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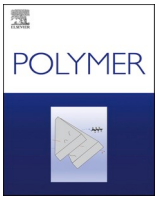

Hydrophilicity and salt tolerance of water soluble (charged and uncharged) polymers

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A B S T R A C T

The dependence of the viscosity of dilute aqueous solutions of polyelectrolytes and uncharged macromolecules on their concentration and on the salinity of the solvent provides information concerning their interactions with water and low-molecular salts. The central parameters are salt tolerance and hydrophilicity. For polyelectrolytes the salt tolerance τ (logarithm of the external salt concentration at which the intrinsic viscosity changes most) is at least one order of magnitude less than for uncharged polymers. This finding is attributed to the fact that low molecular weight ions bind water molecules in their hydration shell that would be required for the solvation of the macro-ions. The hydrophilicity parameter λ_{100} (characterizing the equilibrium of water inside isolated polymer chains and the surrounding pure water) is for polyelectrolytes composed of contributions stemming from the main chain and the attached macro-ions. In the absence of extra salt λ_{100} is at least one order of magnitude larger than for the uncharged polymers due to the high solvation tendency of the macroions. External salt invariably reduces λ_{100} due to water's preferential interaction with free ions compared to macro-ions. Limiting values are reached once micro-ions fully establish their hydration shells. For uncharged polymers, their solubility is determined solely by the backbones, and the hydrophilicity is generally much lower than for polyelectrolytes. Specific polymer/salt interactions can increase or decrease λ_{100}. This approach uses simple experiments and provides quantitative information about the interaction between inorganic salts and polymers, as well as their salting-in and salting-out properties.

1. Introduction

The study of the interactions between salts and macromolecules in aqueous solution originated with experiments concerning protein precipitation more than 100 years ago [1]. Since then, the importance of these interactions have been proved in many other areas, such as reverse osmosis, nanofiltration, forward osmosis, pressure-retarded osmosis, and membrane capacitive deionization (MCDI) [2]. Many different, mostly quite sophisticated methods were used to study specific questions, such as impedance spectroscopy, rheology and DSC [3], ion conductivity [4], phase separation [5], intrinsic viscosity plus viscoelasticity [6], polymer swelling [7], coacervate formation [8], salting-in and salting-out [9,10] and anomalous segmental dynamics [11], to name just a few. Despite this situation and the many fields in which such interactions are of utmost importance, the present knowledge in the area is still far from being satisfactory, especially when it comes to quickly and easily obtaining the desired information.

For the aforementioned reasons, this article addresses the fundamental question of whether the influence of salts on the hydrophilicity of charged or uncharged polymers can be quantified; it demonstrates how phenomenological thermodynamics and simple viscosity measurements can be used to obtain a meaningful parameter for these important properties.

2. Theoretical background

2.1. Intrinsic viscosities and salt tolerance

The following relation [12] was used to determine the intrinsic viscosities $[\eta]$ as a function of the salinity of the solvent

$$ln\,\eta_{rel} = \frac{[\eta]c + \alpha[\eta]^2c^2}{1 + \beta[\eta]c + \gamma[\eta]^2c^2} \quad (1)$$

η_{rel} is the viscosity of the polymer solution with the concentration c (mass per volume) divided by that of the solvent and α, β and γ are viscometric interaction parameters accounting for the composition dependence of the friction between the components of the mixture.

To model the influences of salt on the intrinsic viscosities, we introduce the reduced viscosities, $[\eta]_{red}$ as $[\eta]/[\eta]_o$, where $[\eta]_o$ stands for the intrinsic viscosity of the polymer in pure water. Using $\log M_{salt} = x$ as the independent variable and the Dose-Response relation (x being the dose and $[\eta]_{red}$ the response) for the modeling we can write

$$[\eta]_{red} = A_1 + \frac{A_2 - A_1}{1 + 10^{(\tau - x)\sigma}} \quad (2)$$

The abscissa of the point of inflection of $[\eta]_{red}$ vs x we refer to as τ and called it salt tolerance; this is the parameter that interests in the

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current context; σ of the above equation measures the steepness of the Dose response curve. A_1 and A_2 are parameters stating the minimum and maximum effects of salt on the intrinsic viscosity, respectively.

For practical purposes it is useful to distinguish between the two possible situations: The reduction or the augmentation of $[\eta]$ by salt. In the former case we can write

$$[\eta]_{\mathrm{red}} = [\eta]_{\mathrm{red}}^{\mathrm{min}} + \frac{1 - [\eta]_{\mathrm{red}}^{\mathrm{min}}}{1 + 10^{(\tau - x)\sigma}} \tag{3}$$

where $[\eta]_{\mathrm{red}}^{\mathrm{min}}$ is the maximum **reduction** approached in the limit of high salinity and the steepness σ is negative. In the opposite case in which $[\eta]_{\mathrm{red}}$ rises with salinity eq (2) becomes

$$[\eta]_{\mathrm{red}} = [\eta]_{\mathrm{red}}^{\mathrm{max}} + \frac{1 - [\eta]_{\mathrm{red}}^{\mathrm{max}}}{1 + 10^{(\tau - x)\sigma}} \tag{4}$$

where $[\eta]_{\mathrm{red}}^{\mathrm{max}}$ is the maximum **increase** of $[\eta]_{\mathrm{red}}$ in the limit of high salt concentrations and the steepness parameter σ is positive.

There are systems in which eq (3) and eq (4) become obsolete because the limiting behavior for high salt concentrations is experimentally inaccessible. In these cases, the data can be modeled by means of the following equation

$$[\eta]_{\mathrm{red}} = 1 + \mathrm{p}\ exp(\mathrm{q}\ x) \tag{5}$$

where the sign of the parameter p decides whether the intrinsic viscosity increases or decreases with rising salinity and q how sensitive the system is to salt.

2.2. The hydrophilicity parameters λ

A single, isolated macromolecule, dissolved in an infinite excess of a low molecular weight liquid represents the simplest possible example of a polymer solution. Its theoretical treatment is under such conditions comparatively easy because it is only the equilibrium between the solvent contained in the microphase of the isolated coil and the pure solvent surrounding it that needs to be modeled. Employing the tools of phenomenological thermodynamics [13] instead of the usual considerations in terms of chain elasticity one obtains the following relation

$$ln\,(1 - \Phi_o) + \left(1 - \frac{1}{N}\right)\Phi_o + \lambda \Phi_o^2 = 0 \tag{6}$$

in which Φ_o is the volume fraction of the polymer inside an isolated coil, N the number of segments and λ a differential interaction parameter increasing as the thermodynamic quality of the solvent rises; its value can be calculated from the intrinsic viscosity and the density of the polymer ϱ_2 by means of the following relation

$$\Phi_o = \frac{1}{[\eta]\varrho_2} \tag{7}$$

The parameter λ is given by

$$\lambda = -\frac{ln(1 - \Phi_o) + \left(1 - \frac{1}{N}\right)\Phi_o}{\Phi_o^2} \tag{8}$$

To render the results more comparable, we substitute the number of segments, N (defined in terms of monomeric units which can change significantly from polymer to polymer) with an arbitrary value of 100 mL for the volume a mol of segments occupies, so that we can write

$$N_{100} = \frac{M}{100\ \varrho_2} \tag{9}$$

where M is the molar mass of the polymer. In this manner eq (6) becomes

$$ln(1 - \Phi_o) + \left(1 - \frac{1}{N_{100}}\right)\Phi_o + \lambda_{100}\Phi_o^2 = 0 \tag{10}$$

and one obtains the following relation for λ_{100}, the normalized hydrophilicity parameter

$$\lambda_{100} = -\frac{ln(1 - \Phi_o) + \left(1 - \frac{1}{N_{100}}\right)\Phi_o}{\Phi_o^2} \tag{11}$$

Eq (11) is central for the present study on the salt influences on the hydrophilicity of polymers. It quantifies λ_{100} via their intrinsic viscosity in saline solvents and their molar mass in terms of N_{100}, where the latter contribution is of minor importance only.

The experimental results for polyelectrolytes can be modeled by means of the following relation, which is of the Dose response type again

$$\lambda_{100} = \lambda_{100}^{\mathrm{water}} + \frac{\lambda_{100}^{\mathrm{min}} - \lambda_{100}^{\mathrm{water}}}{1 + 10^{(\vartheta - x)\mu}} \tag{12}$$

$\lambda_{100}^{\mathrm{water}}$ is the value in the absence of salt and $\lambda_{100}^{\mathrm{min}}$ the hydrophilicity in the limit of large salt concentrations; ϑ and μ are parameters characteristic for a given system.

For most of the uncharged polymers it is impossible to model the results by means of eq (12) because limiting values of λ_{100} for high salt concentrations are experimentally inaccessible. In these cases the following simplified relation suffices for the description

$$\lambda_{100} = \lambda_{100}^{water} + a\ exp(b\ x) \tag{13}$$

where the sign of the parameter b determines, whether λ_{100} increases or decreases and a the magnitude of the effect.

2.3. Salt tolerance and bound water

The idea behind the following considerations rests on the observation [14,15] that water is in the hydration shell of salts strongly bound to ions and that unbound ("free") water is the most favorable solvent for water soluble polymers. In order to discuss the effects associated with this feature, we calculate the fraction of bound water f_{bw}, 1 L, the moles per liter of water that are strongly attached to the ions, and divide it by M_{mfw}, the maximum number of moles of water contained in one liter of the saline solvent

$$f_{\mathrm{bw}} = \frac{M_{\mathrm{bw}}}{M_{\mathrm{mfw}}} \tag{14}$$

For M_{mfw} we can write

$$M_{\mathrm{mfw}} = M_{\mathrm{fw,o}} - V_s M_s \tag{15}$$

where $M_{\mathrm{fw,o}}$ is the value M_{mfw} in the absence of extra salt and V_s stands for the molar volume of the salt.

To obtain M_{bw}, the number Z of moles of water bound by one mol of salt, we need to know n_{C} and n_{A}, the numbers of cations and anions in the chemical formula plus their hydration numbers z_{C} and z_{A}. Based on this information we can write

$$Z = z_C n_C + z_A n_A \tag{16}$$

In that manner we obtain the following expression for the moles of bound water

$$M_{\mathrm{bw}} = Z\, M_s \tag{17}$$

and we can write

$$f_{\mathrm{bw}} = \frac{M_{\mathrm{bw}}}{M_{\mathrm{mfw}}} = \frac{Z\, M_s}{M_{\mathrm{fw,o}} - V_s M_s} \tag{18}$$

3. Substances and procedures

The origin and the purity of the substances are described in the documents cited below. All viscosity measurements were carried out on commercially available rotational rheometers, mainly from Ubbelohde. Polyacrylic acid [16] (PAAc), sodium polystyrene sulfonate [17] (PSS Na) and sodium pectinate [18] (PEC Na). The uncharged polymers under investigation are polyethylene oxide [19] (PEO), polyvinyl alcohol [20] (PVAl) and dextran [21] (DEX).

4. Results

The experimental data on the salt dependencies of the intrinsic viscosities were evaluated according to eqs (3) and (4), respect.; eq (11) was used to calculate the hydrophilicity parameter λ_{100}.

4.1. Polyelectrolytes

Fig. 1 shows examples for the effects of increasing salt concentrations on the intrinsic viscosities of charged macromolecules. This graph demonstrates that the polymer coils shrink pronouncedly and that the chemical nature of the polyelectrolytes determines the details of this dependence, like the position of the points of inflection and the limiting value sof $[\eta]$ for high salinities of the solvent.

Fig. 2 depicts the salt dependence of the hydrophilicity parameter λ_{100} for the polyelectrolytes examined. As expected, the interaction of the PEC, which has numerous hydroxyl groups, with pure water is the most favorable. The interaction of PSS-Na is the least favorable due to its aromatic rings.

The behavior of uncharged polymers varies significantly more than that of polyelectrolytes, which is predominantly controlled by the electrical charges. Fig. 3 exemplifies this situation by comparing the three systems with the greatest dissimilarities. Only the combination of PVAl + LiCl shows the behavior typical for polyelectrolytes, i.e. a reduction of $[\eta]$ with rising salinity. In the case of DEX + KSCN, the intrinsic viscosity increases and reaches a saturation value, and for solutions of PEO + NaCl, no limiting value for $[\eta]$ is reached even at the highest salinities.

Fig. 4 demonstrates that the chemical nature of the salt plays a decisive role for changes of $[\eta]$ with the salinity of the solvent; it even determines the sign of the effect.

Fig. 5 illustrates the range within which the normalized hydrophilicity parameters λ_{100} vary in case of the uncharged polymers under investigation.

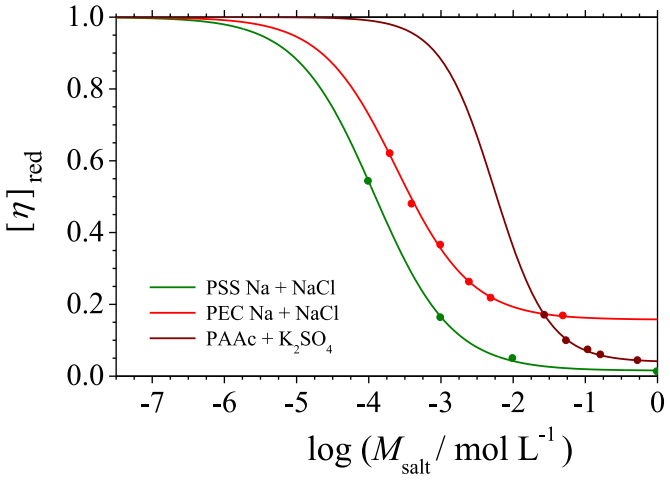

Fig. 1. Salt dependence of the reduced intrinsic viscosities, modeled according to the Dose response relation of eq (15). The polymers and salts under investigation are indicated in the graph.

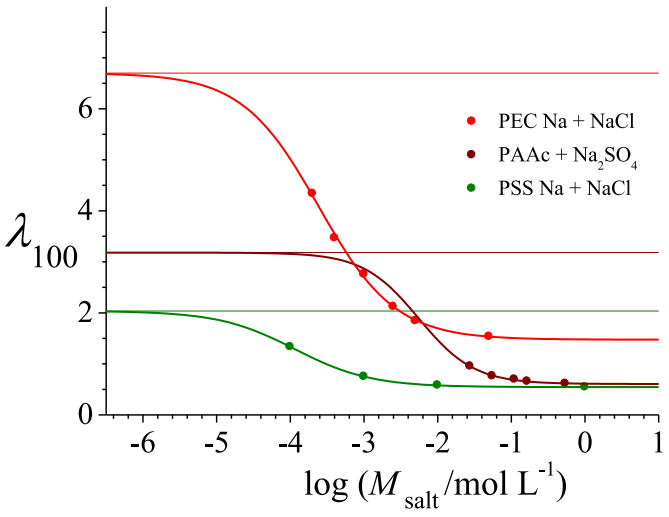

Fig. 2. Salt dependence of the hydrophilicity parameter λ_{100} for the indicated polyelectrolytes.

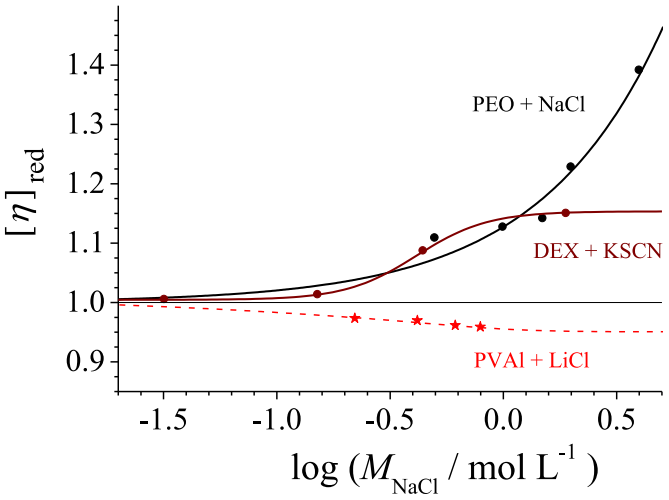

Fig. 3. Like Fig. 1 but for aqueous solutions of the uncharged polymers.

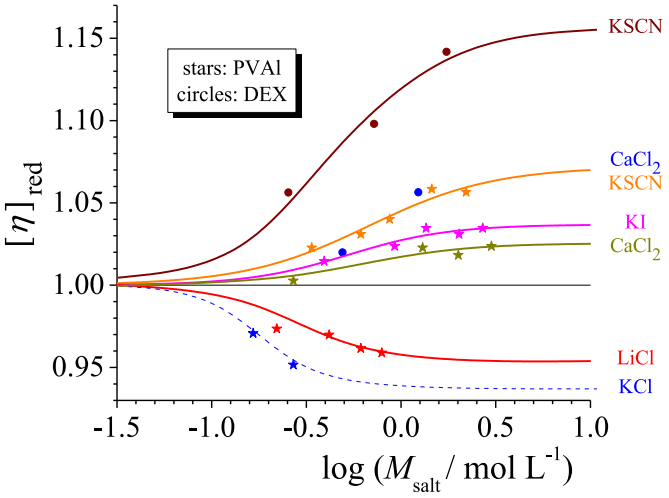

Fig. 4. Reduced intrinsic viscosities as a function of the solvent salinity for solutions of PVAl and of DEX for the indicated salts.

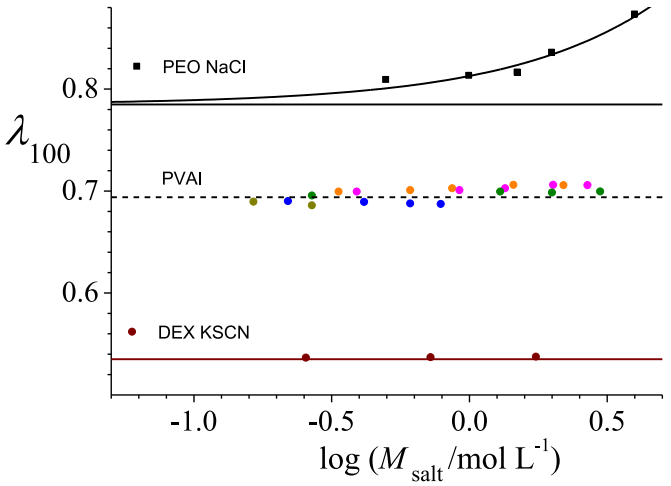

Fig. 5. Comparison of the reduced hydrophilicity parameter λ_{100} for all uncharged polymers under investigation.

PEO interacts most favorably with water and the addition of NaCl to the solvent increases its hydrophilicity. For DEX water represents the least favorable solvent and the hydrophilicity does not change within experimental error as KSCN is added. The behavior of PVAl lies in between these extremes and it is this polymer which exhibits the most diverse salt effects as detailed in Fig. 6.

5. Discussion

5.1. Verification of the concept of bound water

This section examines whether the experimental findings support the idea - described in the theoretical part - that the changes in [η] are caused by the fraction water that is bound in the hydrate shell rather than by the salt itself. To that end we calculate f_{bw} according to eq (18) using the parameters of Table 1.

Fig. 7 shows the results of this calculation for the reduced intrinsic viscosities of the polyelectrolyte solutions. The expectation that introducing f_{bw} instead of salt concentration would result in qualitative changes—for example, eliminating the intersection of the individual curves—is not met. The chemical and architectural differences between the various types of polyelectrolytes are evidently too significant.

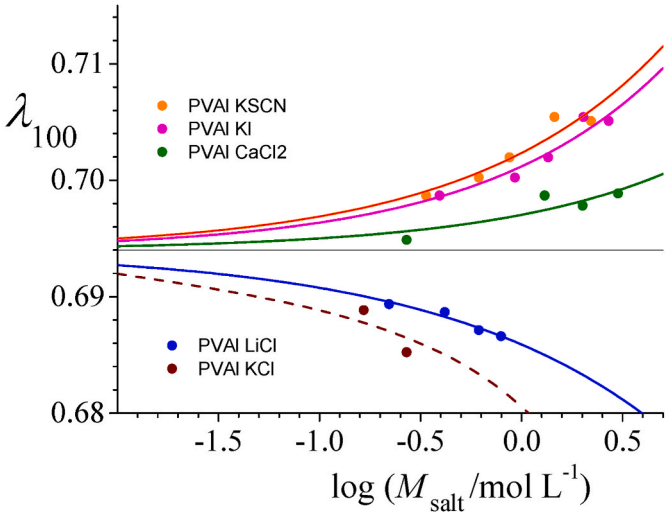

Fig. 6. Influences of the chemical nature of the salt on the hydrophilicity parameter λ_{100} in the case of PVAl.

Table 1
z_C and z_A: numbers of cation and anion in the chemical formula, n_C and n_A: coordination numbers (water molecules "bound" to them), Z: number of moles of water bound by one mol of salt.

	z_C	z_A	n_C	n_A	Z
Na Cl	1	1	6[15]	6.1[15]	12.1
K_2 SO_4	2	1	12[15]	7.5[22]	31.5

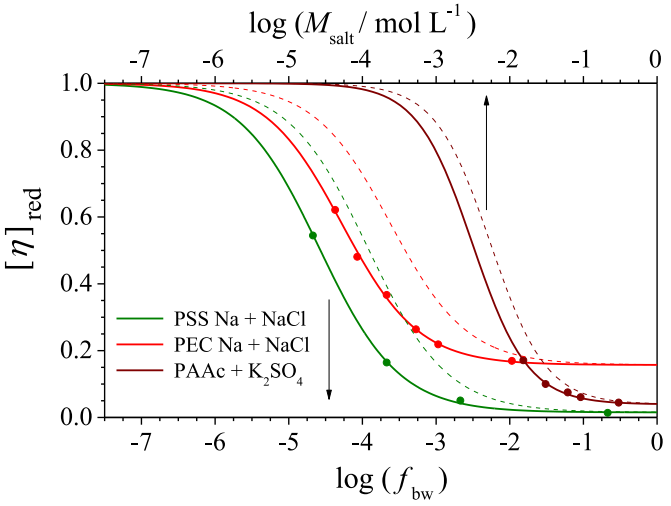

Fig. 7. Dependence of the reduced intrinsic viscosities on the fraction of bound water for the polyelectrolytes and salts specified in the graph. For comparison the broken lines indicate the corresponding dependence on the salt concentrations (upper abscissa).

Fig. 8, showing the dependence of the hydrophilicity parameter λ_{100} on the fraction of bound water for uncharged polymers, also proves the shift of the abscissa as compared to the dependence on M_{salt} (cf. Figs. 5 and 6) and demonstrates that the above consideration also hold true for this class of polymers. Again the sequence of the individual curves remains unchanged; in this case it is the chemical nature and the molecular architecture of the different salts that dominate the salt influences. The uncommonly large augmentation of λ_{100} upon an increase of f_{bw} observed for PEO is probably an outcome of the pronounced end-group association reported in the literature [23].

In summing up it can therefore be stated that the concept of the f_{bw} is

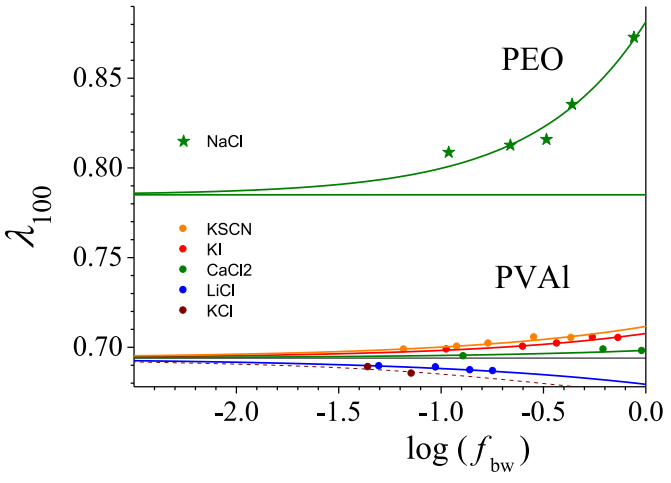

Fig. 8. Dependence of the reduced hydrophilicity parameter λ_{100} on the fraction of bound water f_{bw} for solutions of the indicated uncharged polymers and salts.

correct in principle but not very helpful in the current context.

5.2. Salt tolerance of polyelectrolytes compared to uncharged polymers

The present measurements show, that large differences exist in the response of the relative intrinsic viscosities of polyelectrolytes and of uncharged polymers with respect to an increase in the salinity of the solvent. Fig. 9 compares these data in terms of salt tolerance τ (the value of log M_{salt} at the point of inflection, see Figs. 1 and 4).

Fig. 9 shows that the intrinsic viscosities of polyelectrolytes are generally much more sensitive to extra salt than that of uncharged polymers. For the systems under investigation, the salt tolerance of charged polymers is extremely low, ranging from - 4 to – 2, in contrast to uncharged polymers, whose tolerance is higher by at least one order of magnitude. Further measurements are required to determine how the observed effects are composed of the contributions of the chemical structure, the molecular architecture of polymer chain, and of the special properties of salts.

5.3. The hydrophilicity of polyelectrolytes compared to uncharged polymers

Fig. 10 serves to illustrate these differences. The results for the polyelectrolytes are shown on the left of the graph, with the open rectangles representing the hydrophilicity parameters λ_{100} in pure water and the filled ones their limiting value for high salt concentrations. The results for PVAl as an example for uncharged polymers are displayed on the right, where the horizontal line indicates the behavior in pure water and the filled rectangles represent the hydrophilicity parameters of the polymers in the presence the different salts at the characteristic concentrations at which the fraction of bound water becomes unity (cf. Fig. 8).

The most striking differences noticeable when looking at the results shown in Fig. 10 are as follows: The hydrophilicity of the polyelectrolytes in pure water is up to ten times larger than that of uncharged polymers. In addition, extra salt reduces the λ_{100} values by a factor of about 1/3 or even more for all charged polymers, in contrast to the uncharged polymers, where the hydrophilicity either increases or decreases depending on the nature of the salt and the maximum changes are on the order of 2 % only. It is especially remarkable that the lowest λ_{100} values achievable for polyelectrolytes with extra salt are comparable to those of uncharged polymers in pure water.

For the sake of completeness, it should be noted that salts are not the only compounds that bind water so strongly that it practically breaks down into two components. As was already pointed out in 1984, polyvinyl alcohol (PVAl) exhibits such behavior [24] and a review article on the subject has recently been published [25].

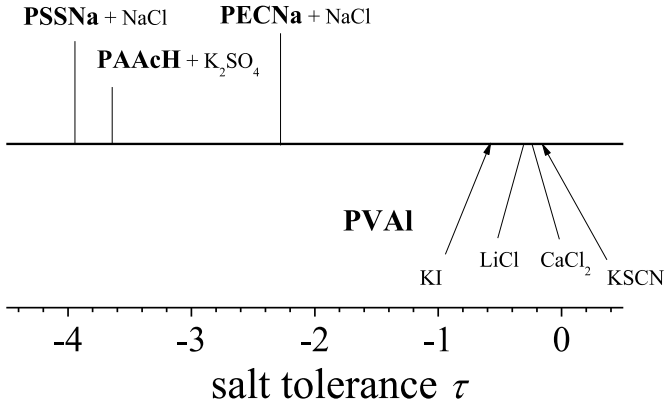

Fig. 9. Comparison of the salt tolerance τ (eqs (3) and (4)) for aqueous solutions of polyelectrolytes and of uncharged polymers.

Let us now turn to more detailed considerations on the behavior of polyelectrolytes. Their hydrophilicity is - in contrast to that of uncharged polymers - made up of two contributions, namely that of uncharged polymer backbone and that of the macro-ions attached to it. For instance in the case of PEC-Na the backbone is hydrophilic and for PSS-Na it is hydrophobic, as immediately noticeable by the fact that pectin is water-soluble and polystyrene is not. The interaction of the macro-ions with water, on the other hand, is always extremely favorable due to large reduction in the Gibbs energy resulting from the formation of the hydration shells, which makes the polyelectrolytes generally water soluble. It is the balance between these two contributions that determines their hydrophilicity in pure water.

The experimental observation that extra salt inevitably reduces λ_{100} can be traced back to the preference of water to interact with free ions as compared with macro-ions. Low molecular weight ions can interact with water in all three spatial directions, in contrast to the macro-ions, which are hindered by their connection to the polymer backbone. What also deserves explanation is the existence of a limiting value of λ_{100} as the solvent's salinity increases. This feature likewise follows from the fact that water strongly prefers contact to micro-ions over contacts to macro-ions. This means that the competition between the two types of ions comes to a standstill as soon as all micro-ions have fully developed their solvate shell.

Let us now focus on the influences of the solvent salinity on the hydrophilicity parameters of the uncharged polymers on the right-hand side of Fig. 10. What is particularly noteworthy here is that different salts can either lower their hydrophilicity (as in the case of polyelectrolytes) or increase it (not observed for polyelectrolytes) because the specific properties of the salts are no longer overshadowed by macro-ions. This enables (to the best of the author's knowledge for the first time) the possibility to determine interaction parameters for the contact formation of uncharged polymer with low molecular weight salts via the initial slopes of the dependencies shown in Figs. 6 and 8. The next section provides a more detailed analysis of the effects of salt on uncharged polymers.

5.4. Why external salt may decrease or increase the hydrophilicity of polymers

In contrast to the situation with polyelectrolytes, where extra salt inevitably reduces the intrinsic viscosity according to present knowledge, the effects can be of opposite nature in the case of uncharged polymers. Depending on its chemical structure, salt can either reduce [η] or increase it, i.e. deteriorate or improve the thermodynamic quality of the solvent. The following thermodynamic considerations are intended to provide a better understanding of this finding. For that purpose we construct the ternary phase diagram for the system polymer/water/salt of interest. To avoid complications due to the inclusion of equilibria between liquid and solid phases, we do not consider salt as a pure component, but rather the hypothetical liquid formed by the salt plus its primary solvate shell.

Fig. 11 sketches the situation for solutions, in which adding extra salt decreases the quality of the solvent. The polymer interacts favorably with water but exhibits liquid/liquid phase separation when mixed with the hydrated salt. The components of the third binary subsystem water/hydrated salt are again completely miscible. The fraction of bound water f_{bw} rises along the base line of the Gibbs phase triangle as indicated.

Fig. 12 sketches the situation for an increase of the solvent quality by the addition of salt.

The above phase diagrams provide a new perspective on the salting-out and salting-in effects described in the literature in great detail [26]. According to the above considerations the simple measurement of the salt effects on the intrinsic viscosity or hydrophilicity parameter of a given polymer should proof helpful for a quick and simple assessment of whether a particular salt will act as a salting-out or salting-in agent.

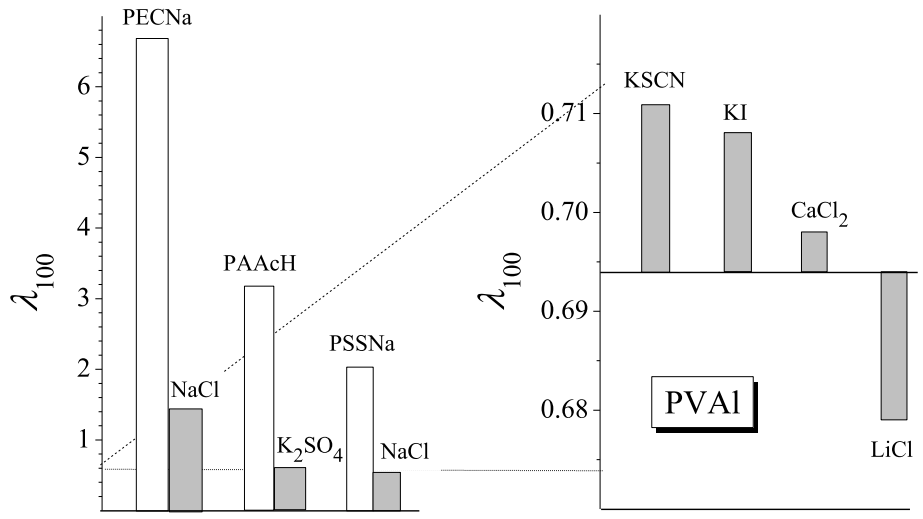

Fig. 10. ***Left***: Reduced hydrophilicity of the different polyelectrolytes in pure water (open rectangles) and their limiting values at high external salt concentrations (filled rectangles). ***Right***: Hydrophilicity of PVAl in pure water (horizontal line) and the limiting values of λ_{100} for $f_{bw} \rightarrow 1$ for the indicated salts.

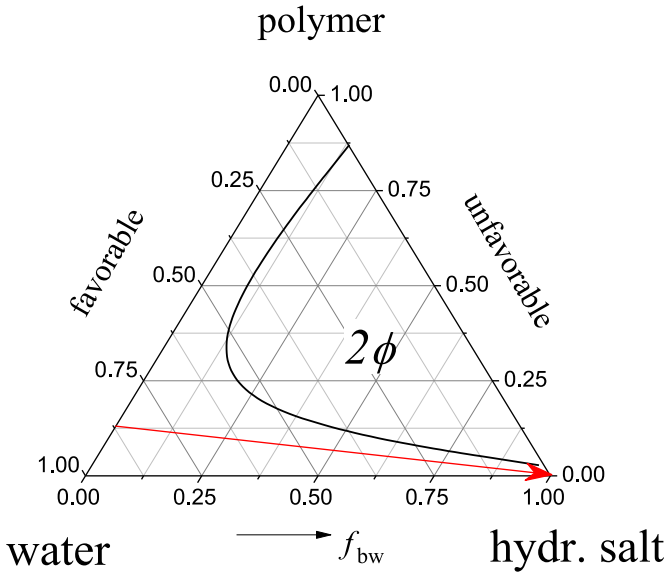

Fig. 11. Schematic representation of the Gibbs phase triangle for systems for which the polymer is only incompletely miscible with the hydrated salt. The extension of the corresponding solubility gap of the ternary system is indicated by 2Φ. The red arrow shows how the compositions change upon an increase of the solvent salinity. The fraction of bound water f bw grows when approaching the lower right corner. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

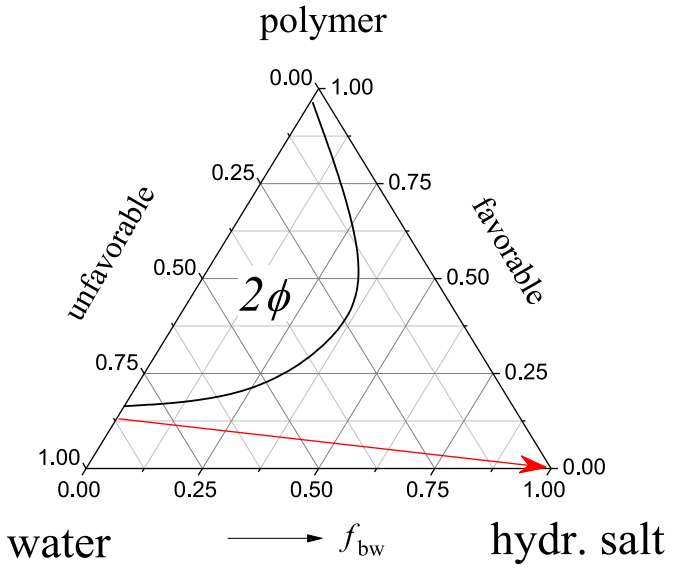

Fig. 12. As Fig. 11 but for systems where the polymer is fully miscible with the hydrated salt, but exhibits a miscible gap with water.

6. Conclusions

The conclusions regarding the salt tolerance τ of polyelectrolytes compared to uncharged polymers are as follows: The values for the charged macromolecules are in all cases at least one order of magnitude lower than for the uncharged ones, regardless of the chemical nature of low molecular weight ions involved. This is attributed to external salt having a higher tendency to incorporate water into its hydration shell than the macro-ions of the polymers do. In other words, salt reduces the availability of water required to make the polyelectrolytes soluble and causes low τ values. As the uncharged polymers are free of ions, this effect does not play a role. In this case it is the interaction of the external salt with the polymer backbone that determines the salt tolerance. According to the present findings, the magnitude of this effect is much smaller than for polyelectrolytes and the τ values are approximately one order of magnitude larger.

The following conclusions can be drawn concerning the hydrophilicity parameter λ_{100} of polyelectrolytes in pure water compared to uncharged polymers: In all cases, these values are considerably higher (up to one order of magnitude) for the former type of macromolecules than for the latter. The reason lies in the already addressed Gibbs energy driven tendency of ions to form stable hydration shells. The observation that the λ_{100} values vary considerably from polyelectrolyte to polyelectrolyte is caused by the fact that they are made up of two dissimilar contributions, the dominating one stemming from the macro-ions and the modifying one from the polymer backbone. External salt invariably reduces λ_{100} due to the preferential interaction of water with free ions as compared with macro-ions, where limiting values are reached once the micro-ions have fully established their hydration shells.

In the case of uncharged polymers the hydrophilicity for pure water is exclusively determined by the chemical nature and architecture of their backbone. External salt may either increase or decrease this value. The details of this dependence open up a possibility to quantify the

interaction between inorganic salts and uncharged polymers. This information can be used to obtain easily accessible information on the salting-out or salting-in properties of different salts for polymers of interest, as demonstrated by means of ternary phase diagrams for polymer/water/salt systems.

The results reported here demonstrate the effectiveness of the current approach. While the findings reveal certain basic correlations, a number of questions remain unanswered. The main questions concern the influence of the chemical nature of salts on the hydrophilicity and degree of charging of polyelectrolytes, as well as the influence of the molecular architecture of macromolecules.

Declaration of competing interest

The author declares that he has no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

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