measurements of the mean and the fluctuating wall shear stress and flow velocity. Segmented electrodes allow measurements of the vector characteristics of the flow [3]. Another application of multi-electrode systems concerns the measurements of the delay-time characteristics. This approach has been developed for studies of fast electrochemical reactions using rotating ring-disc electrodes [16]. Delay-time measurements can also extend the experimental performance of electrochemical methods regarding near wall flow studies. In the present paper, we demonstrate the performance of multi electrode systems and the delay-time method for flow diagnostics, in particular, the possibility to overcome the principal limitation of the amplitude method related to the use of special electrolyte solutions.

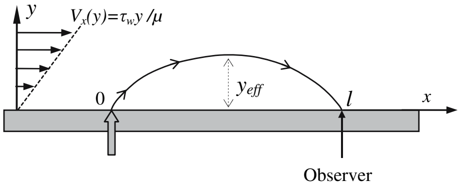

Fig. 1 Diffusion of species in near-wall flow

Figure 1 demonstrates the main principle of the delay-time method. Let us consider a flow with the velocity profile $V_x(y)$. At the moment $t = 0$, a source situated at the origin $x = 0$ begins to generate some species in the flow. The observer, located downstream of the source at the distance l, will detect the species arrival with some delay τ_d. This delay-time depends on the hydrodynamic regime of the flow. At high values of Schmidt number, the mass transfer is located in the near wall zone, where the flow is determined by the wall velocity gradient. In Newtonian flows, the wall velocity gradient is proportional to the wall shear stress τ_w; hence, the delay-time depends on the wall shear stress. Thus, by measuring the delay-time τ_d, it is possible to determine the wall shear stress τ_w. Theoretical and experimental determination of the functional dependence $\tau_d = f(\tau_w)$ has significant practical interest and is the main goal of this paper. For simplification of some points, the present paper is limited to the case of the laminar flows. Nevertheless, the proposed method can also be applied to studies of turbulent flows.

2 Diffusion of species injected in the flow from the wall

Mass transfer in liquids is characterized by high values of Schmidt number $Sc \gg 1$ and the diffusion layer is located in the near vicinity of the wall, where the linear approximation for the velocity profile can be used: $V(y) = \frac{\tau_w}{\mu} y$, where μ is the dynamic viscosity and y is the distance measured from the wall. The mass transport of the species injected from the wall can be described by the non-steady equation of convective diffusion, which, within the frame of the boundary layer approximation, takes the form:

$$\frac{\partial c}{\partial t} + \frac{\tau_w}{\mu} y \frac{\partial c}{\partial x} = D \frac{\partial^2 c}{\partial y^2}, \tag{3}$$

where $c(x, y, t)$ is the species concentration in the flow; D is the molecular diffusion coefficient of the species.

Boundary conditions at the wall $y = 0$ depend on the geometry and on the type of the source that generates the species. Suppose that the electroactive species are generated by a step polarization of the flush-mounted rectangular electrode with the length d in the flow direction (x). The width h of the electrode in the transverse direction (z) is assumed sufficiently large ($h \gg d$), in order to avoid border effects in the transverse direction. The species are detected downstream by an electrode of the same dimensions. The polarization of this electrode assumes the limiting diffusion current regime with respect to the generated species. The distance between the generator and the detector electrodes is equal to l. In this section, we consider the case when the electroactive species assuming the limiting diffusion current regime on the detector electrode are absent originally in the solution. It is accepted also that after polarization, the current density is uniformly distributed along the surface of the generator electrode. So, we can specify the boundary conditions for Eq. (3):

$$\begin{aligned} &t=0: \quad c\,(x,y,0)=0;\\ &t>0; \quad x\in(0,d),\ \ y=0;\ \frac{\partial c}{\partial y}=\frac{j_0}{D}\\ &\qquad x\in(d,d+l),\ \ y=0;\ \frac{\partial c}{\partial y}=0\\ &\qquad x\in(d+l,l+2d),\ \ y=0;\ \ c=0\\ &\qquad x=0;\ c(0,\ y,\ t)=0;\\ &\qquad y\to\infty;\ c(x,\ y,\ t)=0. \end{aligned} \tag{4}$$

Here j_0 is the mass flux density on the surface of the generator electrode. Using dimensionless variables

$$C=\frac{c}{c_{\rm eff}};X=\frac{x}{l};Y=\frac{y}{y_{\rm eff}};T=\frac{t}{\tau_*};\ c_{\rm eff}=\frac{j_0y_{\rm eff}}{D};\quad y_{\rm eff}=\left(\frac{Dl\mu}{\tau_{\rm w}}\right)^{1/3};\tau_*=\frac{y_{\rm eff}^2}{D}, \tag{5}$$

it is possible to transform Eqs. (3) and (4) to the following dimensionless forms:

$$\frac{\partial C}{\partial T}+Y\frac{\partial C}{\partial X}=\frac{\partial^2 C}{\partial Y^2} \tag{6}$$

$$\begin{aligned} &T=0: C(X,\ Y,\ 0)=0;\\ &T>0; X\in(0,\zeta),\ Y=0;\ \frac{\partial C}{\partial Y}=1\\ &\qquad X\in(\zeta,\zeta+1),Y=0;\ \frac{\partial C}{\partial Y}=0\\ &\qquad X\in(\zeta+1,\ 2\zeta+1),\ Y=0;\ C=0\\ &\qquad X=0;\ C(0,\ Y,\ T)=0;\\ &\qquad Y\to\infty;\ C(X,\ Y,\ T)=0. \end{aligned} \tag{7}$$

Equations (6) and (7) depend only on the geometrical parameter $\zeta = d/l$. This means that the time scale of the non-stationary diffusion problem considered is equal to τ_*. So, the delay time between the beginning of the generation of the electroactive species and their detection on the downstream electrode is proportional to:

$$\tau_{\rm d}=a\,(\zeta)\tau_*, \tag{8}$$

where the function a depends on the geometry of the electrode system (ζ) and on the choice of the sensitivity level for the registration of the electroactive species on the detector electrode.

It follows from Eq. (8) that the delay-time $\tau_{\rm d}$ is proportional to $\tau_{\rm w}^{-2/3}$. If other parameters in Eq. (8) are known, it is possible to determine the wall shear stress $\tau_{\rm w}$ by measuring the delay-time $\tau_{\rm d}$. Modern electronic devices allow these measurements to be made with high accuracy.

At constant temperature, the parameters D and μ are constant, so the transformation characteristic "*wall shear stress/delay-time*" has a simple form:

$$\tau_{\rm d}=A\tau_{\rm w}^{-2/3}, \tag{9}$$

where the coefficient A can be obtained using calibration. In the general case, in order to take into account the influence of temperature, additional measurements of the temperature dependence $A = f(T)$ should be made.

Equation (8) can also be obtained using simple physical arguments [6]. Indeed, the mass transfer of the injected species is governed by two mechanisms: molecular diffusion in the normal direction and convection along the flow with the velocity $V(y)=\frac{\tau_{\rm w}}{\mu}y$.

The time of the molecular diffusion across the flow can be estimated from the following relation:

$$\tau_y \sim y^2/\ D. \tag{10}$$

The convection time τ_x is estimated as:

$$\tau_x \sim l/V(y)=l\,\mu/(\tau_{\rm w}\ y). \tag{11}$$

The diffusion time τ_y and the convection time τ_x are both of the same order of magnitude as the delay-time τ_d:

$$\tau_x \sim \tau_y \sim \tau_d \sim \tau_*. \tag{12}$$

Combining Eqs. (10)–(12) enables the "effective" diffusion length in the direction normal to the wall to be estimated. This distance $y_{\rm eff}$ coincides with that given by Eq. (5); substitution of this result for $y_{\rm eff}$ into Eq. (10) or (11), confirms Eq. (8).

3 Experimental investigation of the near wall mass transfer

For experimental verification of the relations presented above, a system was used consisting of four Pt

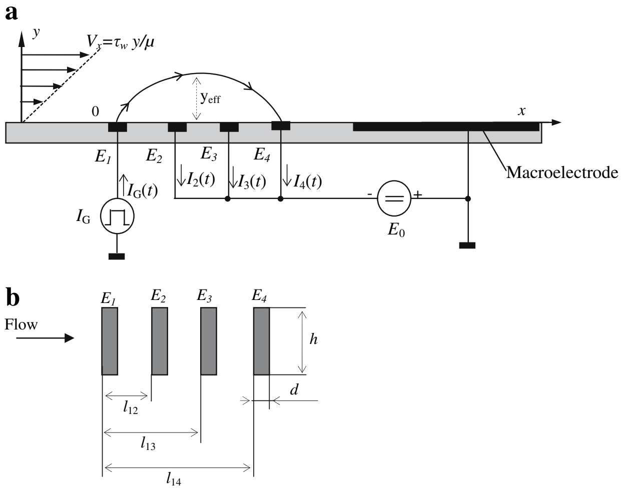

Fig. 2 Electrical scheme of delay-time measurements (**a**) and topology of multi electrode system (**b**)

microelectrodes *E1–E4* and one large counter electrode. All electrodes were flush mounted with the wall as shown in Fig. 2a. The electrical regime of the electrode *E1* was governed by the impulse source of voltage E_G, the electrodes *E2–E4* were polarized using a constant source of voltage E_0. This multi-electrode system was flush mounted in a rectangular channel with a cross section of $120 \times 12\ \mathrm{mm}^2$ at a distance 2000 mm from the entrance, in order to ensure stabilized hydrodynamic flow conditions. The electrolyte temperature during the experiments was constant at $T = 21.0 \pm 0.3\ °\mathrm{C}$. The topology of the electrode system and its geometric parameters are given on Fig. 2 and in Table 1.

Two types of electrolyte solutions were used:

- 25 mol m^{-3} aqueous solution of $K_3Fe(CN)_6$ and $K_4Fe(CN)_6$ with 300 mol m^{-3} concentration of K_2SO_4 as supporting electrolyte;
- 3% aqueous solution of *NaCl* at pH ca. 8; this chemical composition simulates that of seawater.

Initially, a positive voltage step E_G was applied to the generator electrode *E1*. A potential step polarization of this electrode resulted in a step injection of the species into the near wall flow. A negative electrical potential E_0 (with respect to the counter electrode) was applied continuously to three downstream electrodes *E2*, *E3*, and *E4* in order to provide the limiting diffusion current regime. These downstream electrodes were used as the detectors of the concentration signal, which was carried by the flow.

Table 1 Geometrical parameters of multi electrode system

l_{12}	l_{13}	l_{14}	d	h
0.8 mm	1.6 mm	2.4 mm	0.5 mm	2.5 mm

When heaxcyanoferrate(III/II) electrolyte solution is used, the oxidation reaction, Eq. (2a), takes place on the generator electrode and the reduction reactions, Eq. 2b, takes place on the detector electrodes *E2–E4.* In the aqueous solution of *NaCl*, molecular chlorine is generated on the electrode *E1*:

$$2\mathrm{Cl}^- - 2\mathrm{e}^- \rightarrow \mathrm{Cl}_2, \tag{13a}$$

is hydrolysed at $3 < \mathrm{pH} < 7.5$:

$$\mathrm{Cl}_2 + \mathrm{H}_2\mathrm{O} \rightarrow \mathrm{HOCl} + \mathrm{Cl}^- + \mathrm{H}^+ \tag{13b}$$

forms chloric(I) ions at pH > 7.5 at 298 K:

$$\mathrm{HOCl} \rightleftarrows \mathrm{OCl}^- + \mathrm{H}^+ \tag{13c}$$

and the reduction reaction takes place on the detector electrodes *E2–E4*:

$$\mathrm{OCl}^- + \mathrm{H}_2\mathrm{O} + 2\mathrm{e}^- \rightarrow \mathrm{Cl}^- + 2\mathrm{OH}^-. \tag{13d}$$

The arrival of the concentration signal generated by the electrode *E1* changes the limiting diffusion currents of the electrodes *E2–E4*, enabling detection of the arrival of the concentration signal. An 8-channel potentiostat *Solartron Sl 1480A MultiStat* was used as the measuring device.

All electrochemical experiments involved well-controlled hydrodynamic conditions with a known value of the wall shear stress τ_w. Using the potentiostat, a voltage step $E_G(t)$ was applied to the generator electrode *E1* and the time evolution of the currents $I_2(t)$, $I_3(t)$, $I_4(t)$ of the electrodes *E2–E4* was measured. The delay time τ_d was determined using the starting front of these currents and the preliminary normalization:

$$\Delta I_i(t) = \frac{I_i(t) - I_{0i}}{I_{i\max} - I_{0i}}, \tag{14}$$

where $I_i(t)$ is the current of the *i*-electrode; I_{0i} is the steady state (before generation) current of the *i*-electrode; $I_{i\max}$ is the maximal current of the *i*-electrode.

Figures 3 and 4 illustrate the experimental procedure used for determination of the delay-time. The wall shear stress in these experiments is equal to: $\tau_w = 9.4 \times 10^{-3}$ Pa. The voltage-step $E_G(t)$ of the generator electrode *E1* and the time evolution of the currents $I_2(t)$, $I_3(t)$, and $I_4(t)$ are shown in Fig. 3a,b. The initial current value I_0 is subtracted from the total current on Fig. 3b. The beginning of the voltage step corresponds to the time origin. The beginning and the end of the voltage step $E_G(t)$ on all figures are marked by vertical dotted lines. Figure 3c shows the normalized currents $\Delta I_2(t)$, $\Delta I_3(t)$, and $\Delta I_4(t)$ of the electrodes *E2, E3*, and *E4*.

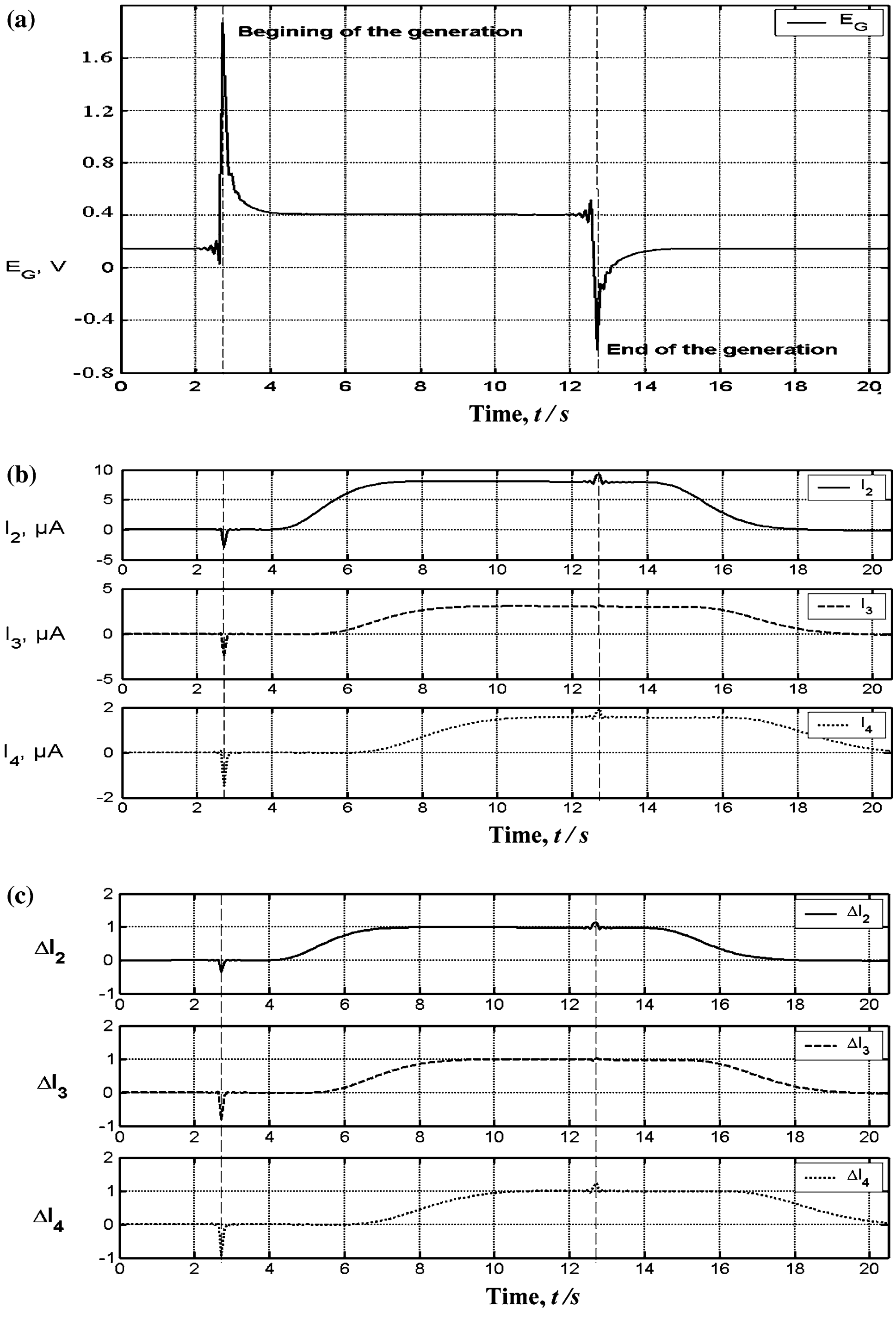

Fig. 3 Potential step on the electrode *E1* (**a**) and corresponding current responses of the electrodes *E2, E3, E4* (**b**) and their normalizing values (**c**). The initial current value I_0 (before step polarization of electrode *E1*) is subtracted from the total current on (**b**)

Figure 4 shows the starting fronts of the normalized currents obtained in hexacyanoferrate(III/II) electrolyte solution. The delay time τ_{di} corresponding to the i-electrode was determined from these charts, using the change in the normalized currents $\Delta I_i(t)$ up to the value 0.5 as the sensitivity level. It should be noted that the starting fronts of the currents were sharp and rose after a well-defined delay time τ_d.

One of the main advantages of the delay time method with respect to the traditional amplitude method was the possibility to work in seawater. In order to confirm this fact, the multi-electrode system was tested in NaCl solution with NaCl concentration similar to seawater. In this electrolyte, the step injection of the chlorine generates a concentration signal. Figure 5 shows the time evolution of the currents of the electrodes *E2–E4* obtained in NaCl

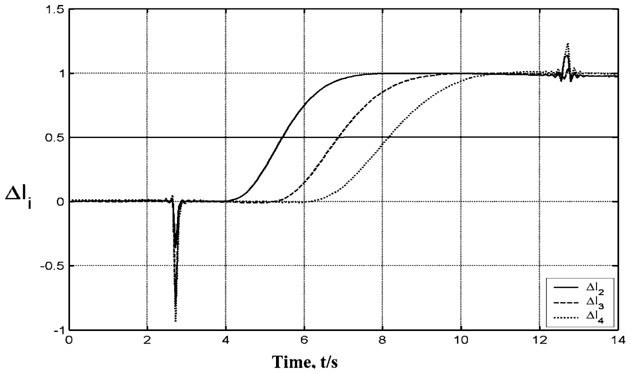

Fig. 4 Normalizing currents of the electrodes E2, E3, E4 (response on the voltage step) in solution containing hexacyanoferrate(III/II)

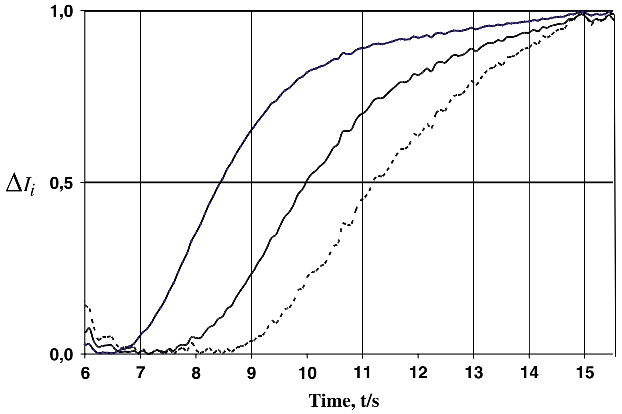

Fig. 5 Normalizing currents of the electrodes E2, E3, E4 (response on the voltage step) in aqueous *NaCl* solution

solution. These charts are similar to those presented in Fig. 4. This result confirms that the electrodiffusion delay-time method can be used in seawater conditions and for industrial applications related to use of the electrolyte solution with Cl^- ions.

Figure 6 with double logarithmic scale presents the dependence $\tau_d = f(\tau_w)$ for the electrodes *E2*, *E3*, and *E4*. This dependence confirms Eq. (9) and justifies the theoretical background of the delay-time method.

4 Accuracy and limitations of the delay-time method

Calibration of the delay-time sensors and the accuracy of this method is a delicate problem. This method has both advantages and disadvantages in comparison with the traditional amplitude method. It means that it is necessary to look for the specific applications where advantages of this new method dominate the possible disadvantages.

The coefficient A in Eq. (9) can be obtained using the experimental data presented on Fig. 6. It should be noted that the coefficient A depends on the geometry of the electrode system (distance between the electrodes and their length), so preliminary calibration is necessary for each delay-time sensor. It is not a principal restriction of this method as usually all types of sensors are calibrated in the known experimental conditions.

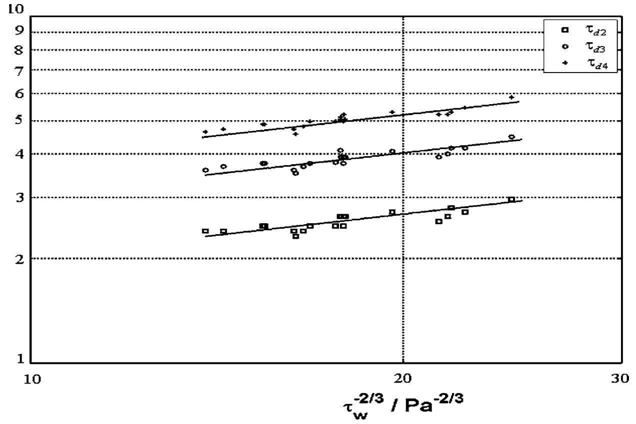

Fig. 6 Delay-time as a function of wall shear stress for different distances between generator and detector electrodes: l_{12}—(□), l_{13}—(○), l_{14}—(+). Experiments carried out with hexacyanoferrate(III/II) and with aqueous *NaCl* solutions

We have limited this paper to the case of laminar flows, but it seems, that the delay-time method can be used in turbulent flows as well. Indeed, if the distance between generator and detector electrodes is sufficiently small, we can expect that the effective diffusion length y_{eff} is very small in comparison with the viscous sub-layer thickness and the turbulent pulsations will not affect mass transfer. In principle, the flow velocity vector can also be detected by this method. In this case, a second detector electrode should be used with different orientation with respect to the generator. These points need further study.

It should be noted that the electrode area and the bulk concentration of the electroactive species do not appear in Eq. (8) for the delay-time. Thus, the number of parameters which describe the transformation characteristic $\tau_d = f(\tau_w)$ in the *delay-time method* decreases in comparison with the *amplitude method*, which is based on Eq. (1). This fact should increase the accuracy of the measurements. Moreover, the delay-time method is not very sensitive to uncontrolled changes in the state of the electrode surface or concentration of electroactive species. This is very important for practical applications.

At present, it is difficult to estimate the accuracy of the delay-time method. We can note only that this accuracy is determined by the accuracy of the measurements of the time characteristics, which can be detected with high precision. In the present work, the delay-time has been measured using the sensitivity level 0.5 with respect to the normalized current. There are different electro-technical methods for registration of the time characteristics, which can probably improve the accuracy of the delay-time measurements, but we have not examined this question in the present study. Another parameter which may influence the accuracy of the delay-time method is the geometry of the electrode system, in particular the geometrical factor $\zeta = d/l$ in Eq. (8). As the accuracy of the delay-time method also depends on the type of electrochemical system used; obviously the measurements with natural or industrial electrolytes are more difficult than with those in a laboratory. Lastly, the accuracy of the delay-time method is related to the thermal dependence of the physico-chemical properties of the electrolyte solution, but it is a general problem for electrochemical flow diagnostics.

The space resolution of the delay-time method is determined by the scale of the electrode system. Using modern microfilm technologies [8, 13], it is possible to fabricate sensors with extremely small working electrodes. Hence, it is interesting to estimate the minimum distance $l_{\min}$ between the generator and the detector electrodes that is admissible for the delay-time method.

The limiting factor for miniaturisation of the electrode system is longitudinal molecular diffusion. With decreasing values of l, the characteristic time of the longitudinal molecular diffusion diminishes as

$$t_{\mathrm{d}} \sim \frac{l^2}{D}. \tag{15}$$

For small values of l, this time becomes smaller than the delay-time τ_{d}. In this case, the propagation of the concentration signal in the longitudinal direction is governed by molecular diffusion and the sensor is not sensitive to flow velocity. Thus, the criterion for the minimum length between the electrodes $l_{\min}$ can be obtained from the relation:

$$\tau_{\mathrm{d}} \gg \tau_{\mathrm{d}} \tag{16}$$

Using Eqs. (8), (15), and (16), we obtain the criterion for the minimum distance $l_{\min}$

$$l_{\min} \gg l_0; \; l_0 = \left(\frac{a\mu D}{\tau_{\mathrm{w\,min}}}\right)^{1/2}, \tag{17}$$

where $\tau_{\mathrm{w\,min}}$ is the minimum range of the wall shear stress for the given measuring device and a is the numerical constant in Eq. (8) that should be obtained in practical applications from calibration experiments. For example, for minimum wall shear stress values equal to $\tau_{\mathrm{w\,min}} = 10^{-2}$; 10^{-1}; 1 Pa, we estimate a critical length l_0 of 16; 5; 1.5 μm, respectively. It was assumed that in aqueous solutions $\mu \approx 10^{-3}$ kg m^{-1} s^{-1}; $D \approx 10^{-9}$ m^2 s^{-1} and the constant $a \approx 1$. This estimation of the minimum length $l_{\min}$ confirms that even for very slow flows, as in microfluidics devices, it is possible to fabricate really miniaturized electrode systems, suitable for wall shear stress measurements with high spatial resolution.

5 Conclusions

The electrodiffusion *delay-time method* allows wall shear stress τ_{w} measurements by means of registration of the delay τ_{d} between the injection of the electroactive species in the flow and their detection at the downstream electrode. With modern electronic devices, it is possible to measure the delay-time with high accuracy. This opens new possibilities for electrochemical flow diagnostics. Indeed, the delay-time method is not very sensitive to the state of the electrode surface and to the bulk concentration of the electroactive species; hence, the accuracy of the delay-time method should be higher in comparison to the amplitude method. This advantage of the delay-time method can be applied for periodical calibration of the amplitude electrodiffusion probes. In this case, two working electrodes separated by an insulator should be used.

It has been derived theoretically and confirmed experimentally that in laminar flow the transfer characteristic "delay-time/wall shear stress" satisfies the power law: $\tau_{\mathrm{d}} = A\tau_{\mathrm{w}}^{-2/3}$, where the constant A can be obtained by calibration.

It should be noted that the delay-time method can be used in different electrolytes that are important for practical applications. In particular, in the present paper we have demonstrated that the method is applicable to wall shear stress measurements in *NaCl* solutions of concentration similar to Mediterranean Seawater.

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