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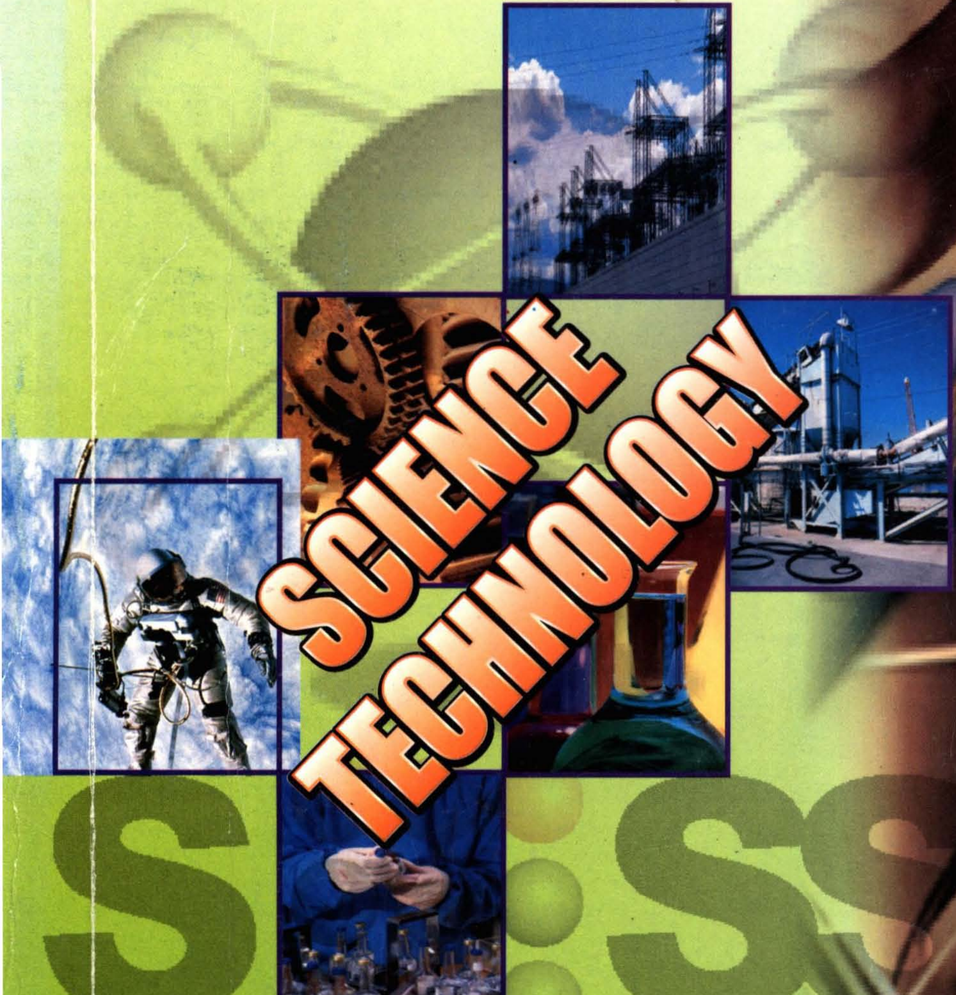

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# Osmotic Pressure Studied of Poly(dimethyl siloxane)

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## ABSTRACT

*The osmotic pressure, p of poly(dimethyl siloxane), PDMS solutions in trans-decalin was measured over the temperature range of 20 to 40 °C at concentrations from 0.5 to 15 wt.%. It was found that the mixing enthalpy $\Delta H$ and mixing entropy $\Delta S$ of trans-decalin, determined from the temperature dependence of the osmotic pressure, were negative and that the magnitude of dH was slightly larger than that of $T\Delta S$. This indicated that the dissolution of the polymers in trans-decalin was dominated by enthalpy or intermolecular interaction. The existence of local structure in the solution was revealed by negative entropy. Virial coefficients were determined from the concentration dependence of the osmotic pressure. The second virial coefficient of PDMS-trans-decalin solution increased linearly with an increased in temperature, and suggested the existence of one q temperature. The third virial coefficient of PDMS-trans-decalin solution increased at lower temperatures.*

**Keywords**: *Osmotic pressure, p; Poly(dimethyl siloxanes); Second virial coefficient, $B_2$; Interaction parameter,c.*

## Introduction

Osmosis occurs everywhere in nature. Since the earliest report on osmotic pressures in 1780, (van't Hoff, 1887; Moore, 1972) many examples of the osmotic-pressure phenomenon have been found in chemical and biological systems. Semi-permeable membranes, which cause osmotic phenomena, are fundamental building materials of all biological systems. Applications of osmosis cover many areas of research and our life, including medical diagnostics (osmolal gap or osmolality) (Medical Encyclopedia, 2004), molecular-weight determination of large molecules such as proteins and polymers, fundamental understanding of protein ultra filtration (Vilker *et al.*, 1981), interactions of proteins in aqueous solutions (Wu & Prausnitz, 1999), purification of drinking water, desalination of seawater (Hecht, 1967, Paccenti *et al.*, 1999) by reverse osmosis, civil engineering (prevention of water intrusion in concrete structures), environmental applications such as soil clean-up by electro-osmosis as well as the understanding of air intrusion in high pressure air-conditioning units.

Consider an osmotic system consisting of pure solvent, together with a solution and an intervening membrane, which is only permeable to the solvent. Let $P_1$ and $P_2$ be the pressures of the pure solvent and of the solution, respectively. At osmotic and thermal equilibrium; the osmotic pressure is defined as

$$p = P_2 - P_1 \quad (1)$$

The equilibrium condition is the equality of the chemical potential ($\mu$) in the two regions. The chemical potentials of the solvent in the solution and in the pure solvent side can be written using an activity, *a* model

$$m_1^2 = m_1^{o}(T, P_2) + RT \ln (a_1) \quad (2)$$

$$m_1 = m_1^{o}(T, P_1) \quad (3)$$

Where $m_1^{o}(T, P_2)$ and $m_1^{o}(T, P_1)$ are the chemical potentials for the pure solvent at the respective pressures and temperatures, and $a_1$ is the activity of the solvent in the solution. At the equilibrium condition, $m_1 = m_1^2$. Eq. (2) and (3) become

$$RT (\ln (a_1)) = m_1^{o}(T, P_1) - m_1^{o}(T, P_2) = \int^{P_1} V_1 \, dP \quad (4)$$

Where the second equals sign results from the integration of one of Maxwell's thermodynamic relations (Denbigh, 1971), and $V_1$ is the molar volume of the pure solvent. When we assume that the liquid is incompressible (i.e., $V_1$ is constant with respect to pressure) and the solution is an ideal solution (i.e., $a_1 = X_1$; the mole fraction of the solvent), Eq. (4) is reduced to:

$$RT (\ln(X_1)) = V_1 (P_1 - P_2) \quad (5)$$

or

$$p = -(RT/V_1)\ln(X_1) \quad (6)$$

This is well-known relation for the osmotic pressure of a solution (Allan,1959). However, this formula does not possess the power to predict the osmotic pressure for a solution whose initial composition is specified, say an initial solute molar concentration $n_2/V_0$ (or mole fraction of solute, $X_2$), since $X_1$ (or $X_2 = 1 - X_1$) in Eq. (6) is the equilibrium mole fraction after osmotic equilibrium is established. In the case of $X_2$ [illegible] 1 (or $X_1 \approx 1$), the logarithm of Eq. (6) can be expanded as:

$$p » (X_2/V_1)\,RT \quad (7)$$

Since the total number of moles $n_T$ is nearly equal to the number of mole of pure solvent in this case, $n_T V_1$ » $V_o$ (the physical volume of the prepared solution (in liter)). Then, Eq. (7) may be written as:

$$p = (n_2/V_o)\,RT \quad (8)$$

This equation can predict the osmotic pressure for a prepared solution having a molar concentration of $n_2/V_o$. In many cases, it is only valid for very dilute solutions. The first theoretical work on osmosis was done by van't Hoff in 1887, and it is known as van't Hoff's formula (van't Hoff, 1887; Moore, 1972; Denbigh, 1971). ($n_2/V_o$) is the molar concentration of a (non-permeable) solute in a solution. Formally, this equation is identical to the familiar ideal gas equation-of-state (EOS). This simple equation is still useful for extremely dilute solutions and in fact is used for the molecular-weight determination of large molecules (Denbigh, 1971). $n_2 = (g_2/M_2)$ number of moles of non-permeable solute in solution, $g_2$ is the weight of the non-permeable solute and $M_2$ is the molecular weight of the non-permeable solute. ($g_2/V_O$) = c the solute concentration. Then Eq (8) can be written as;

$$p = \{g_2/(M_2V_o)\}\,RT = RTc/M_2 \quad (9)$$

According to the more general theory of statistical thermodynamics, the osmotic pressure of general dilute-solutions can be expanded in a power series of solute concentration ($c \equiv n/V$), which is formally identical to the virial expansion for a real fluid

$$\{p/(RT)\} = c + Bc^2 + Cc^3 + Dc^4 + \cdots \quad (10)$$

Where R is gas's constant, $c = (N_A n_2)/V_O$ and $N_A$ is Avogadro's number. The first term corresponds to Eq. (8) and (9). B, C, D is the osmotic viral coefficients. Recent theoretical work on the osmotic pressure, particularly for colloid, or protein solutions, or macromolecule solutions has been largely devoted to calculation of the second virial-coefficient ($B$) based on fundamental molecular interactions, when the higher-order coefficients (the third, $C$; the fourth $D$, ...) can be neglected (Allan, 1959; Flory, 1953; Morqwetz, 1975; Rudin, 1982).

It is well established experimentally that the osmotic pressure of a solution containing a macromolecular solute is higher when the temperature differs higher from a critical solution temperature. Most osmotic pressure measurements have been carried out in dilute solutions with the aims to determine the relative molecular weight and the 'second osmotic virial coefficient. A number of theories for the thermodynamic behaviour of dilute macromolecular solutions have been developed. Little attention has been paid to concentrated solutions. However, lately the interest in concentrated solutions has increased, both experimentally and theoretically (Allan, 1959; Flory, 1953; Morqwetz, 1975; Rudin, 1982).

The concentration, c dependence of the reduced osmotic pressure, p is also expressed by

$$(p/c_2) = [(RT/M_2) + \{(RTV_1{}^O)/(2M_2{}^2)\}c + \{(RT(V_1{}^O)^2)/(2M_2{}^3)\}c^2 + \{RT(V_1{}^O)^3/(2M_2{}^4)]c^3 + \;] \ldots \quad (11)$$

Compared Eq. (9), (10), and Eq. (11). Where $B = (V_1{}^O)/(2M^2)$, $C = (V_1{}^O)^2/(2M^3)$, and $D = (V_1{}^O)^3/(2M^4)$. The osmotic pressures, p of the polymer solution are then expressed as;

$$\{(p/RT)\} = (1/M_n)c + Bc^2 + Cc^3 + Dc^4 + \ldots\ldots \ldots\ldots \quad (12)$$

Where the solute concentration, c is expressed in grams per millilitre; $V_1{}^o$ is the molar volume of pure solvent; $M$ or $M_n$ is molecular weight or number average molecular weight; B, C, and D are the second, third and fourth virial coefficients; R is the gas constant; and T is the absolute temperature.

The present investigation was undertaken to provide thermodynamic data for a well defined polymer-solvent system over an extended concentration region and in broad temperature interval. These data are also valuable in the detailed discussion of sedimentation and diffusion processes. Osmotic pressure measurements were performed on the system poly(dimethyl siloxane)/trans-decalin at concentrations in the interval 0 to 140 g/cm$^3$ and at temperatures ranging from 20 to 40 °C.

The purpose of the present work is to explore the general behavior of osmotic pressures for the entire range of solute concentrations. To do so, we employ a simple EOS for solutions, and develop thermodynamically rigorous equations for osmotic pressures. The present model calculations show complex behaviors including critical phenomena for osmosis. The present model has been successfully applied to several sets of experimental data for osmotic pressures in the literature, which include trans-decalin solutions of poly(dimethyl siloxane).

## Experiment

### Materials

Poly(dimethyl siloxanes) of molecular weight 3.90 x $10^5$ g/mol and polydispersity, $M_W/M_n = 1.10$ from Dow Corning was used in all measurements. Preparation and purification of solvent, trans-decalin has been described in else where (Maimunah, 1980, 2003, 2004). All solutions were prepared by weighing. After addition of solvent the polymer was allowed to swell before the solution was homogenised by stirring. The solute concentration, c, (mass/volume) was calculated from the weight fraction of the PDMS, $W_2$, and the density of the PDMS solutions, $\rho_2$ ($c = W_2\rho_2$) (Maimunah, 1980, 2003, 2004).

### Membrane

The acetylated cellophane membrane described previously (Maimunah, 1980) was found suitable. Solvent permeability was only marginally improved when cellophane was both acetylated. For the preparation of the membrane, cellophane dialysis tubing from Union Carbide Company was used.

### Osmotic pressure

The osmotic pressures measurements were carried out by symmetric block-type osmometer. The osmotic pressure was measured against pure solvent, the reading were always carried out at osmotic equilibrium. The osmometer cell was placed in air thermostat regulated within ± 0.05 °C. Measurements were performed with the same osmometer filling at 20, 25, 30, 35, and 40 °C. Measured osmotic pressures, p are given in Table 1.

Table1. Experimental results from osmotic pressure, p measurements on poly(dimethyl siloxane)

| $W_2$ x $10^2$ | p/Pa | | | | |
|---|---|---|---|---|---|
| | 20 °C | 25 °C | 30 °C | 35 °C | 40 °C |
| 0.5762 | 30.41 | - | - | 36.86 | - |
| 1.057 | 55.42 | 62.63 | 69.85 | 75.66 | 79.92 |
| 1.137 | - | - | - | 85.86 | 93.60 |
| 2.246 | 113.5 | 147.2 | 177.3 | 302.7 | 136.7 |
| 3.368 | 164.1 | 238.0 | 310.7 | 381.9 | 439.1 |
| 4.841 | 231.2 | 381.6 | 536.1 | 679.3 | 790.7 |
| 6.864 | 342.6 | 658.1 | 969.3 | 1265.0 | 1468.0 |
| 8.309 | 422.5 | 921.5 | 1401.0 | 1789.0 | 2189.0 |
| 10.75 | 687.6 | 1492.0 | 2347.0 | 3012.0 | 3727.0 |
| 14.82 | 1453.0 | 3067.0 | 4640.0 | 6102.0 | 7617.0 |

## Result and Discussion

The number average molecular weight, $M_n$ of the PDMS sample used is determined from the limiting value at c = 0 in a plot of (p/RTc) versus c (Fig.1), and is found to be 3.96 x $10^5$ g/mol. This value agrees well with 4.00 x $10^5$ g/

mol obtained from measurements on the same batch in cyclohexane (Maimunah, 2003, 2004). From Eq. (12), the initial slope of the curves in Fig. 1 usually denoted as B, increases almost linearly with increasing temperature as shown in Fig. 2. $(\delta B/\delta T)_{B=0} = 6.5 \times 10^{-6}$ m$^3$ mol Kg$^{-2}$ K$^{-1}$ in accordance with values reported previously (Maimunah, 2003, 2004). The temperature at which B ($M_n$, T) = 0, defined as the θ temperature, is determined to 20.8 ± 0.3 °C. A value for infinite molecular weight obtained from cloud-point curves is 21.2 °C (Maimunah, 1980, 2003, 2004); other values reported for the system poly(dimethyl siloxane)/trans-decalin at in the interval 20 – 24 °C. The expansion of the osmotic pressure as a power series in c will be given elsewhere (Maimunah, 1980, 2003, 2004).

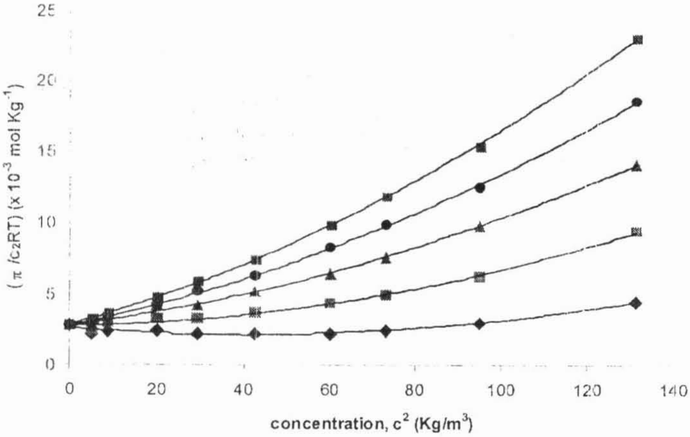

Fig. 1: (p/RTc) as a function of concentration, c for PDMS in trans-decalin at

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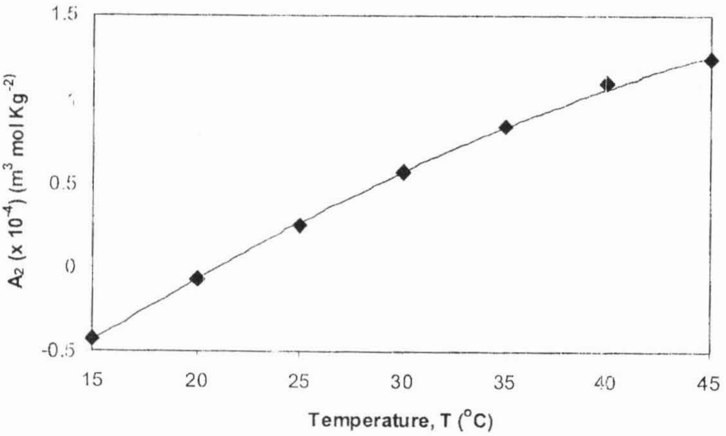

Fig.2: The temperature dependence of second virial coefficient, B for the system PDMS-trans decalin.

It has been suggested that there is a concentration above which the distribution of statistical subunit of a polymer chain is more or less homogeneous. This concentration, $c^+$ defined as

$$c^+ = [M_n/(N_A\langle s^2\rangle)^{3/2}] \quad \ldots\ldots \quad (13)$$

where $\langle s^2\rangle$ is the mean square radius of gyration and $N_A$ is Avogadro's number, has for the present system been estimated using the unperturbed mean-square radius gyration ($\langle s^2\rangle_0$ (m$^2$) = 7.86 x $10^{-22} M_W$). In sufficiently dilute solution where the molecules can be considered as individual units, the physical situation is mainly described by the intrinsic properties of the solute molecules. As the concentration is increased the molecules begin to overlap and to entangle. At still higher concentrations the distribution of segment will become uniform and another physical situation will prevail. In order to elucidate differences between dilute and concentrated solution behaviour, appropriate reference states are an infinitely dilute solution and a solution at the concentration $c = c^+$, respectively. In Fig. 3, $B^*$ is the ratio of the initial slope in Fig. 1, B to the slope at $c = c^+$ shown as a function of temperature. This ratio increases with increasing temperature to a plateau value of approximately 0.7. This may indicate that the transition from dilute to concentrated solution behaviour depends on the "goodness" of the solvent.

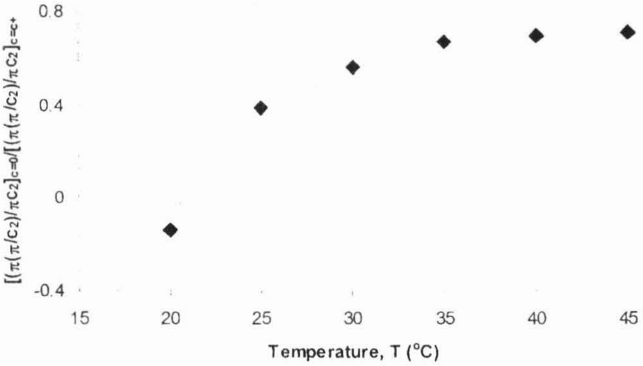

Fig.3. the ratio of the initial slope in Fig. 1 to that at $c^+$ as a function of temperature for PDMS in trans-decalin.

From the equilibrium conditions in a system consisting of a liquid solution of a macromolecular solute separated from pure solvent by membrane, permeable to component 1 (solvent) only and from the definition of the partial molar volume of the pure solvent, $V_1^o$, it follows that

$$\Delta\mu_1 = (-p\ V_1^o)[1 - ((\kappa_1)p/2) + \theta(p^2)] \quad ....... \quad (14)$$

$\Delta\mu_1$ is the chemical potential of mixing and $\kappa_1$ is the partial molar compressibility of the solvent in the solution. Since the compressibility at atmospheric pressure of a pure organic solvent, $\kappa_{10}$, is of the order of magnitude $10^{-10}$ Pa$^{-1}$ (it is assumed that $\kappa_1 = \kappa_{10}$ over the concentration interval studied) and since the measured osmotic pressures are less than $10^4$ Pa, $\Delta\mu_1$ is in good approximation as

$$\Delta\mu_1 = (-pV_1^o) \quad ....... \quad (15)$$

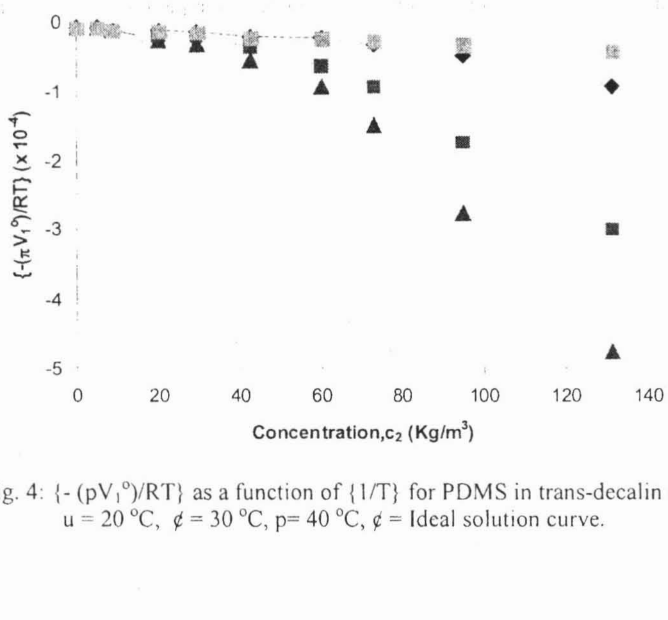

Fig. 4: {- $(pV_1^o)/RT$} as a function of {1/T} for PDMS in trans-decalin at u = 20 °C, ¢ = 30 °C, p= 40 °C, ¢ = Ideal solution curve.

In Fig. 4, $[(-pV_1^o)/RT]$ is plotted against concentration, $c_2$. It illustrated the change of $\Delta\mu_1$ with concentration, $c_2$ and temperature, T. Although the experimental curve at 20 °C follows the ideal solution curve well, except at higher concentrations, the thermodynamic state at 20 °C is not be considered ideal. For a macromolecular solution it is a state where enthalpy and entropic contributions to $\Delta\mu_1$ cancel each other to give a state that can be called "pseudo-ideal" (Flory, 1953; Rudin, 1982).

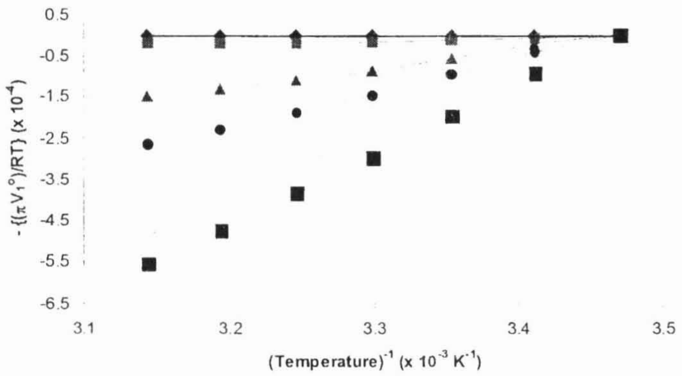

Fig. 5. $\{-(pV_1^o)/RT\}$ as a function of $\{1/T\}$ for PDMS in trans-decalin at the weight fraction, $W_2$
u = $W_2 = 0.011$; ¢ = $W_2 = 0.034$; p = $W_2 = 0.083$; l = $W_2 = 0.11$; and ¢ = $W_2 = 0.15$.

The partial molar enthalpy, $\Delta H_1$, and the partial molar entropy, $\Delta S_1$ of mixing are given by

$$\Delta H_1 = \{\delta(\Delta\mu_1/T)\}/\{\delta(1/T)\} = \{\delta((-pV_1)/T)\}/\{\delta(1/T)\} \quad \ldots\ldots \quad (16)$$

and

$$\Delta S_1 = -\{\delta(\Delta\mu_1)/\{\delta T\}\} = -\{\delta(pV_1)/\{\delta T\}\} \quad \ldots\ldots\ldots\ldots\ldots \quad (17)$$

respectively. From Fig. 5, 6 and 7, it is seen that $\Delta H_1$ and $\Delta S_1$ are positive and increase with increasing concentration. The mixing process is accordingly endothermic and the q-temperature is related to an Upper Critical Solution Temperature, UCST. $\Delta H_1$ and $\Delta S_1$ data at the average temperature 30 °C are given in Table 2.

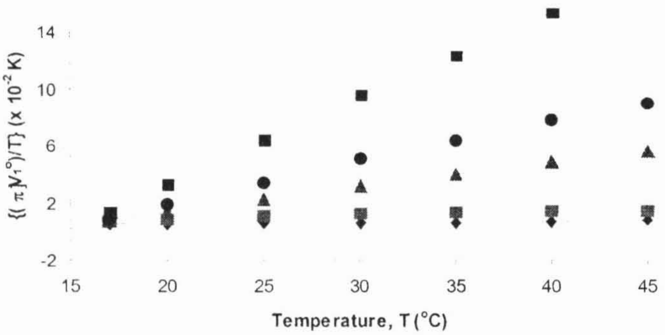

Fig. 6. $\{(pV_1^o)/R\}$ as a function of $\{1/T\ (^oC)\}$ for PDMS in trans-decalin at the weight fraction, $W_2$;
u = $W_2 = 0.011$; ¢ = $W_2 = 0.034$; p = $W_2 = 0.083$; l = $W_2 = 0.11$; and ¢ = $W_2 = 0.15$.

Table 2. Partial molar entropies, $DS_1$ and enthalpies, $DH_1$ of mixing of the solvent trans-decalin at 30 °C for the system.

| $W_2$ (x $10^{-2}$) | $\Delta S_1$<br>($10^{-3}$ J mole$^{-1}$ K$^{-1}$) | $\Delta H_1$<br>(J mole$^{-1}$) |
|---|---|---|
| 1.057 | 0.21 | 0.052 |
| 2.246 | 1.00 | 0.270 |
| 3. 368 | 2.27 | 0.640 |
| 4.841 | 4.62 | 1.320 |
| 6.864 | 9.30 | 2.670 |
| 8.309 | 14.3 | 4.130 |
| 10.750 | 24.7 | 7.140 |
| 14.820 | 50.0 | 14.400 |

The polymer solution thermodynamics (Flory, 1953; Morawetz, 1975), the chemical potential of mixing is usually split into a combinatorial and a residual term.

$$\Delta\mu_1 = \Delta\mu_1^{comb} + \mu_1^R \quad ....... \quad (18)$$

$\Delta\mu_1^{comb}$ is in the zeroth approximation given by

$$\Delta\mu_1^{comb} = -T\Delta S_1^{comb} = RT\,[\ln(1-\varphi_2) + (1-1/r)\,\varphi_2] \quad ....... \quad (19)$$

where $\varphi_2$ is the segment fraction of component 2. The solvent-solute interaction parameter $c^{\circ}\ \{(\mu_1^R)/(RT\varphi_2^2)\}$ can be written as

$$c = -\{(pV_1^{\circ})/(RT\varphi_2^{\,2})\} - \{(1/\varphi_2^2\,[\ln(1-\varphi_2) + (1-1/r)\,\varphi_2]\} \quad ....... \quad (20)$$

In the Flory terminology (Flory, 1953) r is the ratio $(n_{20}^* M_2)/(n_{10}^* M_1)$ and $\varphi_2$ is related to the weight fraction of the solute, $W_2$, through $\varphi_2 = \{(W_2\, n_{20}^*)/\ [(1 - W_2)\,(n_{10}^* + W_2\, n_{20}^*]\}$. $n_{10}^*$ and $n_{20}^*$ are the characteristic specific volumes of solvent and segment, respectively, and are determined from the specific volumes, $n_{10}^*$ and the thermal expansion coefficient, $a_{10}$, of the pure components (Maimunah, 1980) $n_{10}^* = \{n_{10}^*[1 + (a_{10}T)/(3(1+ a_{10}\,T))]^{-3}\}$. Values of the characteristic parameters $n_{10}^*$ and $n_{20}^*$ are calculated from data in previous works (Maimunah, 1980).

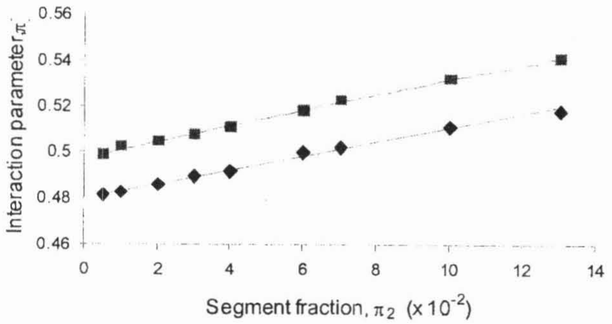

Fig. 7. The dependence of the solvent-solute interaction parameter, c on segment fraction, $j_2$ for the system PDMS/ tans-decalin at u = 20 °C, ¢ = 40 °C

The parameter c determined according to Eq. (20) is plotted vs. $\varphi_2$ in Fig. 7. c increases with increasing segment fraction of the macromolecule, $j_2$ and can be represented by

$$c = c_1 + c_2\varphi_2 \quad \ldots\ldots \quad (21)$$

Over the $\varphi_2$ range studied. $c_1$ and $c_2$ are temperature dependent parameters. Their temperature dependence is illustrated in Fig. 8. $c_1$ and $c_2$ could equally well have been represented by function linear temperature. Values $c_1$ and $c_2$ are given in Table 3. Although the splitting of the chemical potential of mixing, $\Delta\mu_1$, into a combinatorial and a residual term presupposes a uniform distribution of segments, the application of this procedure (choice of $\Delta\mu_1^{comb}$ as a reference state) to the present system gives $c_1$ and $c_2$ values in good accordance with values reported previously. The slight difference between the values is probably due to the different molecular weight used (c has been observed to depend on $M_2$. Furthermore, differences in the extrapolation procedures can also affect the values of $c_1$ and $c_2$.

## Conclusion

General and thermodynamically equations for osmotic pressures have been developed based on a simple equation of states. The equations have been applied to actual osmotic-pressure data using data correlation. To use these equations we studied the osmotic properties of poly(dimethyl siloxane), PDMS in trans-decalin over the temperature range of 20 to 40 °C at concentrations from 0.5 to 15 wt.%. Virial coefficients were determined from the concentration, c dependence of the osmotic pressure. The second virial coefficient of the system, B, increases almost linearly with increasing temperature and suggested the existence of one theta, θ temperature. $(\delta B/\delta T)_{B=0} = 6.50 \times 10^{-6}$ m$^3$ mol Kg$^{-2}$ K$^{-1}$. The theta, θ temperature, is determined to 20.8 ± 0.3 °C which is equivalence to the value for infinite molecular weight obtained from cloud-point curves (21.2 °C) and other values reported for the system at in the interval 20 – 24 °C. The change of $\Delta\mu_1$ with concentration, $c_2$ and temperature, T showed that the experimental curve at 20°C follows the ideal solution curve well, except at higher concentrations. The thermodynamic state at 20 °C is not being considered ideal. For a macromolecular solution it is a state where enthalpy and entropic contributions to $\Delta\mu_1$ cancel each other to give a state that can be called "pseudo-ideal". The mixing process between poly(dimethyl siloxane) and trans-decalin is endothermic and the q-temperature is related to an Upper Critical Solution Temperature, UCST. It was found that the mixing enthalpy $\Delta H$ and mixing entropy $\Delta S$ of trans-decalin, determined from the temperature dependence of the osmotic pressure, were negative and that the magnitude of $dH$ was slightly larger than that of $T\Delta S$. This indicated that the dissolution of the polymers in trans-decalin was dominated by enthalpy or intermolecular interaction. The existence of local structure in the solution was revealed by negative entropy.

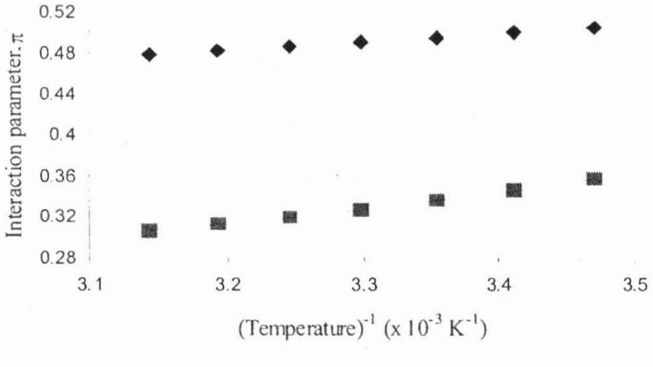

Fig. 8. The temperature dependence of $\chi_1$ and $\chi_2$.

Table 3. Thermodynamic parameters for the system PDMS/trans-decalin.

| T (°C) | B<br>($\times 10^{-4}$ $m^3$ mol $Kg^{-2}$) | $c_1$ | $c_2$ |
|---|---|---|---|
| 20 | -0.06 | 0.5011 | 0.341 |
| 25 | 0.25 | 0.4959 | 0.334 |
| 30 | 0.58 | 0.4909 | 0.324 |
| 35 | 0.85 | 0.4869 | 0.314 |
| 40 | 1.11 | 0.4826 | 0.309 |

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