Scientific paper

Zeta Potential Determination of Polymeric Materials Using Two Differently Designed Measuring Cells of an Electrokinetic Analyzer

Hermina Bukšek,[1] Thomas Luxbacher[2] and Irena Petrinić[1],*

[1] University of Maribor, Faculty of Chemistry and Chemical Engineering, Smetanova 17, SI-2000 Maribor, Slovenia

[2] Anton Paar GmbH, Anton Paar Straße 20, A-8054 Graz, Austria

* Corresponding author: E-mail: irena.petrinic@uni-mb.si

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Abstract

The so-called zeta potential can be determined through electrokinetic measurements and indicates the status regarding surface charges along the interface between solids and liquids. Surface charge gives us information about the condition, quality, and characteristics of a macroscopic surface in the polar medium. In our study the zeta potential was determined using a "SurPASS" electrokinetic analyzer based on the streaming current and streaming potential measurements. The aim of the research was to compare the results of two differently designed measuring cells ("Adjustable Gap Cell" and "Clamping Cell") but operating on the same principle. In order to investigate this problem, the zeta potential was determined for the three polymeric materials: poly(ethylene terephthalate) foil, thin-film polyamide composite membranes for nanofiltration and reverse osmosis. The results obtained with "Clamping Cell" versus "Adjustable Gap Cell" showed differences in zeta potential, where the "Adjustable Gap Cell" gave more reproducible results. One reason for this behaviour could be the different geometries of the streaming channels. A more likely reason is the design of the "Clamping Cell", that requires a sample size larger than necessary for zeta potential determination.

Keywords: Electrical double layer, zeta potential, electrokinetic analyzer, streaming current/potential, polymeric materials

1. Introduction

The zeta potential, also known as the electrokinetic potential, originates from the accumulation of electrical charges at a solid/liquid interface where an electrical double layer is formed. As an interfacial parameter the zeta potential is thus influenced by the properties of the solid surface and the surrounding liquid. It provides insight into the charge and adsorption characteristics of solid surfaces. The determination of zeta potential is applied across various fields of basic research in physics, chemistry, and biology, as well as for the scientific exploration of technological processes. Among these processes, zeta potential determination is applied for the characterization of natural and synthetic fibres[1–2], membranes and filters[3–4], textiles[5], hair[6], or biomaterials[7].

The zeta potential is an experimentally accessible parameter which can be determined using several types of electrokinetic phenomena.[8] For flat solid surfaces the zeta potential is calculated from the measurement of streaming potential or streaming current.[3,9–12]

A streaming potential is generated when an electrolyte solution is forced, by means of hydraulic pressure, to flow through a porous plug of material, across a channel formed by two plates, or down a capillary. The liquid in the channel carries a net charge. Its flow, due to hydraulic pressure, gives rise to a streaming current, thereby generating a potential difference. This potential opposes the mechanical transfer of charge, causing back-conduction by ion diffusion and electro-osmotic flow (due to the potential difference). The transfer of charges due to these two processes is called the leak current. When equilibrium condition is attained, the streaming current cancels the leak current, and the measured potential difference is the streaming potential.[3,13]

Until recently, zeta potential was determined using electrokinetic analyzers based solely on the streaming-potential technique. Such commercial instruments are the "EKA" Electro Kinetic Analyzer (Anton Paar GmbH, Austria) and the "ZetaCAD" zetameter (CAD Instrumentation, France). In these cases, only an apparent zeta potential is determined following the approximation of the Helmholtz-Smoluchowski equation[14]. Here the measured streaming potential is related to the specific conductivity of the electrolyte solution. The influence of interfacial conductance is taken into account after correction, using the approach of Fairbrother and Mastin[15], Eq.5 in Fig. 1.

Several years of experience using the "EKA" electrokinetic analyzer have resulted in the development of the new instrument "SurPASS"[16]. Zeta potential determination using the "SurPASS" electrokinetic analyzer is based on the measurements of streaming potential and streaming current, respectively. The latter enables the calculation of a correct zeta potential without approximation.

The availability of streaming current measurement, in addition to determining the exact geometry of a rectangular streaming channel, enables a deeper insight into the electrokinetic behaviour at the solid/liquid interface between planar surfaces and a surrounding aqueous solution[17]. Recently the first results obtained with the "SurPASS" electrokinetic analyzer for thin-film composite membranes were published in the literature[18–19]. Differently from previous zeta potential results, the use of streaming current measurements revealed significantly higher negative zeta potential, which were interpreted in terms of so-called membrane body conductance. Although this effect has been predicted by Yaroshchuk et al.[20] and an experimental confirmation was expected, the magnitude of this effect was surprisingly high. On the other hand, Makdissy et al. compared zeta potential results for clean and fouled NF and RO membranes determined with "EKA" and "SurPASS", respectively[19]. The results obtained with "EKA" showed a more negative zeta potential for the same type of membrane, and the same principle of the applied measuring cells. Makdissy et al. interpreted this discrepancy in terms of the different geometries of the streaming channels and, thus, of the different cell constants *L*/*A* with *L* and *A* being the length and cross-section of the rectangular slit between adjacent membrane samples[19]. Although this interpretation is reasonable since streaming potential measurement was used to calculate an apparent zeta potential, the measuring conditions (ionic strength of electrolyte, height of the streaming channel) do not explain the magnitude of this difference.

This paper questions the conventional evaluation of zeta potential for flat-sheet polymer membranes from streaming potential and streaming current measurements by comparing results obtained with different measuring cells but based on the same principle of a rectangular slit.

2. Evaluation Methods for Zeta Potential Determination

As mentioned in the "Introduction", the zeta potential is calculated using the Helmholtz-Smoluchowski (H-S) equation. Depending on the available measuring parameter, an approximated equation is used leading to an apparent zeta potential.

The relationship between the measured streaming current or streaming potential[21] and the zeta potential is

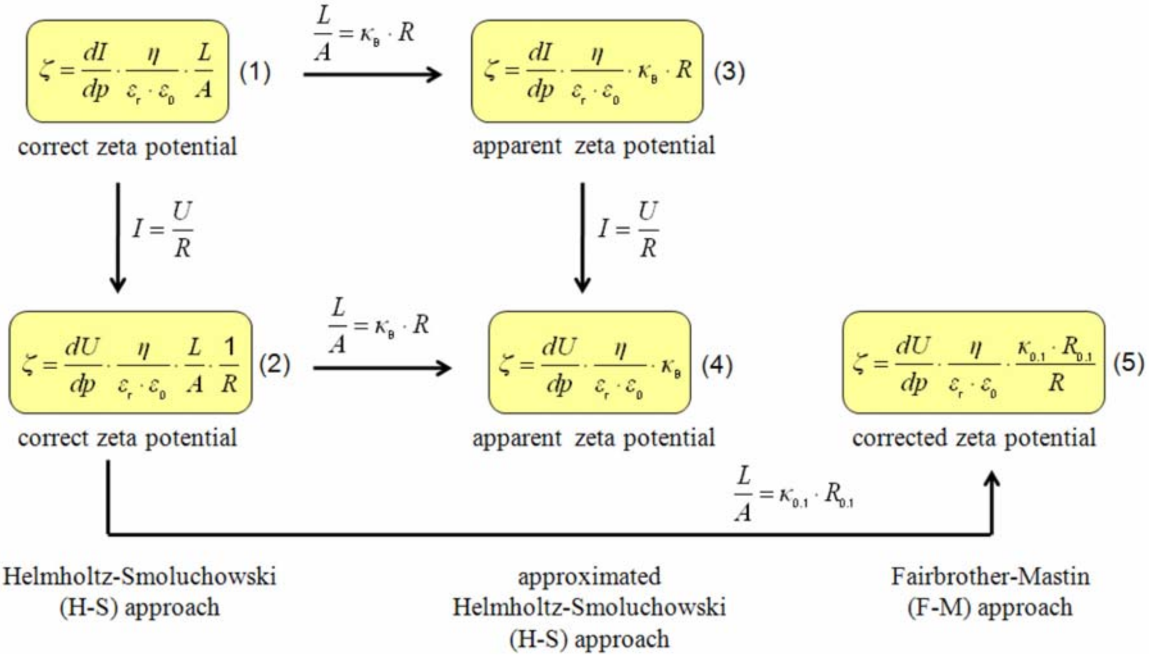

Figure 1: Different equations for zeta potential determination.

given by the equations presented in Figure 1, where ζ is the zeta potential, *dI/dp* the slope of streaming current versus pressure, *dU/dp* the slope of streaming potential versus pressure, η the electrolyte viscosity, ε_r the relative liquid permittivity, ε_0 the vacuum permittivity, *L* the length of the streaming channel, *A* the cross-section of the streaming channel, *R* the DC resistance inside the measuring cell, and κ_B the specific conductivity of the bulk electrolyte solution.

In its initial form (Eq.1, 2) the H-S approach determines the zeta potential in regard to the geometry of the streaming channel. Here the zeta potential is calculated without approximation. In its second (approximated) form (Eq.3, 4) the H-S approach determines the zeta potential in regard to the specific conductivity of the bulk electrolyte solution. This approach assumes that the electrical current inside the measuring cell is conducted by the aqueous electrolyte solution only. However, depending on the measuring conditions, the effect of interfacial conductance may contribute significantly to the streaming current (or to the leak current for streaming potential measurement), and the zeta potential calculated according to Eq.3 or Eq.4 thus becomes underestimated. The possible error of this approximation is corrected by the approach of Fairbrother and Mastin (F-M), Eq.5, which determines the cell constant *L*/*A* from resistance and (electrolyte) conductivity measurements under conditions where interfacial conductance becomes negligible (e.g. at an ionic strength of 0.1 mol/L).

The apparent zeta potential may be underestimated and the determination of a correct zeta potential is, therefore, an important advantage of the "SurPASS" electrokinetic analyzer in comparison with previous electrokinetic analyzers.

3. Experimental

3. 1. Materials

The commercial thin-film composite (TFC) polymer membranes BW-30 (RO membrane, Dow Filmtec, USA) and NFT-50 (NF membrane, Alfa Laval, Denmark) were used for investigating the effects of different measuring cell's designs on the magnitude of the membrane zeta potential. TFC polymer membranes are composed of a dense polyamide layer ("active side") deposited on a polysulfone UF membrane, and a polyester non-woven for mechanical support. In addition the zeta potential of a poly(ethylene terephthalate) foil (GoodFellow Corp., UK) was determined in order to compare the membranes with a nonporous polymer material.

Dry PET samples were mounted, while the membranes were soaked prior to measurement in order to achieve wetting of the membrane support. Thus, before each measurement, the two pieces of composite membrane were soaked for at least 24 hours in the electrolyte solution used during the measurement. Experiments at pH 5.0 +/– 0.2 were performed without any pH adjustment. Solutions of KCl (1 mM, 5 mM or 10 mM) served as the electrolyte for the measurements of membranes. In the case of PET, the measurements were conducted in a 1 mM KCl background solution only. This solution was prepared using high-purity water (Milli-Q, Millipore).

3. 2. Zeta Potential Analysis

The "SurPASS" instrument includes an analyzer, a data control system, and a measuring cell appropriate for the solid sample. This instrument measures the streaming current and streaming potential resulting from the pressure-driven flow of an electrolyte solution that passes through a thin slit channel formed by two identical sample surfaces.

The zeta potential of flat surfaces can be determined using two different rectangular measuring cells: the "Clamping Cell" (CLC) and the "Adjustable Gap Cell" (AGC). The sample mounting is schematically shown in Fig. 2.

For CLC, two samples with 55 mm × 25 mm are mounted opposite of each other and separated by a spacer. In the CLC, an area of only 25 mm × 5 mm of each sample contributes to the measurement (9% of total sample

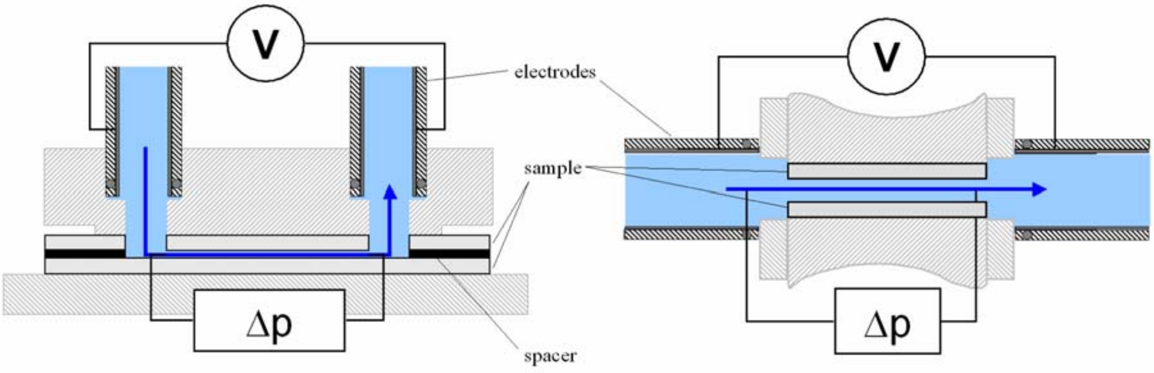

Figure 2: Schematic representation of tangential measuring technique in the "Clamping Cell" (left) and the "Adjustable Gap Cell" (right) of the "SurPASS" instrument.

area). For the AGC two samples with 20 mm × 10 mm are fixed on sample holders using double-sided adhesive tape. The distance between the samples' surfaces is then adjusted continuously. For AGC, the complete sample surface is used for measurement (100% of total sample area).

The differences between a "Clamping Cell" and an "Adjustable Gap Cell" are, therefore, in the geometries of the streaming channels and in the size of sample used. Both measuring cells are designed for suitable flow-rate and pressure to ensure laminar flow. The height of the rectangular channel H is determined from the measurement of flow-rate dV/dt and the differential pressure according to Hagen–Poiseuille equation (Eq. 6),

$$H = \sqrt[3]{\frac{12 \cdot \eta \cdot L}{W} \cdot \frac{dV/dt}{\Delta p}}, \tag{6}$$

where W is the width of the streaming channel.

The cell height for a "Clamping Cell" is about 75 µm, and for an "Adjustable Gap Cell" about 100 µm. At this distance between adjacent sample surfaces, a strong contribution of interfacial conductance is not assumed, even at the used lowest ionic strength of 10^{-3} mol/L.

Measurements were made at 24 ± 2 °C and for a maximum pressure difference of 500 mbar with flow in the two directions. At least 18 experimental points were collected for each run.

4. Results and Discussion

The results for the evaluation of the differently designed measuring cells, "Clamping Cell" and "Adjustable Gap Cell", were obtained on six samples for each of the selected polymeric materials. Six measurements were performed for each sample in order to determine measurement repeatability. These individual measurements were repeated three times over a period of 30 minutes, in order to exclude the effect of measuring time. This procedure added up to 18 measurement points for every specimen. As a representative example, Figure 3 shows a series of 6 streaming current measurements for membrane BW-30, using CLC and AGC. The zeta potential was evaluated by Eq. 1.

It becomes obvious from Figure 3 that the zeta potential of RO membrane BW-30 differs significantly when being determined in either AGC or CLC. The zeta potential for AGC was found to be within the range of –15.4 and –19.5 mV, with an average standard deviation of 1.7%. On the other hand, the zeta potential is more negative for CLC and determined within the range of –63.1 mV and –40.3 mV. Although the average standard deviation for CLC is 3.0% and thus slightly higher than the repeatability of AGC, it remains within the same range of measuring error. The reason for the strong deviation between zeta potential values for CLC and AGC is, therefore, unexplained by measuring uncertainty. When comparing the reproducibility of measurements for membrane BW-30 for measuring cells a more significant parameter is identified for describing the difference between AGC and CLC. The reproducibility for zeta potential determined for 6 individual membrane samples in AGC is 8.8%, while for CLC it rises to 18%. For further discussion, the average zeta potential and reproducibility was therefore determined for six individual samples. The average zeta potential values for PET, BW-30, and NFT-50 as determined from Eq.1, and the corresponding standard deviation, are summarized in Table 1.

In order to compare the effect of the approximation of Eq.1 on the magnitude of the zeta potential, i.e., comparison between correct and apparent zeta potential, the streaming potential was measured for the same samples and the zeta potential was calculated according to Eq.4. Figure 4 shows the comparison between those correct and apparent zeta potential values for membrane BW-30 determined in the AGC.

Again a difference is found between the zeta potential calculated from Eq.1, i.e. without approximation, and the apparent zeta potential calculated from Eq.4. The apparent zeta potential was less negative than the correct one for all six samples tested. On average, the apparent zeta potential is 90% of the correct value. The consistency of

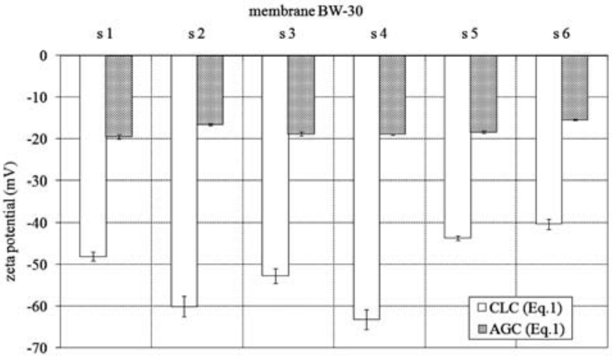

Figure 3: Zeta potential of membrane BW-30 obtained with "Adjustable Gap Cell" and "Clamping Cell" using Eq. 1.

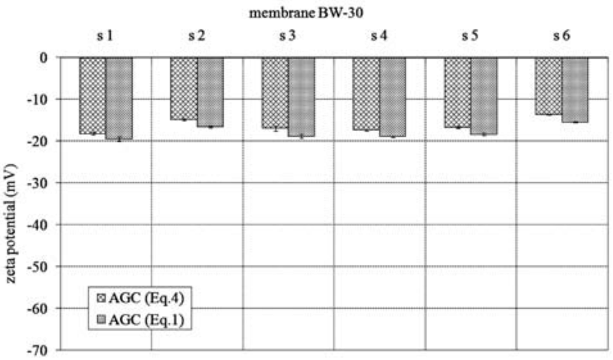

Figure 4: Zeta potential of BW-30 membrane obtained with "Adjustable Gap Cell" using Eq. 4 and Eq. 1.

Table 1: Zeta potential of polymeric materials PET, BW-30, and NFT-50, obtained with measuring cells AGC and CLC in 1 mM KCl solution, and evaluated using equations Eq. 1 and Eq. 4.

Measuring cell	Used equation	Zeta potential (mV) PET	 BW-30	 NFT-50
CLC	Eq.1	–44.10 ± 9.60	–51.30 ± 9.04	–84.41 ± 11.07
	Eq.4	–35.52 ± 5.72	–8.55 ± 2.05	–29.45 ± 2.73
AGC	Eq.1	–33.62 ± 2.24	–17.92 ± 1.58	–36.19 ± 6.06
	Eq.4	–35.81 ± 3.57	–16.26 ± 1.71	–32.40 ± 5.43

Data from Table 1, are shown schematically in Figure 5.

this difference together with the small standard deviation for individual samples, excludes any experimental error being responsible for this observation.

Table 1 summarizes the average zeta potential calculated from Eq.1 and Eq.4, respectively, and the corresponding standard deviation for the measurements of 6 samples for PET, BW-30, and NFT-50. The zeta potential data is also shown in Figure 5.

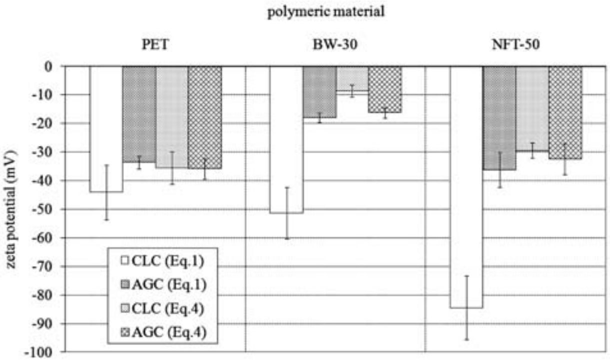

Figure 5: Zeta potential of selected polymeric materials obtained with AGC and CLC cells using Eq. 4 and Eq. 1

In regard to the average zeta potential for membranes BW-30 and NFT-50, respectively, the same trend is found when comparing AGC and CLC. The zeta potential calculated by Eq.1 is always more negative when determined in CLC compared to AGC (Fig. 5). On the other hand, the apparent zeta potential calculated by Eq.4 is less negative for CLC compared to AGC. This is most obvious for membrane BW-30, whereas the difference is within the experimental error for membrane NFT-50. For the nonporous PET foil, a difference is again found for the zeta potential calculated by Eq.1 between CLC and AGC, whereas any differences among the remaining zeta potential values disappear. It is noticeable that the biggest error for reproducibility occurs for the zeta potential of PET, as determined in the CLC using Eq.1. Due to this error the differences between correct and apparent zeta potential values for PET in CLC are statistically insignificant. In summary, the same zeta potential is found for the non-porous polymer foil PET, independently of the selected measuring cell, and the evaluation method. However, for both membranes BW-30 and NFT-50, the zeta potential determined in the CLC using Eq.1 is significantly higher in magnitude compared to the zeta potential evaluated using the other approaches. It also appears that the apparent zeta potential determined with CLC for membrane samples is less negative than any of the zeta potential values, correct and apparent, as determined in the AGC.

A detailed analysis of the sample mounting in the different measuring cells and the composition of the membrane sample is needed in order to explain this observation. The reason for the lower apparent zeta potential compared to the correct zeta potential is an underestimation of the electrolyte conductivity inside the measuring cells. Since the same ionic strength of the electrolyte was used, being an aqueous 1 mmol/L KCl solution for measurements in both measuring cells, the underestimation should have remained within the same range. For membrane BW-30, the ratio between apparent and correct zeta potential for CLC is only 17%, whereas it is 90% for AGC. If we consider the effect of membrane body conductance as a significant contribution to the overall conductance in the streaming channel, and compare the ratio between the soaked membrane area and the membrane area used for measurement, it is possible to estimate the apparent zeta potential as 8% of the correct zeta potential for measurement in the CLC. This is only 50% of the ratio determined experimentally. Inhomogeneous soaking of the membrane sample and or a membrane area slightly smaller than the standard size, may compensate for this deviation.

The effect of the soaked membrane's conductivity does not explain the significant difference between the correct zeta potential values determined with AGC and CLC. Recently Yaroshchuk and Luxbacher[22] introduced a new concept for separating the contributions of external and internal (pore) surface charge to the overall streaming current measurement. They found a significant contribution of streaming current inside pores for microfiltration membranes, depending strongly on the pore size. Although the NF and RO membranes investigated in this paper are considered as dense membranes, the contribution of the porous support must not be neglected when determining the zeta potential. For the tangential method of streaming potential and streaming current measurement, a pressure difference is applied between both sides of the streaming channels but simultaneously between both ends of the porous membrane samples. This pressure difference generates a significant flow of electrolyte inside the streaming channel but a streaming current both inside this channel (which dominates), and inside the porous membrane support. In addition to the streaming current inside the streaming channel and inside the membrane pores, the membrane sample mounting in the CLC is likely to introduce another path for streaming current (which affects the zeta potential evaluation using Eq.1), and conductivity (which adds to the underestimation of the apparent zeta potential according to Eq.4). While the contact between the membrane support and the sample holder is impermeable for membrane mounting in the AGC, an electrolyte film may be formed underneath the soaked membrane when mounted in the CLC. During measurement, the liquid-flow and differential pressure feeds this liquid film by water penetrating the membrane layer. Using these analyses of contributions of possible streaming channels inside the CLC to the streaming current and total conductivity, respectively, it is possible to critically review the literature data available for membrane NFT-50. Table 2 summarizes the zeta potential found in the literature for membranes NFT-50 and BW-30 at different ionic strengths, mainly using the CLC-type of measuring cell and evaluation of the zeta potential based on the streaming potential measurement.

For membrane BW-30, very similar zeta potential is found at an ionic strength of 10^{-2} mol/L. In order to compare the zeta potential obtained for this membrane using the “Adjustable Gap Cell” with literature data, the measurement of streaming current and streaming potential was repeated in 5×10^{-3} mol/L KCl and 10^{-2} mol/L KCl, respectively. It was found that $\zeta = -20$ mV (–16 mV) at 5×10^{-3} mol/L and $\zeta = -17$ mV (–17 mV) in 10^{-2} mol/L KCl using Eq.1 (Eq.4). Although the expected decrease in the negative zeta potential with increasing ionic strength is unobserved, the difference between the apparent and correct zeta potential values diminishes. The dependence of membrane body conductance does not follow the same dependence on the electrolyte conductivity as the electrical conductance inside the streaming channel. Another explanation is the increasing contribution of streaming potential inside the porous support of the RO membrane to the total streaming potential due to a suppression of the interfacial conductance inside pores with increasing ionic strength.

The scatter of zeta potential data presented in the literature is significant for membrane NFT-50. Zeta potential can be found that differ by factors 2–4. However, even the most negative zeta potential reported, $\zeta = -15$ mV, is only 50% of the value determined in the “Adjustable Gap Cell”. Although literature data is only reported for 10^{-3} mol/L KCl, the measurements of streaming current and streaming potential were repeated at higher ionic strength in order to compare with those results obtained for membrane BW-30. Interestingly, the zeta potential for NFT-50 decreases as expected from $\zeta = -36$ mV (–32 mV) in 10^{-3} mol/L KCl to $\zeta = -27$ mV (–25 mV) in 5×10^{-3} mol/L KCl and $\zeta = -18$ mV (–19 mV) in 10^{-2} mol/L KCl. Again the difference between zeta potential calculated by Eq.1 and Eq.4 (data in brackets) decreases with increasing ionic strength. In summary, the zeta potential found in the literature is always smaller than the zeta potential determined with the AGC using both Eq.1 and Eq.4. It can, therefore, be concluded that a measuring cell with the principle based on sample sheets separated by a spacer is unsuitable for the zeta potential determination of membrane surfaces. Taking into account these considerations, the determination of zeta potential for flat-sheet membranes is more reliable when using the “Adjustable Gap Cell”.

5. Conclusions

We have compared the zeta potential determined from streaming current and streaming potential measurements using two differently designed measuring cells for solid samples with a planar surface and of rectangular shape. Although a random selection of any of these measuring cells, the “Clamping Cell” or the “Adjustable Gap Cell”, seems adequate for the zeta potential analysis of flat sheet membranes, a comparison of data evaluated by

Table 2: Zeta potential for membranes BW-30 and NFT-50 reported in the literature.

Electrolyte	Type of measuring cell	Used equation	Zeta potential (mV)	Reference
BW-30				
10 mM KCl	CLC	4	–5	[23]
10 mM KCl	CLAMP [a]	5	–3	[24]
NFT-50				
1 mM KCl	RECT [b]	5	–7	[25]
1 mM KCl	home-made RECT	5	–15	[26]
1 mM KCl	home-made RECT	4	–4	[27]

[a] A single membrane sample is measured vs. a poly(methyl methacrylate) reference surface.

[b] The dimensions of the rectangular cell differ significantly from the CLC and introduce an even higher contribution of both excess membrane area and liquid film between cell body and membrane support.

using equations based on different assumptions demonstrates significant differences. These differences are interpreted in terms of contributions from the porous support of thin-film composite polymer membranes to the streaming current, streaming potential, and electrical conductance inside the rectangular slit between adjacent membrane surfaces. A second contribution to the discrepancy of zeta potential determined in CLC and AGC arises from the excessive area of the membrane mounted in the CLC that does not contribute to the streaming current (streaming potential) measurement but to membrane body conductance. Since this contribution is hard to control, the "Adjustable Gap Cell" is recommended for the zeta potential determination of flat sheet membranes.

6. Acknowledgement

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Povzetek

S pomočjo elektrokinetičnih meritev lahko določimo t. i. potencial zeta, ki določa stanje površinskega naboja na mejni površini trdnega telesa in tekočine. Podatki o površinskem naboju nam dajejo informacijo o stanju, kvaliteti in lastnostih površine makroskopskega telesa v polarnem mediju. V naši raziskavi smo potencial zeta določali z elektrokinetičnim analizatorjem »SurPASS«, ki temelji na merjenju pretočnega toka in pretočnega potenciala. Namen raziskave je bil primerjati rezultate, dobljene z različno oblikovanima merilnima celicama (»Adjustable Gap Cell« in »Clamping Cell«), ki delujeta na istem principu. V ta namen smo potencial zeta določili trem polimernim materialom: poli(etilentereftalatna) folija, tankoslojna poliamidna kompozitna membrana za nanofiltracijo in reverzno osmozo. Rezultati, pridobljeni s »Clamping Cell« v primerjavi z »Adjustable Gap Cell«, kažejo na razlike v potencialu zeta, kjer merilna celica »Adjustable Gap Cell« daje ponovljivejše rezultate. Eden izmed možnih razlogov za takšno obnašanje bi lahko bil v različnih geometrijah pretočnih kanalov. Veliko verjetnejši razlog leži v geometriji merilne celice »Clamping Cell«, ki zahteva večjo površino vzorca kot je potrebna za določitev potenciala zeta.