Thermodynamic Properties of Dilute Aqueous Polymer Solutions from Low-Angle Laser-Light-Scattering Measurements

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ABSTRACT: Weight-average molecular weights and osmotic second virial coefficients obtained from low-angle laser-light-scattering (LALLS) measurements are reported for binary aqueous solutions of eight nonionic polymers at 25 °C. For dilute ternary aqueous solutions containing two polymers, cross osmotic second virial coefficients were also obtained from LALLS measurements. Using the truncated osmotic virial expansion, we find that the calculated water-vapor pressures for aqueous solutions of Dextran T-70 are in reasonable agreement with previously reported experimental vapor-pressure data in the dilute (<10 wt %) polymer region. At higher polymer concentrations, calculated vapor pressures are too low, indicating a need to include higher coefficients in the virial expansion.

Introduction

In recent years, water-soluble polymers have found increasing commercial applications in mineral processing, pharmaceuticals, detergents, cosmetics, and textiles production.[1] Water-soluble polymers are finding rising industrial application in the formation of aqueous two-phase systems for separation of protein mixtures.[2,3] However, few fundamental studies have been reported on the solution properties of water-soluble polymers.

The literature is rich in theoretical studies for describing polymer solutions.[4-20] Unfortunately, most polymer solution theories are not useful for describing aqueous systems. A possible theoretical description for concentrated aqueous polymer solutions was recently proposed by Prange et al.,[21] who extended the Guggenheim lattice theory by distinguishing between ordinary and hydrogen-bonding segment–segment interactions. In this work, however, we are concerned with dilute aqueous multisolute solutions which are well described by the osmotic virial expansion.[22,23]

Our current understanding of aqueous polymer solutions is severely limited, in part, because of a lack of fundamental experimental data. This work reports new experimental measurements at 25 °C for binary and ternary aqueous solutions of eight nonionic water-soluble polymers: dextran, poly(vinylpyrrolidone), methoxypoly(ethylene glycol), poly(vinyl alcohol), methylcellulose, Ficoll, Aquaphase PPT, and Bermocoll E.

The results reported here supplement earlier experimental work by our group[24-26] and by others.[27-33] For dilute binary aqueous solutions, we have measured polymer weight-average molecular weights and osmotic second virial coefficients using low-angle laser light scattering (LALLS); for dilute ternary aqueous polymer solutions, we have used LALLS to measure cross osmotic second virial coefficients.

Experimental Section

Materials. Poly(vinyl alcohol) (PVA), methylcellulose (MC), and poly(vinylpyrrolidone) (PVP) fractions were purchased from Aldrich Chemical Co. (Milwaukee, WI). Methoxypoly(ethylene glycol) (MOPEG) fractions were purchased from Sigma Chemical Co. (St. Louis, MO). Dextran fractions and the Ficoll fraction (a synthetic polymer made from the copolymerization of sucrose and epichlorohydrin) were purchased from Pharmacia Fine Chemicals (Piscataway, NJ). Aquaphase PPT, a hydroxypropyl starch, and Bermocoll E, an ethyl hydroxyethyl cellulose ether, were kindly provided by Perstrop Biolytica AB (Lund, Sweden) and Berol Kemi (Stenungsund, Sweden), respectively. Water used to prepare the polymer solutions was filtered through a Barnstead Nanopure water purification system.

Analytical Methods. In binary aqueous solutions, concentrations of dextran, Aquaphase PPT, or Ficoll were measured with a Perkin-Elmer 241 polarimeter. The specific optical rotation constants used for dextran, Aquaphase PPT, and Ficoll were +199.5, +190.0, and +54.9°, respectively. The specific optical rotation constants are independent of molecular weight for polymers with molecular weight >10 000. The concentration of any nonoptically active polymer in binary aqueous solutions was determined by using size-exclusion, high-performance liquid chromatography (SE-HPLC). The chromatographic system consisted of an LDC-Milton Roy metering pump, one Bio-Gel TSK-40 column, two Bio-Gel TSK-30 30-cm columns, and a Knauer differential refractometer detector connected in series.

Low-Angle Laser Light Scattering. Static light scattering measurements at 25 °C were made with an LDC-Milton Roy KMX-6 low-angle laser-light-scattering (LALLS) photometer which employs a 2-mW helium–neon laser at a fixed wavelength of 633 nm. Five or more 25-mL dilute aqueous polymer solutions were prepared for each polymer fraction, covering a portion of the allowable solute concentration range (0.5–50 mg/mL) for light scattering. In general, for a given polymer concentration, a higher molecular weight polymer gives greater light scattering. Thus, polymer concentrations were near the low end of the allowable range for solutions containing a high-molecular-weight polymer; polymer concentrations were near the high end for solutions containing a low-molecular-weight polymer. Prior to analysis, each solution was clarified by passing it through a Millipore 0.22-μm filter. The clarified sample solution was then pumped through the light-scattering cell at a rate of 0.35 mL/min with a Sage Instruments syringe pump. The laser beam was focused to a diameter of 80 μm and passed through 0.1 μL of the 150-μL sample cell. Scattered light was detected by the photomultiplier at a free-space scattering angle between 6 and 7°.

The Rayleigh factor, R_θ (cm^{-1}), describes the light-scattering property of any molecule whose size is small compared to the wavelength of the incident light. It is related to J_θ, the radiant intensity of scattered light in the direction θ, by[34]

$$R_\theta = J_\theta / I_0 V \qquad (1)$$

where I_0 is the irradiance of the illuminating beam and V is the volume of the scattering medium. The LALLS photometer measures the Rayleigh factor of each sample solution, $R_{\theta,\mathrm{sample}}$ (cm^{-1}), as it passes through the light-scattering cell.

LALLS experiments were performed for binary aqueous polymer solution samples in a two-step sequence. First, the LALLS photometer was used to measure the $R_{\theta,\mathrm{sample}}$ of each of the five or more sample solutions. Next, the Rayleigh factor for pure water (solvent) was measured with the LALLS photometer and subtracted from $R_{\theta,\mathrm{sample}}$ to give the contribution of the polymer

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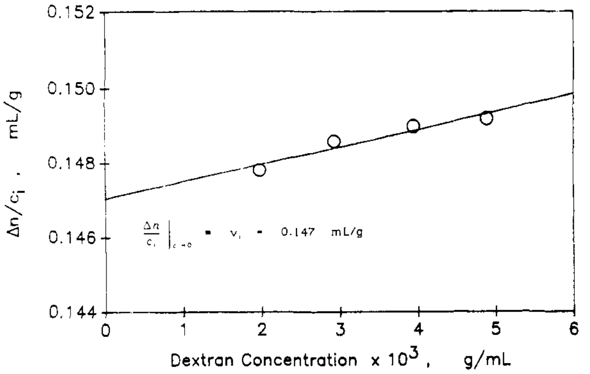

Figure 1. Determination of the specific refractive index increment for Dextran T-70 in water at 25 °C.

to $R_{\theta,\mathrm{sample}}$. This difference, known as the reduced Rayleigh factor, $\bar{R}_\theta$, is defined as

$$\bar{R}_\theta = R_{\theta,\mathrm{sample}} - R_{\theta,\mathrm{solvent}} \qquad (2)$$

Reduced Rayleigh factors were also measured for ternary aqueous polymer systems; for each total polymer concentration in a given system, the ratio of individual polymer concentrations was held constant.

To use $\bar{R}_\theta$ data for calculating the osmotic second virial coefficient and the weight-average molecular weight of a polymer fraction i, it is necessary to know the specific refractive index increment, ν_i (mL/g), for the fraction. Values of ν_i were determined by first measuring the difference in refractive indices, Δn, of each polymer sample solution with respect to the solvent (water). Then a plot was made of $\Delta n/c_i$ as a function of c_i, where c_i (g/mL) is the concentration of polymer fraction i; ν_i, defined by

$$\nu_i = \lim_{c_i \to 0} (\Delta n/c_i) \qquad (3)$$

was found by extrapolating to $c_i = 0$, as shown in Figure 1 for Dextran T-70.

Results and Discussion

Data Reduction. From LALLS measurements for binary aqueous solutions of each polymer fraction i, $Kc_i/\bar{R}_\theta$ values were determined over a range of polymer concentrations. The weight-average molecular weight and osmotic second virial coefficient of the polymer fraction were calculated by using the data for $Kc_i/\bar{R}_\theta$ as a function of polymer concentration.

For ternary aqueous solutions containing two different water-soluble polymers, i and j, $K'(c_i + c_j)/\bar{R}_\theta$ was measured as a function of total polymer concentration with the ratio of polymer concentrations held constant. These measurements were used to calculate cross osmotic second virial coefficients, as briefly discussed below.

Chemical Potentials and Osmotic Virial Coefficients. For dilute binary aqueous polymer solutions, the chemical potential, μ_0, of the solvent (0) may be expressed as a series expansion in the concentration of solute (i)

$$\mu_0 - \mu_0^\circ = -RTV_0^\circ c_i\left(\frac{1}{M_{\mathrm{w}_i}} + A_{ii}c_i + A_{iii}c_i^2 + \ldots\right) \qquad (4)$$

where μ_0 is the chemical potential of the pure solvent, V_0° is the molar volume of the solvent (mL/mol), c_i is the concentration of the polymer (g/mL of solution), M_{w_i} is the weight-average molecular weight of the polymer fraction, A_{ii} is the osmotic second virial coefficient (mL·mol/g^2), and A_{iii} is the osmotic third virial coefficient (mL^2·mol/g^3), all at system temperature and pressure. At normal pressures, virial coefficients are functions only of temperature and the nature of the solvent and solute. Statistical-mechanical considerations suggest that A_{ii} is a measure of two-body interactions, while A_{iii} accounts for three-body interactions in the solvents.[35]

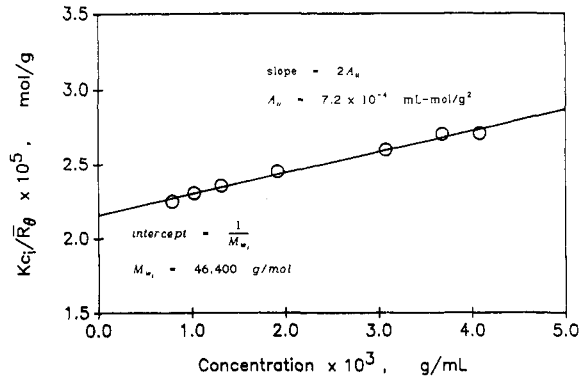

Figure 2. Low-angle laser-light-scattering data for poly(vinylpyrrolidone)-40 in water at 25 °C.

For dilute solutions of particles small compared to the wavelength of the incident light, it is the rate of change of solvent chemical potential with polymer concentration $(\partial\mu_0/\partial c_i)_{T,P}$, which is related through fluctuation theory to the reduced Rayleigh factor, $\bar{R}_\theta$ (eq 2). Differentiation of eq 4, truncated after the second virial coefficient term, yields the working equation for determination from LALLS data of the weight-average molecular weight of the polymer fraction and the osmotic second virial coefficient

$$\frac{Kc_i}{\bar{R}_\theta} \equiv -\frac{1}{RTV_0^\circ c_i}\left(\frac{\partial\mu_0}{\partial c_i}\right)_{T,P} = \frac{1}{M_{\mathrm{w}_i}} + 2A_{ii}c_i \qquad (5)$$

where K (cm^2·mol/g^2), an optical constant characteristic of the polymer and the solvent, is given by

$$K = \frac{2n_0^2\pi^2\nu_i^2(1 + \cos^2\theta)}{\lambda^4 N} \qquad (6)$$

where λ is the wavelength of the incident light (cm), n_0 is the refractive index of the solvent, and N is Avogadro's number.

The osmotic second virial coefficient and weight-average molecular weight for each polymer fraction i were determined from a plot of $Kc_i/\bar{R}_\theta$ as a function of polymer concentration, c_i, as shown in Figure 2 for poly(vinylpyrrolidone)-40.

Table I reports the experimental osmotic second virial coefficient, weight-average molecular weight, and specific refractive index increment for each polymer fraction studied here. Table I also shows the molecular weight of each polymer fraction as reported by the manufacturer. Except for dextran and Ficoll fractions, the polymer fraction molecular weights reported by the manufacturers are not weight-average molecular weights. Thus, they should not be used to check the accuracy of our experimental weight-average molecular weights; rather, they are shown to identify the polymer fractions studied here.

Table II shows experimental cross osmotic second virial coefficients for several ternary aqueous polymer systems. With the individual osmotic second virial coefficients, weight-average molecular weights, and specific refractive index increments for the two polymer fractions i and j known, the cross osmotic second virial coefficient, A_{ij}, was determined from the limiting slope of a plot of $K'(c_i + c_j)/\bar{R}_\theta$ as a function of total polymer concentration $(c_i + c_j)$ according to the linear relationship[36]

$$K'(c_i + c_j)/\bar{R}_\theta = m(c_i + c_j) + b \qquad (7)$$

with

Table I
Osmotic Second Virial Coefficients and Weight-Average Molecular Weights for Several Polymers in Water at 25 °C

polymer	manufacturer's reported mol wt	measd M_w	$A_{ii} \times 10^4$, mL·mol/g^2	ν_i, cm^3/g
poly(vinylpyrrolidone)				
PVP-10	10 000	10 100	10.6	0.181
PVP-24	24 000	35 100	8.7	0.181
PVP-40	40 000	46 400	7.2	0.185
PVP-360	360 000	1 540 000	1.7	0.179
poly(vinyl alcohol)				
PVA-14	14 000	39 900	7.9	0.151
PVA-78	78 000	120 000	6.1	0.153
PVA-108	108 000	160 000	3.6	0.153
methylcellulose				
MC-41	41 000	160 000	10.0	0.134
MC-63	63 000	210 000	8.9	0.135
MC-86	86 000	234 000	8.4	0.132
methoxypoly(ethylene glycol)				
MOPEG-0.5	500	700	45.0	0.128
MOPEG-2000	2 000	3 500	35.6	0.129
MOPEG-5000	5 000	8 160	30.0	0.127
Dextran				
T-10	10 100	10 100	8.2	0.152
T-40	35 600	43 400	5.0	0.151
T-70	67 900	68 100	4.0	0.147
T-500	486 000	508 900	1.3	0.147
Aquaphase PPT	35 000	90 000	1.7	0.148
Bermocoll E	60 000	295 000	8.0	0.180
Ficoll	400 000	440 000	1.0	0.148

Table II
Osmotic Second Virial Cross Coefficients for Several Polymer Pairs in Water at 25 °C

polymer pair	$A_{ij} \times 10^4$, mL·mol/g^2
MOPEG-5/Dextran T-70	9.5
MOPEG-5/Dextran T-500	9.6
PVP-10/Dextran T-70	9.1
PVP-40/Dextran T-70	5.6
PVP-360/Dextran T-70	3.0
MOPEG-5/PVP-10	15.3
MOPEG-5/PVP-40	10.9
MOPEG-5/PVP-360	8.0
MOPEG-5/Dextran T-10	12.1
Dextran T-70/AQPPT	2.8
PVA-14/Dextran T-70	6.1
Dextran T-70/MC-41	7.5

$$m \text{ (limiting slope)} \equiv 2(\nu_i^2 M_{w_i}^2 w_i^2 A_{ii} + 2\nu_i\nu_j M_{w_i} M_{w_j} w_i w_j A_{ij} + \nu_j^2 M_{w_j}^2 w_j^2 A_{jj}) / (\nu_i^2 M_{w_i} w_i + \nu_j^2 M_{w_j} w_j)$$

and

$$b \text{ (intercept)} \equiv 1/(\nu_i^2 M_{w_i} w_i + \nu_j^2 M_{w_j} w_j)$$

In eq 7, K' (mol/cm^4) is K/ν_i^2 and w_i is the weight fraction of polymer fraction i, defined as $c_i/(c_i + c_j)$. Equation 7 is valid only when the ratio of polymer concentrations, c_i/c_j, is held constant for all measured values of $\bar{R}_\theta$. Figure 3 shows a plot of $K'(c_i + c_j)/\bar{R}_\theta$ as a function of $(c_i + c_j)$ for the ternary aqueous system containing Dextran T-70 and PVP 40.

Accuracy of LALLS Data. The accuracy of our LALLS data is indicated by comparison with light-scattering data reported by Edsman et al.[27] For Dextran T-40 and Dextran T-70, Table III compares weight-average molecular weights and osmotic second virial coefficients reported here with those reported by Edsman et al. for the same dextran fractions from the same manufacturer. Weight-average molecular weights agree to within 13% and osmotic second virial coefficients to better than 1%. Errors in the determination of weight-average molecular weights are due to the difficulty of accurately extrapolating $Kc_i/\bar{R}_\theta$ data to zero polymer concentration.

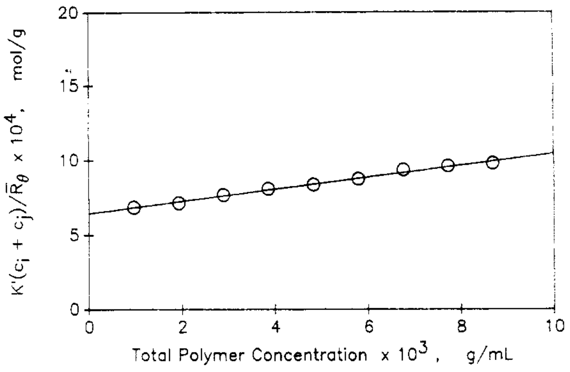

Figure 3. Determination of the osmotic second virial cross coefficient for the ternary aqueous system containing Dextran T-70 and poly(vinylpyrrolidone)-40 at 25 °C.

Table III
Comparison of Light-Scattering Data Reported in This Work for Binary Aqueous Solutions of Dextran T-40 or Dextran T-70 with Data Reported by Edsman et al.[27]

	M_w			$A_{ii} \times 10^4$, mL·mol/g^2		
polymer fraction	this work	Edsman et al.	% error	this work	Edsman et al.	% error
Dextran T-40	43 400	38 300	11.8	5.00	5.04	0.8
Dextran T-70	68 100	76 900	12.9	3.99	4.01	0.5

Comparison of Calculated and Measured Solvent Vapor Pressures. The weight-average molecular weights and osmotic second virial coefficients shown in Table I may be used in the osmotic virial expansion to calculate solvent vapor pressures of binary aqueous polymer solutions. The osmotic second virial coefficient, defined as

$$A_{ii} \equiv \lim_{c_i \to 0} \left[-\frac{\ln (a_0)}{c_i^2 V_0^\circ} - \frac{1}{c_i M_{w_i}} \right] \tag{8}$$

is strictly valid only for infinitely dilute solutions; in eq 8, the solvent activity, a_0, is defined by

$$a_0 \equiv (P_0^\circ - \Delta P)/P_0^\circ \tag{9}$$

Here, P_0° (mmHg) is the vapor pressure of the pure solvent at 25 °C and ΔP (mmHg) is the vapor pressure of the pure solvent minus that of the polymer solution. Contributions to the solvent activity from three-body and higher order interactions increase as the solute concentration rises beyond infinite dilution. However, eq 4 truncated after the second virial coefficient term has been used extensively to calculate solution properties of semidilute aqueous polymer solutions. By comparing calculated vapor pressure with those directly measured, it is possible to determine the polymer concentration range for which the truncated osmotic virial expansion can accurately predict the solution properties of binary aqueous polymer solution.

Trucating eq 4 after the second virial coefficient term and solving for ΔP, we obtain

$$\Delta P = P_1^\circ \left[1 - \exp\left\{ -V_1^\circ c_2 \left(\frac{1}{M_{w_2}} + A_{22} c_2 \right) \right\} \right] \tag{10}$$

Figure 4 compares vapor-pressure differences for binary aqueous solutions of Dextran T-70 (containing up to 20 wt % Dextran T-70) calculated with eq 10 and M_{w_i} and A_{ii} data listed in Table II with experimental ΔP data reported by Haynes et al.[26] Figure 4 indicates that eq 10 is useful for predicting solvent vapor pressures only in the dilute (<10 wt %) polymer region; at higher concentrations, where three-body and higher order interactions become increasingly important, calculated vapor-pressure differences are too low. For example, for an aqueous

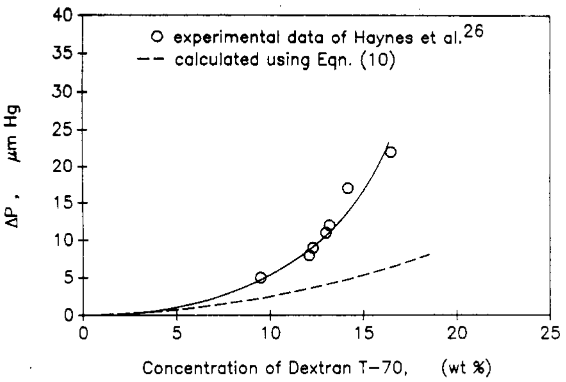

Figure 4. Comparison of experimental and calculated vapor pressures of water for aqueous solutions of Dextran T-70 at 25 °C. Calculations are based on the osmotic virial expansion truncated after the second virial coefficient term.

solution of Dextran T-70 at a polymer concentration of 16.5 wt %, the predicted ΔP is 0.0064 mmHg, compared to the experimental value of 0.022 mmHg, a difference of 71%.

To describe solution behavior at higher polymer concentrations, the osmotic virial expansion must include an "effective" third virial coefficient. Haynes et al.[26] used experimental ΔP data, together with osmotic second virial coefficients measured by LALLS, to fit an "effective" osmotic third virial coefficient for aqueous polymer solutions.

Conclusions

Low-angle laser-light-scattering experiments provide a relatively simple, rapid, and accurate method for determining the weight-average molecular weight and osmotic second virial coefficient of a nonionic water-soluble polymer dilute in water. This work reports osmotic second virial coefficients and weight-average molecular weights obtained from LALLS measurements for eight nonionic water-soluble polymers in dilute binary aqueous solutions at 25 °C. Cross osmotic second virial coefficients are reported for ternary aqueous solutions containing two nonionic water-soluble polymers.

Using the osmotic virial expansion truncated after the second term, we found that calculated solvent vapor pressure differences between pure water and dilute aqueous solutions containing Dextran T-70 were consistently lower than the observed values for polymer concentrations between 10 and 20 wt %; however, they were reasonably accurate for polymer concentrations <10 wt %. At higher concentrations, calculated results diverged quickly from experiment, indicating increasing contributions from three-body and higher order interactions.

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Registry No. Ficoll, 25702-74-3; Aquaphase PPT, 9049-76-7; dextran, 9004-54-0; poly(vinylpyrrolidone), 9003-39-8; methoxypoly(ethylene glycol), 9004-74-4; poly(vinyl alcohol), 9002-89-5; methylcellulose, 9004-67-5; ethyl hydroxyethyl cellulose, 9004-58-4.