

57 (2016) 26031–26040 November



Determination of correct zeta potential of polyether sulfone membranes using CLC and AGC: ionic environment effect

Sema Salgın*, Uğur Salgın, Nagihan Tuzlalı

Faculty of Engineering, Department of Chemical Engineering, Cumhuriyet University, 58140 Sivas, Turkey, Tel. +90 346 2191010, ext. 2804; Fax: +90 346 2191179; emails: ssalgin@cumhuriyet.edu.tr (S. Salgın), usalgin@cumhuriyet.edu.tr (U. Salgın), nagehansoyer@hotmail.com (N. Tuzlalı)

Received 5 December 2015; Accepted 25 February 2016

ABSTRACT

In this study, the correct and apparent zeta potential of polyether sulfone (PES) ultrafiltration membranes at different ionic environments were determined using streaming potential measurements. The apparent zeta potential was found by calculation with classic Helmholtz-Smoluchowski (HS) and Fairbrother-Mastin (FM) equations of results obtained from clamping cell (CLC) and adjustable gap cell (AGC). Correct zeta potential was determined by calculation with a modified HS equation of obtained results from AGC. Obtained apparent zeta potential results from CLC and AGC differ from each other, and the reproducibility of results was found to be higher for measurements of AGC. Except for low pH values, apparent zeta potential that was obtained from classic HS equation with AGC could be accepted as correct zeta potential. In CLC measurements, underestimated or overestimated zeta potential values were determined in comparison with the correct zeta potential. The effect of membrane surface conductance on zeta potential decreased with increasing KCl concentration. In CLC, the deviation was observed from electrical double layer theory when KCl concentration was 0.1 M, therefore streaming potential measurements did not produce correct results at higher salt concentration. 0.001 M KCl concentration was the value at which surface conductance became crucial.

Keywords: Zeta potential; Streaming potential; Surface conductance; Adjustable gap cell; Clamping cell

1. Introduction

Membrane separation processes have broad industrial application in fields like chemistry, pharmaceuticals, textiles, paper, and wastewater. Although membrane processes have many advantages, such as low energy consumption, no phase changes, easy scale-up, the fundamental problem of industrial membrane processes when compared with classic

*Corresponding author.

separation techniques is membrane fouling [1–3]. In order to solve this problem, many researchers have used membrane zeta potential as a key parameter [4–8], because membrane and particle zeta potentials provide important information about the nature and magnitude of membrane fouling caused by the membrane-particle interfacial interactions. The correct value of zeta potential is significant for determining the direction of the next steps in the process.

In addition to characteristics of solutions such as ionic strength, pH, and ions type, zeta potential is

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affected by the chemical structure of the surface and surface conductance. The streaming potential technique is generally used for zeta potential measurements of flat surfaces like membranes. In studies where this technique was used to determine zeta potential, different results were found for membranes with the same chemical structure at the same ionic environment. In particular, the reproducibility of measurement results is not good for membranes having surface conductance, and the obtained zeta potentials are not correct zeta potential, they are apparent zeta potential [9–12].

Electrical conductivity characteristics which result from the porous structure of membranes dramatically affect zeta potential, because the conductivity within pores is higher than the conductivity of bulk solution [13]. Therefore, the correct zeta potentials of membranes that have surface or body conductance could not be determined by referring to the classical HS equation [14]. Ignoring a membrane surface conductance causes decreasing reproducibility of measurement results, and also complicates determination of apparent zeta potential which depends on measurement cell design. In the literature, the effects of the surface/body conductance of polymeric and ceramic membranes on zeta potential were experimentally investigated, and the correct zeta potentials of membranes were determined by measurements at various heights of micro flow channel with the approaches that developed from the studies [15–19].

In this study, zeta potentials of PES ultrafiltration membranes were determined by streaming potential measurements with an Electro Kinetic Analyzer (EKA) using VisioLab software program. The apparent zeta potentials of membranes were found by using classical HS and FM equations with the measurement results from clamping cell (CLC) and adjustable gap cell (AGC). Correct zeta potential was found by using a modified HS equation with included effects of surface conductance with the measurement results from AGC. Therefore, this study aims to determine correct zeta potential values and the ionic environment in which membrane surface conductance becomes significant, and also compare the performances of AGC and CLC, and to investigate the reproducibility of measurement results.

2. Materials and methods

2.1. Materials

30 kDa PES membranes (14659-47D, Sartorious) were used in the measurement of zeta potentials. Membranes were washed, respectively, with deionized

water (Milli-Q Gradient, Millipore) and salt solution which was studied, in order to eliminate contamination from production medium and to equilibrate with electrolyte. The salt solutions were prepared with deionized water. Sonication was applied by ultrasonic bath (Elmasonic S 100 H) for 30 min in order to remove air bubbles from salt solutions and to obtain the flow check.

2.2. Measurements of zeta potential with CLC

The apparent zeta potential of PES membranes was determined by calculation with HS and FM equations from experimental results at CLC by using EKA (SurPASS, Anton-Paar, Graz, Austria) device. 0.1 M KOH or 0.1 M HCl were used for adjusting the pH of the solution. Membranes were cut with a punching tool appropriate to the flow channel geometry, and then they were prepared in sandwich form using sealing foil and spacer before being placed in CLC [3]. Schematic representation of CLC is shown in Fig. 1.

The streaming potential measurements have been extensively applied to flat polymer and glass surfaces to study solid–liquid interface electrical properties in a parallel plate microchannel. In this technique, the downstream convection of ions via pressure-driven flow induces a streaming potential which, for steady incompressible and laminar flow, can be related to the ζ -potential via HS equation (Eq. (1)) [20]. Eq. (1) is the classical streaming potential equation, which is valid only if all or almost all of the conduction current is transported by the bulk liquid [21]. A more detailed theoretical model of the streaming potential was described by Erickson and Li [22]:



Fig. 1. Schematic representation of CLC.

$$\zeta = \frac{\mathrm{d}E_{\mathrm{s}}}{\mathrm{d}P} \frac{\mu}{\varepsilon\varepsilon_0} \frac{L}{WhR} \tag{1}$$

The classic HS equation (Eq. (1)) is generally used for calculation of zeta potential, and in this equation there is no term regarding membrane surface conductance. For samples with negligibly small surface conductivity, the zeta potential equation may be rewritten as the Fairbrother–Mastin equation (Eq. (2)). In FM equation, there is a term that includes bulk electrolyte conductivity. The effect of surface conductance on zeta potential is neglected in this equation, referring to the assumption of suppressing surface conductance at high electrolyte concentration [23,24].

$$\zeta = \frac{\mathrm{d}E_{\mathrm{s}}}{\mathrm{d}P} \frac{\mu}{\varepsilon\varepsilon_{\mathrm{o}}} \lambda_{\mathrm{b}} \tag{2}$$

In Eqs. (1) and (2), ζ is the zeta potential, dE_s/dP is the slope of streaming potential vs. differential pressure, μ is the electrolyte viscosity, ε_0 is the vacuum permittivity, ε is the dielectric constant of electrolyte, *L* is the length of the streaming channel, *W* is the width of the streaming channel, *h* is the gap height of the streaming channel, *R* is the electrical resistance inside the streaming channel, and λ_b is the conductivity of bulk electrolyte solution.

2.3. Measurements of zeta potential with AGC

While streaming potential technique is inherently suitable for flat surfaces, accurately measuring the streaming potential may not be simple in practice. Additionally, both ζ and membrane surface conductance must be determined, which requires a series of measurements of $E_{\rm s}$ and ΔP at several different



Adjustable Gap Cell

Fig. 2. Schematic representation of AGC.

channel heights [20]. The correct zeta potential of PES membranes was determined by calculation with modified HS equation (Eq. (3)) from experimental results in AGC by using EKA. Schematic representation of AGC is shown in Fig. 2. Membranes were cut into a size of 20×10 mm and were fixed on the sample holder facing with double-sided adhesive tape.

The gap height between two membranes was adjusted by the knurled nuts of measurement cell. These measurement results were used in Eq. (4), which was obtained by linearization of the modified HS equation (Eq. (3)) [25]:

$$\zeta = \frac{dE_{\rm s}}{dP} \frac{\mu}{\varepsilon \varepsilon_0} \frac{L}{WhR} = \frac{dE_{\rm s}}{dP} \frac{\mu}{\varepsilon \varepsilon_0} \left(\lambda_{\rm b} + 2\frac{\lambda_{\rm s}}{h}\right) \tag{3}$$

$$\frac{\varepsilon\varepsilon_{\rm o}}{\lambda_{\rm b}\mu} \left(\frac{\mathrm{d}P}{\mathrm{d}E_{\rm s}}\right) = \frac{1}{\zeta} + \left(\frac{2\lambda_{\rm s}}{\zeta\lambda_{\rm b}}\right)\frac{1}{h} \tag{4}$$

where λ_s is the membrane surface conductance.



Fig. 3. The change of apparent zeta potentials of PES membranes with the change of pH at different KCl concentrations: (a) HS equation and (b) FM equation.

The parameter $(\varepsilon \varepsilon_o / \lambda_b \eta (dP/dE_s))$ is plotted as a function of 1/h, and linear regression is performed. According to Eq. (4), the *y*-intercept of the regressed line is then related to the correct zeta (ζ_c) potential and its slope to the membrane surface conductance (λ_s) [25]. Furthermore, the data obtained from AGC was used for calculating the apparent zeta potential by using Eqs. (1) and (2) for 100 µm gap height. Therefore, the ionic environment conditions which give

results close to the correct zeta potential, and type of measurement cell, were determined.

3. Results and discussion

3.1. Determination of apparent zeta potentials with CLC

The apparent zeta potentials which were calculated by HS and FM equations (Eqs. (1) and (2)) using the



Fig. 4. Determination of the zeta potential and surface conductance from Eq. (4): 0.001 M KCl: (a) pH 3, (b) pH 4, (c) pH 6, (d) pH 8, and (e) pH 10.

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streaming potential results of sandwich-formed PES membranes in different KCl concentrations and pH values are given in Fig. 3.

The zeta potential of PES membranes calculated by HS and FM equations were negative in all test conditions. The zeta potentials determined by HS and FM equations became more negative as the pH increased in all KCl concentrations. The absolute zeta potential calculated according to the HS equation was 10-fold bigger than the zeta potential calculated by FM in all KCl concentrations. This difference is attributed to the terms present in the HS and FM equations. The effective parameters in HS equation are the geometry of the micro flow/streaming channel ($L \times W = 25 \times 5$ mm) in the measurement cell and the electrical resistance (*R*) inside the streaming channel. The effective parameter in FM equation is the conductivity of the bulk electrolyte solution. It was



Fig. 5. Determination of the zeta potential and surface conductance from Eq. (4): 0.01 M KCl: (a) pH 3, (b) pH 4, (c) pH 6, (d) pH 8, and (e) pH 10.

considered that the one reason for obtaining different zeta potentials from HS and FM equations was the different ionic environment in the streaming channel from in the bulk solution.

The effect of KCl concentration on zeta potential which was calculated from HS and FM equations gave similar results. With increasing KCl concentration from 0.001 to 0.01 M, the absolute value of zeta potential decreased, so it was consistent with electrical

double layer (EDL) theory. According to the EDL theory, the increase in the KCl concentration, i.e. the increase at ionic strength, caused the compression of the diffuse layer, so caused a lower absolute value of zeta potential by decreasing the thickness of the double layer [3,4,26]. However, when the KCl concentration was increased to 0.1 M, an increase in absolute values of zeta potentials was observed, in contrast to the EDL theory. The concentration of Cl^- anions



Fig. 6. Determination of the zeta potential and surface conductance from Eq. (4): 0.1 M KCl: (a) pH 3, (b) pH 4, (c) pH 6, (d) pH 8, and (e) pH 10.

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increased in solution with increasing KCl concentration up to 0.1 M. Because anions are less hydrated relative to the cations, anions can approach closer to the surface of the membrane. The membrane gains more zeta potentials thanks to this approach of anions toward the shear plane [11,22]. Therefore, the zeta potential at 0.1 M KCl was observed to be more negative than at 0.001 M KCl solution.

3.2. Determination of correct zeta potentials with AGC

The correct zeta potentials and surface conductance values of the membranes were determined by estimating results in different pH and KCl concentrations with Eq. (4). Measurements at different gap heights h = 100, 120, 140, and 150 µm were carried out for all KCl concentrations.

The graphs drawn for pH 3, 4, 6, 8, and 10 at the KCl concentrations, respectively, 0.001, 0.01, and 0.1 are presented in Figs. 4–6. The r^2 values of straight lines seen in the graphics varied between 0.91 and 0.99. This linearity shows that the laminar flow conditions were provided for the use of Eq. (4) at different gap height values [11].

The correct zeta potentials (ζ_c) and membrane surface conductance (λ_s) values calculated from the graphs in each condition are given in Table 1. As the pH increased, a rising concentration of OH⁻ ions caused an increase in the absolute values of ζ_c at all KCl concentrations. When the concentration of KCl increased from 0.001 to 0.01 M, the absolute ζ_c values decreased (consistent with the EDL theory). The obtained ζ_c values at 0.1 and 0.01 M KCl solution were close to each other. It was observed that λ_s values increased with increases in the KCl concentration. As the λ_s values generally decreased with increasing pH at 0.001 and 0.01 M KCl and λ_s values were increased with increasing pH at 0.1 M KCl solution.

Table 1	
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The	change	of	correct	zeta	potential	and	surface	conduc-
tance	e with j	эΗ	in differ	ent k	Cl concer	ntrati	ons	

	0.001 M	KCl	0.01 M k	KC1	0.1 M KCl	
pН	$\zeta_{\rm c}$ (mV)	$\lambda_{\rm s}$ (mS)	$\zeta_{\rm c}~({\rm mV})$	$\lambda_{\rm s}$ (mS)	$\zeta_{\rm c}~({\rm mV})$	$\lambda_{\rm s}$ (mS)
3	-17.51	0.0927	-9.00	0.5520	-5.48	0.313
4	-24.15	0.0034	-12.78	0.0651	-15.93	0.295
6	-38.16	0.0008	-26.28	0.0532	-26.51	0.529
8	-69.93	0.0014	-37.16	0.0390	-39.27	1.297
10	-68.49	0.0009	-39.26	0.0344	-42.58	1.754

3.3. Comparison of CLC and AGC measurements results

As the apparent zeta potentials collected from the measurements in CLC and AGC with streaming potential method were calculated by classic HS (Eq. (1)) and FM (Eq. (2)) equations, correct zeta potentials calculated by modified HS (Eq. (4)) equation after the measurements in AGC at different gap height values.

All the results obtained from each ionic environment are presented in Tables 2–4 for the comparison of performance two cells. While the zeta potential values calculated with HS and FM (ζ_{HS} and ζ_{FM} respectively) with AGC were close to each other, with CLC, ζ_{HS} and ζ_{FM} values obtained from both equations were quite different from each other. This closeness of the ζ_{HS} and ζ_{FM} values with AGC shows that membrane surface conductance could be ignored beside the bulk conductivity value [16].

In AGC at 0.001 M KCl, absolute ζ_{HS} values were 1.4-fold higher than the values of ζ_{FM} , while at 0.01 M and 0.1 M KCl they were 1.3 and 1.12-fold higher, respectively. It could be said that as the concentration of KCl increases, the effect of surface conductance on zeta potentials is reduced. It was determined that the surface conductance was effective at low KCl concentrations and this finding matches with the literature [16,27].

The measurement results carried out at AGC were evaluated in two different aspects; Eq. (4) was used to determine the correct zeta potential (ζ_c) and Eqs. (1) and (2) were used to determine the apparent zeta potential. When the results in the tables were evaluated, ζ_c values at 0.001 M KCl except at pH 3 and 4 and at 0.01 M KCl except at pH 3 were approximately equal to the apparent zeta potential (ζ_{HS}) values calculated by the equation HS. In these salt concentrations in which the surface conductance is more crucial, the values (ζ_{HS}) obtained by the equation HS were approved as correct zeta potential. At the 0.1 M KCl concentration where the effect of surface conductance was more reduced, the ζ_c values except in pH 3 were approved approximate to the ζ_{HS} and ζ_{FM} values. Therefore, the values of $\zeta_{\rm HS}$ and $\zeta_{\rm FM}$ which were obtained from the equation HS or FM are accepted as correct zeta potentials.

When the CLC results were examined, it is observed that absolute value of $\zeta_{\rm HS}$ was nearly 10-fold higher than the $\zeta_{\rm FM}$ values. When these two calculated values, $\zeta_{\rm c}$ and $\zeta_{\rm HS}$ were compared, it was observed that the absolute values of $\zeta_{\rm HS}$ in all KCl concentrations were overestimated, $\zeta_{\rm FM}$ values were underestimated with regard to $\zeta_{\rm c}$ values at 0.001 and 0.01 M KCl and the $\zeta_{\rm FM}$ values were closer to the $\zeta_{\rm c}$ values at 0.1 M KCl. When the $\zeta_{\rm HS}$ values was depended on the

	AGC ^a		CLC			
pН	$\zeta_{ m HS}$ (mV)	$\zeta_{\rm FM}~({ m mV})$	$\zeta_{\rm HS}$ (mV)	$\zeta_{\rm FM}~({ m mV})$	$\zeta_{\rm c}~({\rm mV})$	$\lambda_{\rm s}$ (mS)
3	-0.94	-0.69	-68.51	-6.88	-17.51	0.0927
4	-13.08	-9.87	-103.38	-12.29	-24.15	0.0034
6	-38.09	-27.90	-135.60	-13.42	-38.16	0.0008
8	-62.29	-37.85	-138.95	-13.90	-69.93	0.0014
10	-65.69	-44.75	-142.00	-15.45	-68.49	0.0009

Table 2 The changes of apparent, correct zeta potentials and λ_s values of PES membranes with pH 0.001 M KCl

 ${}^{a}h = 100 \ \mu m.$

Table 3

The changes of apparent, correct zeta potentials and λ_s values of PES membranes with pH 0.01 M KCl

	AGC ^a		CLC		$\zeta_{\rm c}$ (mV)	$\lambda_{\rm s}$ (mS)
pН	$\zeta_{\rm HS}$ (mV)	$\zeta_{\rm FM}~({ m mV})$	$\zeta_{\rm HS}$ (mV)	$\zeta_{\rm FM}$ (mV)		
3	-0.728	-0.56	-55.59	-4.50	-9.00	0.5520
4	-10.31	-7.927	-76.92	-7.06	-12.78	0.0651
6	-27.96	-20.29	-118.00	-9.89	-26.28	0.0532
8	-36.10	-27.73	-121.32	-9.83	-37.16	0.0390
10	-38.75	-29.71	-125.06	-10.31	-39.26	0.0344

 ${}^{a}h = 100 \ \mu m.$

Table 4

The changes of apparent, correct zeta potentials and λ_s values of PES membranes with pH 0.1 M KCl

рН	AGC ^a		CLC		ζ _c (mV)	$\lambda_{\rm s}$ (mS)
	$\zeta_{ m HS}$ (mV)	$\zeta_{\rm FM}~({ m mV})$	$\zeta_{\rm HS}~({ m mV})$	$\zeta_{\rm FM}~({ m mV})$		
3	-14.00	-12.22	-111.60	-10.24	-5.48	0.313
4	-17.00	-14.73	-136.60	-13.80	-15.93	0.295
6	-28.00	-25.00	-198.20	-20.60	-26.51	0.529
8	-40.00	-37.20	-260.20	-28.06	-39.27	1.297
10	-43.33	-41.00	-430.20	-46.40	-42.58	1.754

 ${}^{a}h = 100 \ \mu m.$

micro flow channel geometry of the measurement cell and the cell resistivity, ζ_{FM} values were only depended on the conductivity of the bulk electrolyte solution.

4. Conclusion

It was considered that the main reasons for obtaining different zeta potential values which were determined from CLC and AGC, were the different geometry of the micro flow channel and the size of sample used. Dimensions of micro flow channel in both cells are different, which influence flow properties into the electrokinetic channel. It is conceivable that the applicability of assumptions for Eqs. (1) and (2) are invalid. Also, the conductivity of the micro channel may be significantly affected by surface conduction especially for small channels and low ionic strengths [28]. The sample size has to be $55 \times 25 \text{ mm}^2$ in order to make an estimate at CLC, however about 9% of this stated size was used at streaming potential measurement. Therefore, the sample size demanded from CLC was higher than the needed sample size for calculation of zeta potential. In the case of AGC, the whole sample surface cut in $20 \times 10 \text{ mm}^2$ dimension, forms the micro flow channel and it is used for the investigation of zeta potential. All streaming potential

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measurements were performed at least six streaming potential measurements (three flowing from left to right and three from right to left) and then averaged to calculate the zeta potential. The reproducibility of results from AGC and CLC were tested. The average changes were 5 and 12% with AGC and CLC, respectively. Hence AGC has to be preferred, considering the reproducibility of the results. At the concentrations of 0.001 and 0.01 M KCl, the correct zeta potential could be obtained except in low pH without the necessity of using modified HS equation. At 0.1 M KCl, the zeta potentials ζ_{HS} and ζ_{FM} could be approved as correct zeta potential. The effect of surface conductance was observed at AGC, similar effect was not observed at CLC because of different values obtained from HS and FM. 0.001 M KCl concentration is the value that became the important parameter for PES membrane surface conductance.

Acknowledgment

This study was financially supported by The Scientific and Technological Research Council of Turkey (TÜBİTAK) with the project number 113M047.

List of symbols

dE_s/dP	_	slope of streaming potential vs.
		differential pressure (mV/mbar)
h		gap height of the streaming channel (μm)
L		length of the streaming channel (mm)
R		electrical resistance inside the streaming
		channel (kOhm)
W		width of streaming channel (mm)
З		dielectric constant of electrolyte
E _O		vacuum permittivity (C/V m)
ζ		zeta potential (mV)
λ _b		conductivity of bulk electrolyte solution
		(mS/m)
λ_s		membrane surface conductance (mS)
μ		electrolyte viscosity (mPa s)

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