

## Some Properties of Poly(ethylene oxide)<sup>1</sup> in Aqueous Solution

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### INTRODUCTION

While low molecular weight polymers of ethylene oxide have been familiar articles of commerce for many years, it is only very recently with the large scale preparation of truly high polymers of ethylene oxide that the strikingly unique character of poly(ethylene oxide) has become apparent.<sup>2,3</sup> Over the enormous molecular weight range from the liquid polyglycols to resins with molecular weights of ten million or more, poly(ethylene oxide) retains complete room temperature miscibility with water. While water solubility in the case of ethylene oxide polymers is not surprising in the light of familiarity with polyglycol liquids and waxes, it is instructive to compare this solubility in water with that of several closely related structures (Table I). In contrast with the sol-

TABLE I  
Polyether Structures

	Polymer unit	Soluble in water at room temperature
Methylene oxide	$-\text{CH}_2-\text{O}-$	No
Ethylene oxide	$-\text{CH}_2-\text{CH}_2-\text{O}-$	Yes
Acetaldehyde	$-\text{CH}-\text{O}-$   $\text{CH}_3$	No
Propylene oxide, tri- methylene oxide	$-(\text{C}_3\text{H}_6)\text{O}-$	No

ubility of poly(ethylene oxide) is the insolubility in water under ordinary conditions of such closely related structures as poly(methylene oxide) and poly(propylene oxide). Even the isomer of poly(ethylene oxide), polyacetaldehyde, is insoluble in water.

It is the purpose of this paper to discuss the properties of poly(ethylene oxide) in aqueous solution from the point of view of the behavior of a high molecular weight, neutral molecule in media of high dielectric constant and to relate

these properties of poly(ethylene oxide) to current polymer solution theory. From this examination, an interpretation of the rheology of aqueous solutions of ethylene oxide polymers will be developed.

### EXPERIMENTAL

The polymers investigated in this study are some of the Carbowax compounds and several Polyox water-soluble resins of Union Carbide Chemicals Company as well as experimental high polymers, methods of preparation of which have recently been disclosed.<sup>3</sup> Reduced viscosities have been measured in distilled water using a Ubbelohde-type capillary viscometer with a capillary of one millimeter internal diameter and two meter length. This viscometer was so constructed that viscosities could be measured under several pressure heads, thus permitting extrapolation of all reduced viscosities to zero flow rate (zero shear rate). Intrinsic viscosities were then determined by extrapolation of zero shear rate reduced viscosities to infinite dilution.

The inverse solubility-temperature relationship was investigated by sealing solutions of poly-

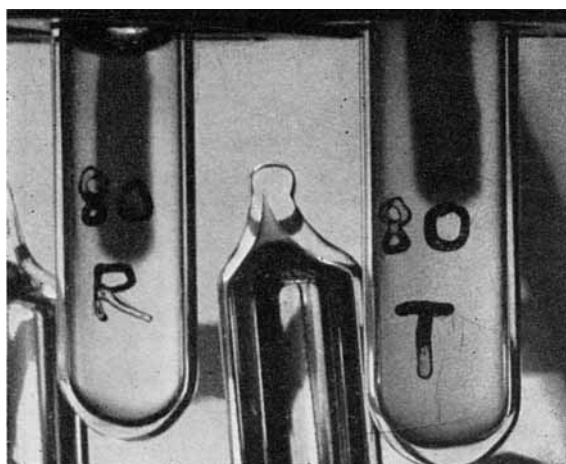


Fig. 1. Poly(ethylene oxide) above the upper temperature limit of solubility in water.

(ethylene oxide) in water at appropriate concentrations in glass tubes. These tubes were then suspended in a stirred heating bath (see Fig. 1) in such a manner that the rack suspending the sealed tubes was mechanically connected to the motor mount of the bath stirrer. The tubes were thus subjected to constant vibration as the temperature of the bath was slowly raised (at a rate of about  $1/4^{\circ}\text{C./min.}$ ). The precipitation temperature ( $T_p$ ), or upper temperature limit of solubility of the ethylene oxide polymers, was observed as a cloud point which occurred sharply within a tenth of a degree temperature rise and occurred reproducibly within  $\pm 1^{\circ}$ .

In Figure 1, polymers can be seen to have precipitated in some instances as a reticular structure which tends to shrink on standing above the precipitation temperature. This phenomenon occurred in cases in which high molecular weight polymer was present at concentrations above three-tenths of 1%.

In studying both the viscosity and upper temperature limit of solubility in salt solution, reagent grade salts were used. Intrinsic viscosities have been reported in units of deciliters per gram.

### DISCUSSION OF RESULTS

In a previous study of the solution properties of poly(ethylene oxide) of high molecular weight,<sup>2</sup> it was found that the upper temperature limit of solubility appeared to be a function of both resin concentration and molecular weight. It is, therefore, appropriate to examine these dependencies in more detail. In Figure 2, the concentration dependence of the precipitation temperature of ethylene oxide polymer covering a wide range of molecular weight is shown. These polymers cover

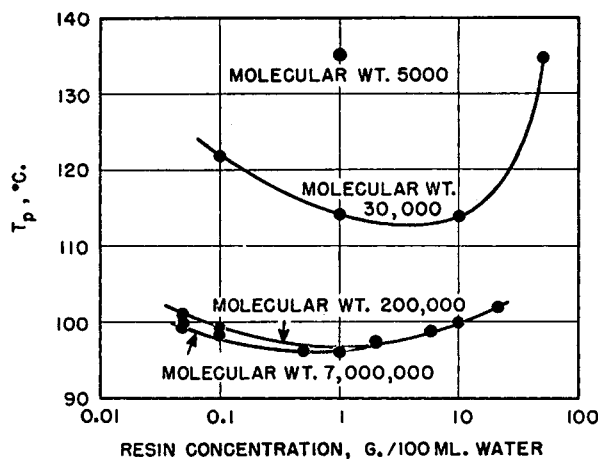


Fig. 2. Solubility-temperature relations of ethylene oxide polymers.

a range of three decades in molecular weight from about 5000 (Carbowax 6000) to more than 5 million (Polyox coagulant). Above a molecular weight of about 50,000 and a resin concentration of about 0.3 g./100 g. water the precipitation temperature becomes insensitive to both molecular weight and resin concentration. The remarkable insensitivity of the upper temperature limit of solubility above a critical molecular weight to variation in molecular weight is shown in Figure 3. The precipitation temperature of 1% solutions is shown as a function of the intrinsic viscosity of the poly(ethylene oxide) sample.

Inverse solubility-temperature relationships of other high polymer-solvent systems have been interpreted<sup>4</sup> on the basis of dual character either of the polymer structure or of the diluent. It has been considered that under these circumstances both solvophilic and solvophobic influences are reflected in the heat of dilution and entropy of dilution factors of the polymer-solvent interaction parameters. The temperature dependence is such that the diluent becomes a nonsolvent at a critical temperature. In the case of poly(ethylene oxide), the alternate structure along the polymer chain of ether oxygen and ethylene units offers a ready picture of hydrophilic and hydrophobic character. Evidence for the definite association of the ether oxygen units of poly(ethylene oxide) with water has been observed in the case of lower molecular weight polyoxyethyl chains by Rösch,<sup>5</sup> who interpreted viscosity data in terms of an association of two water molecules per ether oxygen.

If the interpretation based on hydrophilic-hydrophobic balance in the polymer structure is to be accepted as a basis for the inverse solubility-temperature relation, then an increase in the hydrophobic character of the resin should lead to a

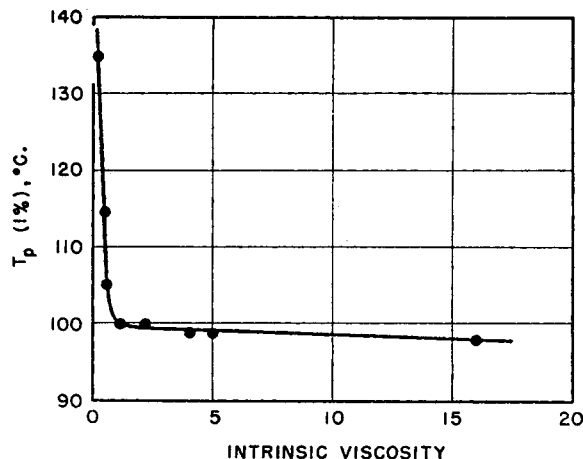


Fig. 3. Upper temperature limit of solubility versus intrinsic viscosity.

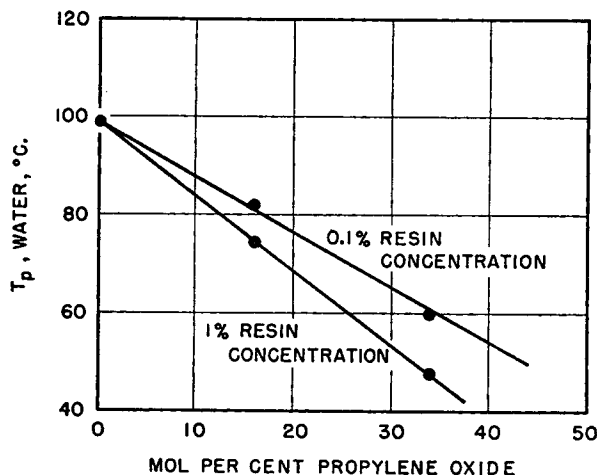


Fig. 4. Upper temperature limit of solubility of copolymers of ethylene oxide-propylene oxide.

lowering of the upper temperature limit of solubility in water. This prediction is tested in Figure 4 with respect to the solubility limits of copolymers of ethylene oxide and propylene oxide. For a given resin concentration in water, the upper temperature limit of solubility decreases linearly with increasing propylene oxide content of the copolymer, at least up to the rather high propylene oxide content thus far studied.

The phase separation of poly(ethylene oxide) from water solution can be considered as due to the increase of the activity of this neutral molecule as temperature is raised. The effect of neutral salts on poly(ethylene oxide) can be considered with respect to the effect of neutral salts in increasing the activity of neutral molecules in media such as water. The theoretical effect of neutral salts is expressed by the Debye-McAulay<sup>6</sup> equation for the change of the logarithm of the activity coefficient of a neutral molecule with salt concentration:

$$\ln f_n = \frac{\beta}{2kTD_0} \sum_i \frac{n_i e_i}{r_i}$$

in which  $\beta$  is a constant, characteristic of the nonelectrolyte, defined by the relation  $D = D_0 - \beta n$ ,  $D_0$  is the dielectric constant of the media (water),  $n$  is the number of molecules of nonelectrolyte, and  $D$  is the dielectric constant of a solution of the nonelectrolyte in water;  $n_i$ ,  $e_i$  and  $r_i$  are the number, charge, and ionic radii of the ions present, respectively. Thus, the upper temperature limit of solubility of poly(ethylene oxide) in water should be lowered by the presence of salts in a manner proportional to the concentration of salt and the valencies of the ions of the salt. Furthermore, small ions should be more

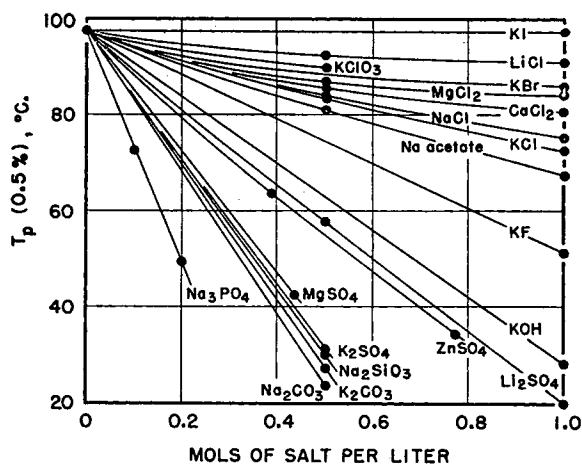


Fig. 5. Upper temperature limit of solubility versus salt concentration.

effective in salting out the polymer than large ions.

In Figure 5, the upper temperature limit of solubility of poly(ethylene oxide) of molecular weight four million<sup>7</sup> and at a concentration of  $1/2\%$  is shown as a function of salt concentration for a number of salts in water. It has been found that similar relations of salting out behavior hold over a wide molecular weight and concentration range. It is noted that the upper temperature limit of solubility decreases nearly linearly with salt concentration. In the case of the potassium halides, the effectiveness of the salt in lowering the precipitation temperature increases as the ionic radius of the anion decreases. The effectiveness of salts, however, does not follow the ionic strength principle as is clearly seen, for example, in the essentially equal salting out power of potassium sulfate and magnesium sulfate. Indeed, the

TABLE II  
Approximate Salting Out Effectiveness of Ions on an Ionic Strength Basis

For poly(ethylene oxide)	For small neutral solute molecules
Anions:	
OH <sup>-</sup>	OH <sup>-</sup>
F <sup>-</sup>	
CO <sub>3</sub> <sup>--</sup> SO <sub>4</sub> <sup>--</sup>	CO <sub>3</sub> <sup>--</sup> SO <sub>4</sub> <sup>--</sup>
Acetate	
Cl <sup>-</sup>	Cl <sup>-</sup> Acetate ClO <sub>3</sub> <sup>-</sup>
PO <sub>4</sub> <sup>---</sup>	
Br <sup>-</sup> ClO <sub>3</sub> <sup>-</sup>	Br <sup>-</sup> I <sup>-</sup>
Cations:	
K <sup>+</sup> Na <sup>+</sup>	K <sup>+</sup> Na <sup>+</sup>
Li <sup>+</sup>	
Ca <sup>++</sup> Mg <sup>++</sup>	Li <sup>+</sup> Ca <sup>++</sup> Mg <sup>++</sup> Zn <sup>++</sup>
Zn <sup>++</sup>	
H <sup>+</sup>	H <sup>+</sup>

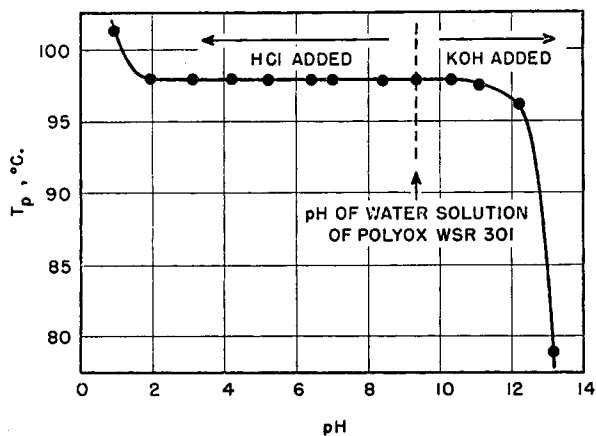


Fig. 6. Upper temperature limit of solubility versus pH.

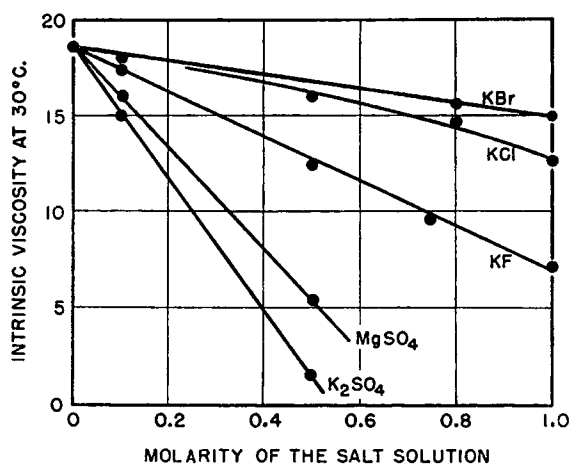


Fig. 7. Intrinsic viscosity in salt solutions.

particular anion appears to have a distinct effect. This departure from the salting out behavior predicted from theory is not unique to poly(ethylene oxide). In Table II a comparison is made between the relative salting out power of a number of ions, on an ionic strength basis, for poly(ethylene oxide) and the salting out power of these ions in the case of small neutral solute molecules in water.<sup>6,8</sup> The order of effectiveness of salting out of poly(ethylene oxide) from water is reminiscent of the classic "Hofmeister Series" for proteins.

While the specific salting out interactions of particular ions are beyond the scope of this discussion, the special effect of pH on the upper temperature limit of solubility must be noted. The lowering of the precipitation temperature is essentially a linear function of hydroxyl concentration. In Figure 6, the upper temperature limit of solubility is shown as a function of pH in water solution. At high hydroxyl ion concentration, the precipitation temperature is drastically reduced.

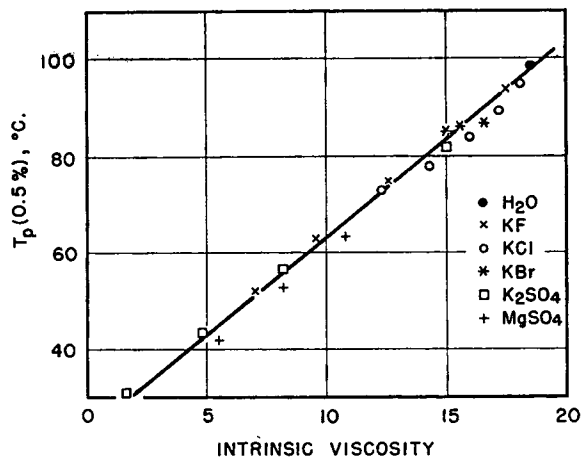


Fig. 8. Precipitation temperature versus intrinsic viscosity at 30°C. in salt solutions.

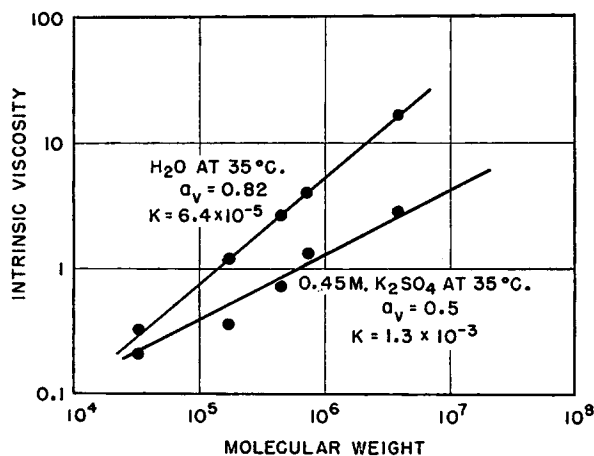


Fig. 9. Intrinsic viscosity-molecular weight relations at 35°C.

Conversely, higher hydrogen ion concentrations tend to raise the precipitation temperature. Imposed, therefore, on the thermodynamic salting out effect, there is a hydrogen bonding effect which is, among other interactions, strongly dependent upon pH.

These salting out effects are reflected in the collapsing of the polymer coil in solution. This changing of the polymer dimensions in solution can be observed in the reduction of the intrinsic viscosity of the polymer. To study this effect, intrinsic viscosities have been determined in water and in salt solutions by extrapolation both to infinite dilution of polymer and to zero shear rate.<sup>2</sup> In Figure 7, the intrinsic viscosity of a particular sample of poly(ethylene oxide)<sup>7</sup> is shown as a function of salt concentration for a number of salts. It is seen that these salts fall in the same order of effectiveness in reducing the ethylene oxide polymer coil dimensions in water, reflected

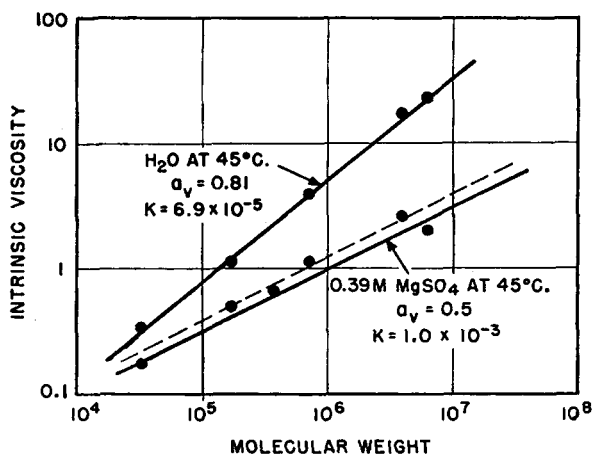


Fig. 10. Intrinsic viscosity-molecular weight relations at 45°C.

in lowering of the intrinsic viscosity, as they do in their effectiveness in lowering the upper temperature limit of solubility in water. The order of effectiveness shown holds in highly dilute solutions of salts (ca.  $10^{-2} M$ ). This correlation is seen in Figure 8 in which the upper temperature limit of solubility of this poly(ethylene oxide) in a particular salt solution is plotted as a function of the intrinsic viscosity of the polymer in the same salt solution at 30°C. Figure 8 is essentially a plot of the "theta point"<sup>10</sup> of this resin in an aqueous salt solution versus the intrinsic viscosity in the particular salt solution at 30°C.

To examine more closely the intrinsic viscosity-molecular weight relations of poly(ethylene oxide) in water and in salt solutions, a comparison is made in Figure 9 of the intrinsic viscosity at zero shear rate versus molecular weight relation of poly(ethylene oxide) over a very wide range of molecular weights in water and in 0.45  $M$  potassium sulfate solution. The latter solution, from data leading to Figures 5 and 8, was found to have a "theta point" close to 35°C.<sup>10</sup> The molecular weight values were determined by interpolation from intrinsic viscosity data and sedimentation and light scattering measurements of the molecular weight of poly(ethylene oxide) resins.<sup>9</sup>

The intrinsic viscosity-molecular weight relations are found to be:

$$[\eta] = 6.4 \times 10^{-5} M^{0.82} \text{ in water at } 35^\circ\text{C.}$$

$$[\eta] = 1.3 \times 10^{-3} M^{0.5} \text{ in } 0.45 M \text{ K}_2\text{SO}_4 \text{ at } 35^\circ\text{C.}$$

These data are shown in Figure 9.

In Figure 10, the intrinsic viscosity-molecular weight relation is shown in water and in 0.39  $M$   $\text{MgSO}_4$  at 45°C. The relations found at 45°C. are:

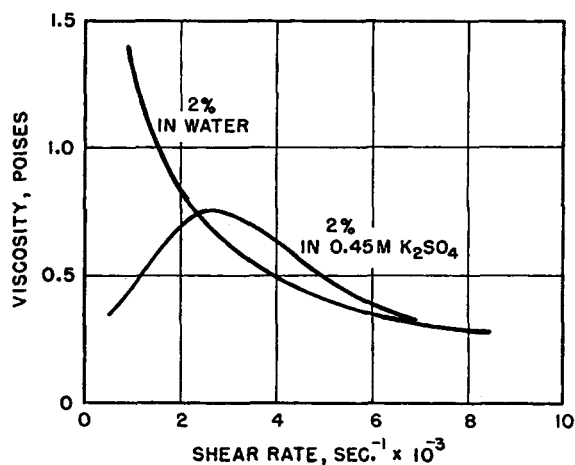


Fig. 11. Viscosity at high shear rates in water and in 0.45  $M$   $\text{K}_2\text{SO}_4$  at the  $\theta$  point.

$$[\eta] = 6.9 \times 10^{-5} M^{0.81} \text{ in water at } 45^\circ\text{C.}$$

$$[\eta] = 1 \times 10^{-3} M^{0.5} \text{ in } 0.39 M \text{ MgSO}_4 \text{ at } 45^\circ\text{C.}$$

The theta point in 0.39  $M$  magnesium sulfate solution was found to be close to 45°C. The dashed curve in Figure 10 represents the relation for potassium sulfate solution shown in Figure 9. The intrinsic viscosity was very sensitive to temperature change in the neighborhood of the theta point in salt solutions.

From the intrinsic viscosity relations in the "ideal water-salt solvent" in Figures 9 and 10, the unperturbed polymer coil dimensions can be calculated and compared with those obtained on the basis of a random coil model with free bond rotation. The result of such calculation of the root-mean-square end-to-end distance of the polymer coil leads to a value of 800–900 Å. on the basis of the intrinsic viscosity for a polymer of one million molecular weight in 0.45  $M$  potassium sulfate solution at 35°C. These coil dimensions were calculated from the viscosity equation taking the value of the "Universal Parameter,"  $\Phi$ , to be  $2.1 \times 10^{11}$ .<sup>10</sup> This end-to-end distance is about twice the value calculated on the basis of the free bond rotation model. Although the free rotation model would at first seem a good approximation for this polyether structure, the free rotation due to the ether linkages is apparently compensated by enhanced rigidity due to hydration in the salt solution so that the ratio of the viscosity determined dimension to that calculated on the free rotation model is similar to that found<sup>10</sup> for hydrocarbon polymers in which hindered rotation plays a considerable role in leading to expanded configurations. In water, at 35°C., the polymer coil is expanded and the root-mean-

square end-to-end distance from intrinsic viscosity data for poly(ethylene oxide) of one million molecular weight is calculated to be 1200–1300 Å.

In Figure 11, the effect of potassium sulfate on the structural viscosity of more concentrated solutions of high molecular weight (four million)<sup>7</sup> poly(ethylene oxide) is shown. The apparent viscosity of this poly(ethylene oxide) as a function of shear rate was determined using an automatic recording, rotational viscometer<sup>2,13</sup> for solutions both in water and in 0.45 *M* potassium sulfate near the "theta point" (34.5°C.). The effect of salts, such as potassium sulfate, in salting out poly(ethylene oxide) from water solution by the collapsing of the polymer coil is reflected in the reduction of the low shear-rate viscosity. The marked difference in the dependence of the solution viscosity in water and in the water-salt "theta solvent" as a function of shear rate is of special interest. This effect apparently becomes pronounced only at temperatures approaching the theta point.

The unique thickening action of high molecular weight poly(ethylene oxide) resins in water has been found to be associated with the highly pseudo-plastic character<sup>2</sup> shown in Figure 11 in the case of the solution in water. From the solution volumes of poly(ethylene oxide) calculated from intrinsic viscosities, the high degree of entanglement of polymer chains which must be present in the 2% solutions in water and in salt solution, described in Figure 11, is apparent. Exact interpretation of the structure development in the water-salt "theta solvent" under shearing force and the onset of pseudo-plasticity at higher shear rates must await further investigation. Tentatively, however, it is considered that the effect of the salt in collapsing the polymer coil near the "theta point" in these more concentrated solutions is to sharpen the peaks of the radial distribution function of polymer segments while preserving the basic overlapping of these distributions. The effect of shear, then, is to distend these collapsed coil configurations with a resultant increase in the structural viscosity until, at sufficient distension, the coil configuration approaches that developed under shear forces by the fully expanded coil in water solution.

#### CONCLUSION

Poly(ethylene oxide) is very soluble at room temperature in water and in certain organic solvents such as chloroform, acetonitrile, and acetic acid; but it is insoluble, for example, in anhydrous methanol except at temperatures considerably

above room temperature. This polymer, which is highly crystalline,<sup>3,11</sup> is soluble in water due to hydration of the ether oxygens in a manner which is apparently unique to the polyoxyethylene structure. This hydration, which must be a hydrogen bond interaction, is disrupted by thermal and salting out influences. The salt effects are, however, small in comparison with those observed in the case of polyelectrolytes.<sup>14</sup>

In comparison with all other water-soluble resins, the structure of poly(ethylene oxide), being both completely linear and as a polyether relatively non-polar, is among the least complicated. Yet, in water solution poly(ethylene oxide) exhibits a high degree of structural viscosity and this polymer may serve as a model for the interactions which take place in the case of more highly polar natural and synthetic polymers.

The high degree of shear dependence of the viscosity of high molecular weight poly(ethylene oxide) in water solution is in part due to the essential linearity of the structure together with the enormous degrees of polymerization which can be achieved. While a free rotation model for poly(ethylene oxide) might appear to be a good approximation in view of the added flexibility of the chain imparted by the ether linkages, in water solution these linkages are apparently "stiffened" by hydration so that the polymer coil extension approaches that found in hydrocarbon polymers in which hindered rotation leads to more extended configurations. The high degree of entanglement of polymer chains in more concentrated solutions (several per cent) appears to result in a net-work structure which is distended by relatively low shearing forces. Depending on the degree of entanglement, that is, depending on the polymer dimensions in solution and on concentration, the degree of distortion of the polymer configurations in solution tends to reach a limiting value at very high rates of shear.

Poly(ethylene oxide) resins are unique in a linearity of structure, non-ionic character, and water solubility. This linear, relatively nonpolar structure displays a high degree of polymer-solvent interaction in water solution which is observed in the development of structural viscosity to an unusual degree.

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### Synopsis

High polymers of ethylene oxide have been shown to have a number of unique properties in water solution. In this study, these properties have been investigated from the view of the behavior of large neutral molecules in media of high dielectric constant. Predictably, these high polymers of ethylene oxide undergo salting-out effects which are reflected in changes of solution viscosity with salt concentration. Salts vary in effectiveness in a manner similar to that observed in the case of small neutral solute molecules. The salting-out and viscosity effects are paralleled by the inverse solubility-temperature relationship in water. Above a minimum molecular weight of about 50,000, the precipitation temperature (the temperature at which polymer separates from solution as a separate phase as the temperature is raised) becomes essentially independent of molecular weight. For these very high polymers the upper temperature limit of solubility in water is independent of polymer concentration over a wide range of concentration. These effects are discussed in terms of the balance of hydrophilic and hydrophobic character in the polymer structure which can be altered by increasing the hydrocarbon content of the polymer through copolymerization of ethylene oxide with other olefin oxides such as propylene oxide. This investigation permits the development of an interpretation of the unusual rheology of high molecular weight poly(ethylene oxide) in water solution.

### Résumé

On a montré que les hauts polymères de l'oxyde d'éthylène ont un certain nombre de propriétés uniques en solution aqueuse. Dans cette étude, ces propriétés ont été étudiées du point de vue de comportement de grandes molécules

neutres en milieu de constante diélectrique élevée. On peut prévoir que ces hauts polymères de l'oxyde d'éthylène subissent des effets de sels qui se remarquent dans les changements de viscosité en solution avec les concentrations en sel. Les sels varient en efficacité d'une manière semblable à celle observée dans le cas de petites molécules neutres en solution. Les effets de sels et de viscosité sont mis en parallèle avec la relation inverse solubilité/température dans l'eau. Au-dessus d'un poids moléculaire minimum de 50.000, la température de précipitation (température à laquelle le polymère se sépare de la solution en une phase séparée si l'on augmente la température) devient essentiellement indépendante du poids moléculaire. Pour ces très hauts polymères la température limite supérieure de solubilité dans l'eau est indépendante de la concentration en polymère sur une grande échelle de concentration. Ces effets sont discutés en tenant compte du caractère hydrophile et hydrophobe dans la structure polymérique, caractère qui peut être changé, en augmentant l'élément hydrocarboné du polymère par copolymérisation de l'oxyde d'éthylène avec d'autres oxydes oléfiniques tels que l'oxyde de propylène. Cette recherche permet de développer une interprétation de la rhéologie inhabituelle de l'oxyde de polyéthylène de haut poids moléculaire en solution aqueuse.

### Zusammenfassung

Hochmolekulare Äthylenoxydpolymeren weisen in wässriger Lösung eine Anzahl einzigartiger Eigenschaften auf. In der vorliegenden Arbeit wurden diese Eigenschaften vom Gesichtspunkt des Verhaltens grosser, neutraler Moleküle in einem Medium hoher Dielektrizitätskonstante aus, untersucht. Wie vorauszusehen, treten bei diesen hochmolekularen Polymeren des Äthylenoxyds Aussalzeffekte auf, die sich in einer Abhängigkeit der Viskosität der Lösung von der Salzkonzentration merklich machen. Die Wirksamkeit der Salze variiert in einer Weise, die der bei Lösungen von kleinen neutralen Molekülen beobachteten ähnlich ist. Den Aussalz- und Viskositätseffekten geht die inverse Löslichkeits-Temperaturbeziehung in Wasser parallel. Oberhalb eines Mindestmolekulargewichts von etwa 50000 wird die Fällungstemperatur (die Temperatur bei der das Polymere bei Temperatursteigerung als getrennte Phase aus der Lösung ausgeschieden wird) im wesentlichen vom Molekulargewicht unabhängig. Für diese sehr hochmolekularen Polymeren ist die obere Temperaturgrenze für die Löslichkeit in Wasser über einen weiten Konzentrationsbereich von der Polymerkonzentration unabhängig. Die beobachteten Effekte werden auf Grund des Verhältnisses zwischen hydrophilen und hydrophoben Strukturelementen im Polymeren diskutiert; dieses Verhältnis kann durch Vergrösserung des Kohlenwasserstoffgehaltes des Polymeren durch Copolymerisation von Äthylenoxyd mit anderen Olefinoxyden, wie etwa Propylenoxyd, verändert werden. Die vorliegende Untersuchung erlaubt es, eine Erklärung der ungewöhnlichen Rheologie von hochmolekularen Poly(äthylenoxyden) in wässriger Lösung zu entwickeln.

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