Adsorption Effects in the Flow of Polymer Solutions through Capillaries

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ABSTRACT: The hydrodynamic resistance of adsorbed polymer layers to the flow of aqueous and organic polymer solutions in stainless steel capillary tubes was quantified experimentally by using a surface treatment method. The effective hydrodynamic thickness of the adsorbed polymer was found to be as high as 1.5 μm, which is much larger than the root-mean-square end-to-end distance of the free macromolecules in solution. Furthermore, it decreased with increasing shear stress.

1. Introduction

Adsorbed polymer layers are of interest in applications such as liquid chromatography, filtration, capillary viscometry, and enhanced oil recovery. In such applications, the adsorbed layers may represent both an additional resistance to flow (i.e., permeability reduction) and a loss of the costly polymeric additives.[1-6] Furthermore, the aqueous polymer solutions that are generally used may exhibit significant slip effects when flowing through small channels.[7-10] Moreover, polymer adsorption on the walls of such small channels may affect the flow behavior significantly.[1,4,5,8,11] and hence warrants quantification.

It is well-known that polymer molecules adsorb well at solid interfaces[12-14] and in the case of a flowing polymer solution may lead to an increase in the flow resistance. This effect has been well documented in the literature and has served as a measure of the thickness of such adsorbed polymer layers,[12,15-19] primarily for dilute polymer solutions flowing through capillary tubes.

It appears that the hydrodynamic effect of adsorbed polymers in flowing, nondilute polymer solutions (where intermolecular interactions may be significant) has not received much attention in the technical literature. Most of the existing experimental studies[1,11,16,17,20,21] usually compare the flow behavior of the solvent through a given capillary tube before and after the flow of the polymer solution. Then the extra resistance to the flow of the solvent (due to the adsorbed polymer layer) is expressed as an effective thickness of the adsorbed layer, δ_a

$$\delta_a/R = 1 - (Q_a/Q)^{1/4} \tag{1}$$

where Q_a and Q are the flow rates in the presence and absence of adsorption, respectively, and R is the tube radius. It should be noted that in the above technique some desorption may occur when the solvent is used to determine the effect of adsorption. Consequently, such a procedure defines only the resistance of the residual irreversibly adsorbed layer to the flow of the solvent. Furthermore, it has been generally found that at low shear rates the effective hydrodynamic thickness (EHT) of the residual adsorbed layer is of the order of the hydrodynamic size of the free polymer molecules in solution.[1,11,27,28] This led most researchers to assume that the polymer coils were adsorbed in a unimolecular layer without significant distortion. This conclusion has not been confirmed when the actual resistance of the adsorbed layer to the flowing polymer solution is considered. Clearly, such information would be invaluable to the understanding of hydrodynamic adsorption effects, and in particular when flow through small channels is involved.

In this work we illustrate an alternate method for studying hydrodynamic adsorption effects in capillary tube flow where slip effects are also present. It is our intention to demonstrate that adsorption effects can be significant and hence must be considered in the design and interpretation of flow experiments and processes in which adsorption occurs.

2. Experimental Section

In this study the effect of adsorption is demonstrated for the flow of three polymer solutions through stainless steel capillary tubes. The polymer solutions were 1% aqueous partially hydrolyzed polyarcylamide (Separan AP-30, MW = 1.75×10^6, Dow Chemical Co.) and (b) 4% and 3.15% polystyrene (MW = 1.8×10^6, Pressure Chemical Co.) in toluene and decalin, respectively. The flow experiments were carried out in a constant-pressure rheometer (Cohen, 1981) for tubes of diameter 0.0184–0.12 cm and L/D (length/diameter) ratios of 250–2000. In these experiments the measured pressure drop data were corrected for the excess pressure drop due to end effects by using the standard Bagley method,[8,24] and the resulting shear stress–shear rate curves were then calculated.

The experimental method for studying adsorption effects was based on the determination of the pressure drop–flow rate relationship for a given tube diameter in the presence and absence of adsorption effects. The case of adsorption refers to the natural adsorption of polymer onto the capillary wall, which is in contact with the flowing polymer solution. In contrast, the case of no adsorption was achieved by chemically treating tubes with a solution of 2% dimethyldiethoxysilane in a solvent mixture made of 95% ethanol and 5% distilled water, adjusted to a pH of about 4.5–5.5 with acetic acid. The details of the treatment procedure, which is based on the method described by Arkles,[22] can be found elsewhere.[8]

Finally, the rheological characterization of the polymer solutions at steady shear was obtained by using a cone-and-plate viscometer (Weissenberg Model R-18). This viscometer is a standard rheological tool and its detailed description, method of operation, and data reduction can be found in two excellent references.[23,24]

3. Effective Hydrodynamic Thickness of the Adsorbed Polymer

The effect of adsorption can be quantified in terms of the effective reduction in the tube diameter, thereby allowing an effective hydrodynamic thickness (EHT) of the adsorbed polymer layer to be defined.

In order to calculate the EHT, we approximate the shear stress–shear rate behavior of the polymer solutions by a power law model

$$\tau_w = k\dot{\gamma}_w{}^n \tag{2}$$

where τ_w and $\dot{\gamma}_w$ are the wall shear stress and corresponding shear rate and k and n are the power law parameters. Then from the flow curves for a given tube diameter in the absence and presence of adsorption, we can calculate the EHT of the adsorbed polymer. The equation corresponding to eq 1 for a non-Newtonian power law fluid is

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$$\delta_a/R = 1 - (\dot{\gamma}_{ad}/\dot{\gamma}_a)^{n/(3n+1)} \qquad (3)$$

where $\dot{\gamma}_a$ and $\dot{\gamma}_{ad}$ are the apparent Newtonian shear rates (based on the actual tube diameter) in the absence (silane-coated tubes) and presence (untreated tubes) of adsorption, respectively.

Since polymer solutions may also exhibit slip effects,[7-10] both adsorption and slip may occur simultaneously. Therefore, in such a case, the determined EHT, δ_a, reflects the effect of adsorption in the presence of slip flow. Although the above method does not require an explicit quantification of slip effects, it will be shown that by quantifying slip flow with surface-treated tubes, one can demonstrate the effectiveness of the treatment method in allowing the separation of slip and adsorption effects.

The slip flow phenomenon is believed to be due to a very thin, low-viscosity region near the tube wall. Slip effects are usually quantified in terms of the effective slip velocity, V_s, at the tube wall

$$V_s = Q_s/(\pi D^2/4) \qquad (4)$$

where Q_s is the contribution of slip to the total flow rate and D is the tube diameter. The effective slip velocity, V_s, can be determined experimentally, given the flow curves for different tube diameters[25]

$$V_s = \frac{1}{8}\,\frac{\partial}{\partial(1/D)}\left(\frac{32Q}{\pi D^3}\right)_{\tau_w} \qquad (5)$$

where plots of $32Q/\pi D^3$ vs. $1/D$ should yield straight lines with a slope equal to V_s. This analysis is based on the assumption that the slip layer is thin and that no adsorption effects are present over the range of diameters at which V_s is determined. Equation 5 then serves to analyze slip flow in surface-treated tubes and to demonstrate the effectiveness of the surface treatment method.

4. Results and Discussion

In this section the effectiveness of the adsorption elimination technique and its utility in the quantification of slip flow for treated capillary tubes are demonstrated. Subsequently, the hydrodynamic adsorption effects are presented and discussed for three different polymer solutions.

4.1. Effectiveness of Surface Treatment. The presence of adsorbed polymer layer can be detected through an effective reduction in the tube diameter. Therefore, the comparison of the surface-treated capillary diameters before and after the flow of a given polymer solution serves as a measure of the effectiveness of the surface treatment method.

The capillary diameters were determined from flow experiments with Newtonian fluids of known viscosity (toluene, water, and water–glycerin solutions). These experiments were carefully designed such that the errors in both the pressure drop and flow rate measurements were not larger than 0.5% (Cohen, 1981). Typical results for different tube diameters and polymer solutions are given in Table I. The accuracy of these calibration experiments is demonstrated by the standard deviation (based on five or more determinations), which is no larger than 0.78% and less than 0.4% for all but a single diameter calibration. The important observation is that the variation in the tube diameters before and after the flow of the polymer solution is about the magnitude of the standard deviation of the diameter calibration; moreover, it is random. Consequently, we conclude that the surface treatment is effective in minimizing any possible flow reduction due to irreversibly adsorbed polymer.

Table I
Tube Diameters and the Effectiveness of the Surface Treatment Method

av tube diam, cm	% dev	recalibrated tube diam,[a] cm
0.018 85	0.397	
0.019 06	0.115	
0.026 62	0.207	0.026 72 (1)
0.035 84	0.309	0.035 73 (1)
0.052 58	0.308	0.052 63 (2)
0.084 37	0.072	0.084 27 (3)
0.109 70	0.784	

[a] Determined with the appropriate solvent after the run with the following polymer solutions: (1) 1% Separan AP-30; (2) 4% polystyrene in toluene; (3) 3.15% polystyrene in decalin.

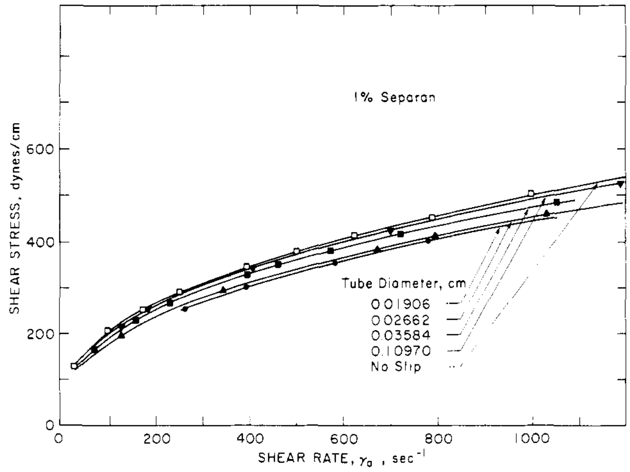

Figure 1. Flow curves for 1% aqueous Separan AP-30 solution (treated tubes; deviations from the uppermost curve are a measure of slip effects).

Additional support of the effectiveness of the surface treatment method in minimizing adsorption effects can be provided via the quantification of slip flow as described in section 3.

Typical flow curves (surface-treated tubes) for a 1% Separan AP-30 solution expressed in terms of the wall shearing stress τ_W vs. the apparent shear rate $\dot{\gamma}_a$ ($\dot{\gamma}_a = 32Q/\pi D^3$) are illustrated in Figure 1. Also included in this figure is the prediction (no-slip curve) from the viscometric cone-and-plate data. It is seen that the flow curves for the capillary tubes exhibit a progressively higher shear rate (for a given τ_w) compared with the no-slip curve as the tube diameter decreases. This slip behavior was quantified by determining the slip velocity as described previously. As an illustration, the resulting plots of the apparent shear rate ($\dot{\gamma}_a$) vs. $1/D$ for the 1% Separan solution are shown in Figure 2. The linearity of these plots indicates that the slip velocity (i.e., the slope of the curves divided by 8, eq 5) in these surface-treated tubes is independent of the capillary tube diameter. These results demonstrate the *effectiveness of the surface treatment method.*

4.2. Adsorbed Polymer Layer. The effect of adsorption is demonstrated by the flow curves for the aqueous and organic solutions for different capillary diameters (Figures 3–5). The curves designated by NST, ST, and R refer to no surface treatment, surface treatment, and the viscometric (cone and plate) rheological prediction, respectively.

The results indicate that for a given shear rate the shear stress is higher for the untreated tubes (curve NST) than for the treated tubes (curve ST). In other words, a higher apparent viscosity is exhibited in the untreated tubes.

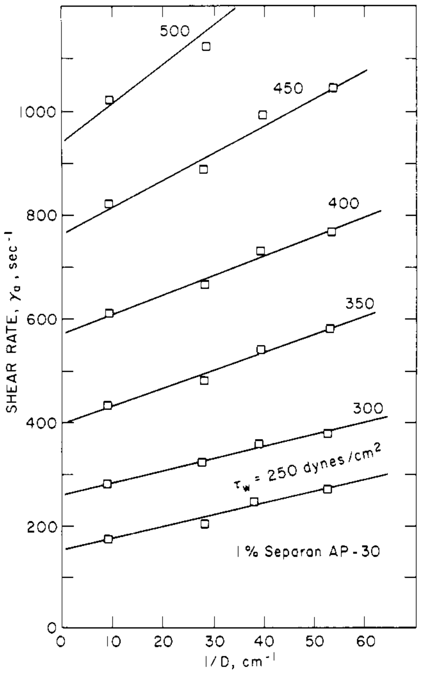

Figure 2. Shear rate vs. $1/D$ (1% Separan, treated tubes). Evaluation of slip.

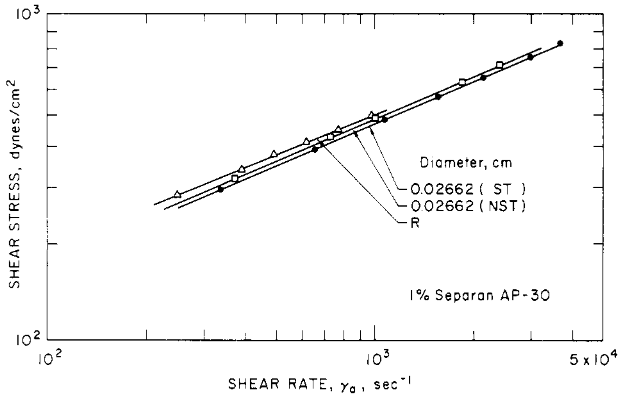

Figure 3. Effect of adsorption on the flow curves for 1% Separan (D = 0.026 62 cm).

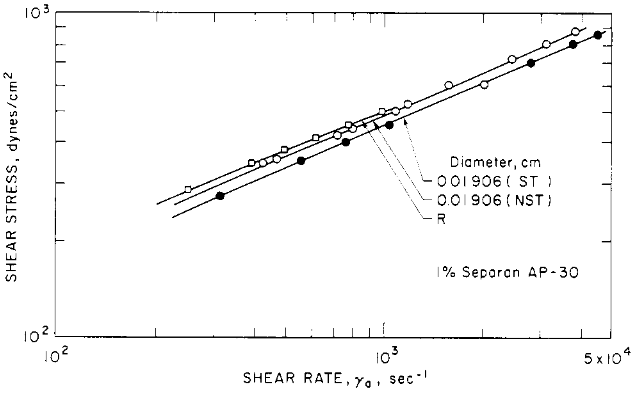

Figure 4. Effect of adsorption on the flow curves for 1% Separan (D = 0.019 06 cm).

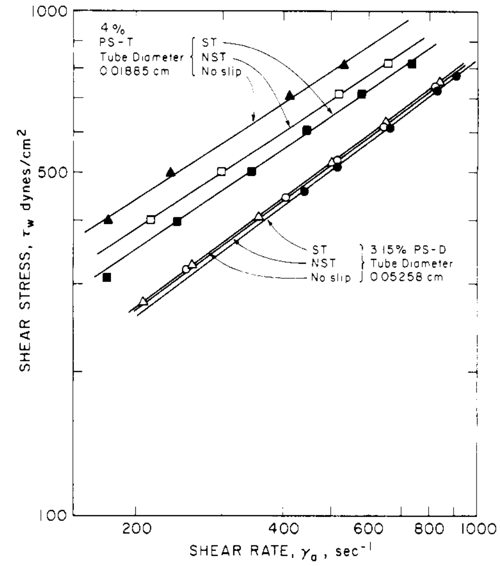

Figure 5. Effect of adsorption on the flow of polystyrene solutions.

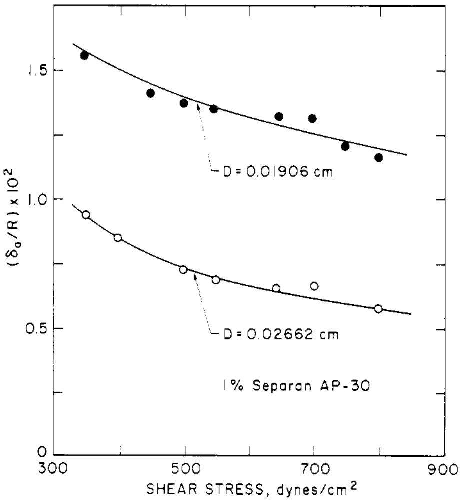

Figure 6. Effective hydrodynamic thickness for an adsorbed polyacrylamide layer as a function of shear stress (1% Separan AP-30).

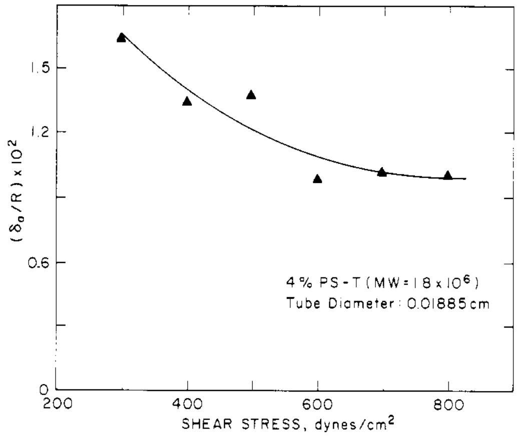

Figure 7. Effective hydrodynamic thickness for an adsorbed polystyrene layer as a function of shear stress (for 4% PS–toluene solution, MW = 1.8 × 10^6).

It is also noted that even in the presence of adsorption effects, the flow curves (both ST and NST) are below the viscometric predictions (curve R). This means that both slip and adsorption occur simultaneously, with slip effects dominating at least for the given set of conditions.

The above results (Figures 3–5) were used to determine the EHT (δ_a) relative to the tube radius (R) as a function of the wall shear stress for the three different polymer solutions, as shown in Figures 6–8. The general trend that is revealed in the above results is the decrease in the EHT of the adsorbed polymer layer with increasing wall shear stress. This may be attributed to the increased alignment of the loose macromolecular segments of the adsorbed

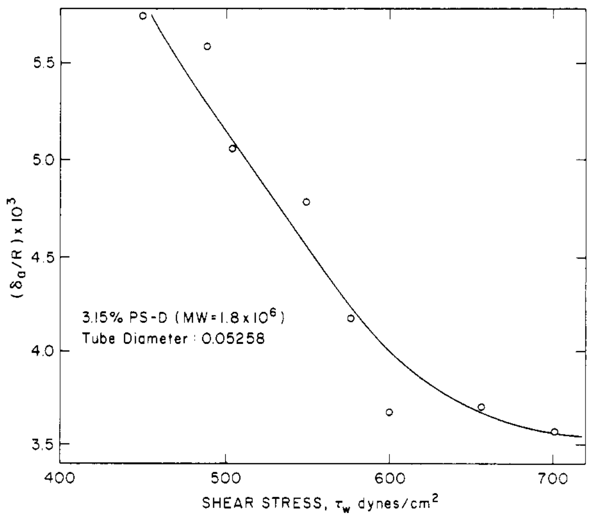

Figure 8. Effective hydrodynamic thickness for an adsorbed polystyrene layer as a function of shear stress (for 3.15% PS-decalin, MW = 1.8 × 10^6).

Table II
Effective Hydrodynamic Thickness for Polyacrylamide and Polystyrene

polymer solution	δ_a, µm	τ_w, dyn/cm^2
1% Separan/water	1.5–0.8	250–800
4% PS-toluene (MW = 1.8 × 10^6)	1.5–0.8	250–800
3.15% PS-decalin (MW = 1.8 × 10^6)	1.5–0.9	450–700

polymer and/or the tearing of adsorbed polymer as the wall shear stress increases. An additional observation is that the EHT increases somewhat with decreasing tube diameter. However, this change is barely outside the experimental accuracy and hence remains to be quantified experimentally.

The range of the EHT (δ_a) values for the three polymer solutions is summarized in Table II. These values of the EHT are larger than the root-mean-square end-to-end distance, $\langle r^2\rangle^{1/2}$, of the polymer molecule by as much as a factor of 5 for Separan AP-30 ($\langle r^2\rangle^{1/2} \approx 2360$ Å[8,28,30]) and an order of magnitude for polystyrene ($\langle r^2\rangle^{1/2} = 1400$ Å[28,31,32]). The present EHT values are much larger than previously reported for flow experiments in capillary tubes[11,16,17,19,27] and porous media.[1,4,5,28] The differences are believed to be due to the different determination methods (as described previously), which actually define two different hydrodynamic thicknesses. All of the previously published results (to the knowledge of the authors) define only the effect of the residual adsorbed polymer on the flowing solvent (or dilute solutions with shear independent viscosity). Consequently, these studies have resulted in a residual EHT that is of the order of the hydrodynamic size of the macromolecules in the solution.[27] An exception is the recent study of Gramain and Myard,[33] who report abnormally large thicknesses of the residual adsorbed layer (in Nuclepore filters), which were found to increase with flow rate. Their results contradict the lower commonly observed EHT of the residual adsorbed layers, which are found to decrease with flow rate in both porous media and capillary flow experiments. The reason for the above discrepancy is not clear and it warrants further experimental clarification in order to explain the unusual results of Gramain and Myard.[33]

In closing, the present results are believed to reflect more closely the actual effect of the adsorbed polymer layer on the flowing polymer solutions; hence they represent a more realistic description of the hydrodynamic adsorption effect. Furthermore, we suggest that the present results dispute the validity of the procedure of determining polymer retention and permeability reduction in porous media due to adsorption based on a unimolecular adsorbed layer, as has been done by several investigators.[1,3,28,29]

5. Summary

A method of studying hydrodynamic adsorption effects in the flow of polymer solutions through capillary tubes was devised. The current method enables the quantification of the resistance of adsorbed layers to the flow of polymer solutions. This study revealed that the effect of adsorption for capillary tube flow of polyacrylamide and polystyrene solutions was reflected by a significant flow reduction (by as much as 20% in some cases). Of course, the flow reduction would be even more important for smaller tube diameters and, as shown in Figures 6–8, at low stress levels. Finally, the thicknesses of the adsorbed polymer layers found in this work (0.8–1.5 µm) are larger than found previously (usually less than 0.3 µm.[1,5,11,16–18,27,28]

Nomenclature

D	tube diameter
k	power law consistency index
n	power law parameter
Q	flow rate
Q_a	flow rate in the presence of adsorption
Q_s	contribution of slip to the total flow rate
R	tube radius
V_s	slip velocity
$\dot\gamma$	shear rate
$\dot\gamma_a$	apparent Newtonian shear rate ($32Q/\pi D^3$)
$\dot\gamma_{ad}$	apparent Newtonian shear rate in the presence of adsorption
η_a	apparent Newtonian Viscosity
τ_w	wall shear stress

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Nonlinear Dielectric Effect of Flexible Polar Polymers

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ABSTRACT: The nonlinear dielectric effect (NLDE) is the change of the dielectric constant of a medium when subject to a strong electric field. It is the result of a sensitive interplay between the fourth and second moments of the dipole moment, $\langle \mu^2 \rangle^2 \sigma$, where $\sigma = \langle \mu^4 \rangle / \langle \mu^2 \rangle^2 - 5/3$, μ is the dipole moment, and the angular brackets denote statistical averaging over all internal configurations in the absence of the external field. σ is a measure of the non-Gaussian behavior of a polymeric chain of x repeat units, since $\sigma \rightarrow 0$ as $x \rightarrow \infty$. We employ the rotational isomeric state models of Flory and co-workers to calculate the NLDE of the stereoregular polymers poly(oxyethylene glycol) and poly(oxyethylene dimethyl ether), the stereoirregular poly(vinyl chloride) and its oligomers 2,4-dichloropentane and 2,4,6-trichloroheptane, and the copolymer poly(styrene-*co*-*p*-bromostyrene). We examine the dependence of the NLDE on chain length, tacticity, and, in the case of the copolymer, composition. We find the NLDE to be sensitive to all these parameters and conclude that the NLDE is an important configurational property of a polar polymer.

The non-Gaussian behavior of short polymeric chains has been the subject of many theoretical studies.[1,2] These have shown that the deviation of the probability distribution function of the end-to-end distance from a Gaussian distribution is related to the even moments of the end-to-end distance. Mathematical methods have been devised to calculate these moments, but to date only the second moments of a polymer have been the subject of extensive experimental study. These include the mean square end-to-end distance $\langle r^2 \rangle$, mean square dipole moment $\langle \mu^2 \rangle$, and radius of gyration $\langle s^2 \rangle$.

One experimental approach to evaluating the higher moments of the end-to-end distance is by scattering radiation from a polymer. The theory for this is given by Flory.[1] In this paper we wish to discuss an experiment by which a higher moment of the dipole moment of a polymeric chain may be obtained, namely, the nonlinear dielectric effect (NLDE) or dielectric saturation.[3-5] In this experiment a strong electric field is applied across a medium to render it anisotropic, and a low-frequency weak probe electric field is used to detect the anisotropy of the dielectric constant. If a high-frequency probe electric field were used, then the experiment would observe the anisotropy of the refractive index squared, i.e., the Kerr effect.[4] The applied electric field will influence the molecule in two ways: first, it will orient the total dipole moment and second, it will perturb the equilibrium conformations of the molecule in favor of those conformations with the larger dipole moment. Thus the orienting effect of the field will cause a decrease while the perturbation of the conformations will cause an increase in the dielectric constant, the net result depending on a sensitive interplay between the two effects. For a flexible polar molecule the observed effect depends essentially on $\langle \mu^2 \rangle^2 \sigma$, where

$$\sigma = \frac{\langle \mu^4 \rangle}{\langle \mu^2 \rangle^2} - \frac{5}{3} \tag{1}$$

μ is the dipole moment, and the angular brackets denote statistical averaging over all internal configurations of the flexible molecule in the absence of a strong electric field. σ tends towards zero as $1/x$ as x tends to infinity, where x is the number of repeat units in the polymeric chain. However, $\langle \mu^2 \rangle^2$ increases as x^2 and so the product $\langle \mu^2 \rangle^2 \sigma$ increases linearly with x for large values of x. The factor σ differs significantly from zero only when the chain is relatively short, say 50 bonds or less, and is therefore a measure of the non-Gaussian behavior of the chain.

Recently, we presented calculations of the NLDE of the α,ω-dibromoalkanes[6] using the rotational isomeric state scheme (RIS) with nearest-neighbor interactions. Matrix multiplication methods of Flory[1] were used to calculate the fourth and second moments of the dipole moment and we found good agreement with the experimental results. We also found the NLDE calculation to be sensitive to the details of the RIS model used, so that it could be used with other configurationally sensitive quantities to characterize a polymer.

In this paper we present calculations of the NLDE for a number of commonly studied polymers: poly(oxyethylene glycol), $H(OC_2H_4)_xOH$ (POEG), poly(oxyethylene dimethyl ether), $CH_3(OC_2H_4)_xOCH_3$ (POEDE), the stereoirregular poly(vinyl) chloride), $CH_3(CHClCH_2)_xH$ (PVC) and its oligomers 2,4-dichloropentane (DCP) and 2,4,6-trichloroheptane (TCH), and the copolymer poly(styrene-*co*-*p*-bromostyrene), $CH_3(CHRCH_2)_xH$ ($R = C_6H_4Br$, C_6H_5) (PPBS–PS). We examine the dependence of the NLDE on chain length, tacticity, and, in the case of the copolymer, composition. We employ the same RIS models as used by us[7-9] recently to calculate the Kerr effect and

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