The Effect of Solvents on High Molecular Weight, Stable Acetal Resins

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INTRODUCTION

The molecular characterization of a polymer is greatly facilitated by studies of the properties of the polymer in solution. Molecular weight measurements of high polymers, as determined by osmometry, light scattering, viscometry, and the ultracentrifuge techniques, are obtained by observing the properties of the polymer at low concentrations in solution.[1] A search for suitable solvents for high molecular weight polyoxymethylene has been made so that the polymer could be characterized.

At the other extreme of concentration, the absorption of solvents by the polymer can influence the properties of the polymer. This study was made with the objective of developing a method for predicting solvent-polymer interaction from the properties of the solvent and polyoxymethylenes.

EXPERIMENTAL

A. Polymer

The polyoxymethylene glycol used was prepared by the method described by MacDonald.[2] The polyoxymethylene diacetate was obtained by treating the glycol with acetic anhydride as reported by Punderson.[3] In those cases where standard tensile bars[4] and 5 in. $\times$ $1/2$ in. $\times$ $1/8$ in. bars were made, an antioxidant[5] was incorporated into the polymer. The mixture was then compacted by extrusion and cutting into cylindrical pellets. Finally, the bars were made by injection molding these pellets in a one-ounce Watson-Stillman injection molding machine at 200–220°C. into the appropriate mold.

B. Solvents

Purity of the solvents was largely an unknown factor. Solvents were used "as received" unless the material was obviously in poor condition.

C. Solubility Studies: Procedure

Ten milliliters of solvent was added to 0.1 gram (1% w/v) of the polyoxymethylene in a test tube and stirred continuously with a thermometer while heating. The temperature was noted when the polymer dissolved or melted or the solvent boiled. If the polymer dissolved, the solution was stirred continuously and allowed to cool. The first visual sign of haziness or solid formation was noted as the gel temperature. When the polymer dissolved but did not gel on cooling to room temperature, the solution was poured into 100 ml. of a nonsolvent (acetone or methanol). If a precipitate did not form, the solvent was reported as completely degrading.

D. Absorption Studies

Weighed, injection-molded test bars were placed in a bottle with enough solvent to cover the bars. The bottle was purged with nitrogen (to eliminate the explosion hazard), sealed, and placed in an air-circulating oven heated to 60 $\pm$ 5°C. After 3 months and again after 1 year, bars were removed, wiped with a cloth, and weighed. Measurements of length, before and after the soaking, were also made. The values for per cent gain in volume were calculated with the assumption that the volume gain was entirely solvent and that no interaction between solvent and polymer existed.

E. Density Measurements

The density of the polymer samples was measured by the water displacement method. This procedure gives values of density reliable to about ± 0.001, which is equivalent to a change in crystallinity of about 1%.

F. Molecular Weight

Molecular weights of all the samples of the acetal polymer used in these experiments were determined by measuring the melt viscosities of the samples and converting these data to number-average molecular weights as described by Koch and Lindvig.[6]

TABLE I. Solvents for High Molecular Weight Polyoxymethylenes at 1.0% Polymer Concentration
Solvents are listed according to increasing gel temperatures of the solutions

Compound	Dissolving temp., °C.	Gel temp., °C.	Compound	Dissolving temp., °C.	Gel temp., °C.
m-Chlorophenol	89	55	Acetic anhydride	—	120
Phenol	109	58	o-Methoxyaniline (Anisidine)	138	120
p-Chlorophenol	98	60	p-Methoxybenzaldehyde (Anis-		
o-Chlorophenol	—	70	aldehyde)	—	120
p-tert-Butylcatechol	—	70	3,4-Dimethoxybenzaldehyde		
2-Thenyl alcohol	139	80	(Veratraldehyde)	150	120
m-Cresol	—	83	o-Ethoxyaniline	—	120
1,1,5-Trihydroperfluoropentanol	138	84	Tetrahydrofurfuryl alcohol	—	120
m-Methoxyphenol	—	85	Diethyl malonate	150	120
3,4-Xylenol	128	88	Phenoxypropylene oxide	—	120
2-Bromo-4-tert-butylphenol	—	88	1,2-Glyceryl formal	142	120
α-Methylallylphenol	—	90	1,1-Di-(β-chloroethoxy)methane	149	122
o-(α-Phenylethyl)phenol	—	90	N-Methyl-N-phenyl formamide	145	122
2,6-Dichlorophenol	—	96 (m.p.)	Cyclohexanedione-1,4	160	122
o-Cyclohexylphenol	—	97	Cyanoethyl acetate	150	122
Furfuryl alcohol	—	99	Methyl o-hydroxybenzoate	—	122
1,1,7-Trihydroperfluoroheptanol	144	99	β-Naphthol	—	122 (m.p.)
p-tert-Butylphenol	—	99 (m.p.)	Benzonitrile	—	122
4-Ketodioxolane	—	100	8-Quinolinol	150	122
Acetylene tetrabromide	—	100	3-Methoxytetramethylene sulfone	145	122
α-Naphthol	—	100 (m.p.)	3,4-Dihydro-2H-pyran-2-carbox-		
Aniline	130	102	aldehyde	137	123
p-Bromoaniline	160	104	Acetylbutyrolactone	—	123
N-Formylmorpholine	—	105	p-Chlorobenzonitrile	—	123
Ethylene bromide	—	108	2-Acetothiophene	146	123
1,1-Diethoxyethane	—	110	o-Dichlorobenzene	—	124
o-Hydroxybenzyl alcohol	—	110	p-Chlorobenzaldehyde	—	124
p-Nitrofluorobenzene	—	110	Ethyl hydroxyacetate	—	124
Diphenylamine	—	110	Succinonitrile	—	124
Eugenol	—	110	Phenylacetonitrile	140	124
Malonitrile	130	110	Allyl glycidyl ether	—	124
2-Aminobenzenethiol	137	111	α-Pinene, carbonylated	140	125
Cyclopentanone	—	112	2,3-Dihydro-2-formyl-1,4-pyran	141	125
1,4-Cyclohexanedione	160	112	2-Methyl-3-ketobutanol	—	125
Furfuryl	130	112	m-Nitrochlorobenzene	—	125
γ-Butyrolactone	134	112	p-Methoxybenzyl alcohol	139	125
β-Chloroethyl acetate	132	114	Cinnamyl alcohol	150	125
p-Nitrochlorobenzene	—	114	Pentamethoxy butane	140	125
Dithiodiethanol	162	114	m-Dimethoxybenzene	150	125
o-Hydroxyacetophenone	—	114	Styrene oxide	146	125
o-Toluidine	132	115	p-Bromoacetophenone	—	126
N,N-Dimethylformamide	135	115	Methoxymethoxyethylmethoxy		
3-Chloro-4-methyltetramethylene sulfone 150		115	acetate	150	126
Methyl hydroxyacetate	137	115	Dimethyl oxalate	137	126
Dimethylthiodipropionic acid	150	115	β-Methoxypropionitrile	150	126
Acetonyl acetone	—	115	4-Nitromethoxybenzene	—	126
Benzaldehyde	—	115	N-Methylformamide	147	127
Pentachloroethane	140	117	Cyanoethylmorpholine	—	127
o-Hydroxybenzaldehyde	140	117	β-Hydroxypropionitrile	150	127
Ethylene carbonate	145	117	Phenyl isocyanate	148	128
Tetramethylene sulfone	150	117	Acetylcaprolactam	146	128
m-Phenylenediamine	155	118	Methoxyacetic anhydride	150	129
Acetophenone	—	118	Isopropylidene glycerol	145	130
Benzyl formate	140	118	Bromobenzene	150	130
Benzothiazole	—	119	Iodobenzene	—	130
Cyclohexanone	—	119	α,β-Dichlorophenylethane	142	130
1,4-Dicyano-2-butene	—	119	Cyanocyclohexane	147	130
Benzyl alcohol	132	119	Formamide	150	135

(table continued)

TABLE I (*continued*). Solvents for High Molecular Weight Polymethylenes at 1% Polymer Concentration
Solvents are listed according to increasing gel temperatures of the solutions

Compound	Dissolving temp., °C.	Gel temp., °C.	Compound	Dissolving temp., °C.	Gel temp., °C.
Methoxybenzene	—	130	1,1,1-Dimethoxycyanocyclo-		
Benzyl acetate	150	130	hexylmethane	149	140
4-Chloromethoxybenzene	—	130	*N*,*N*-Dimethylaniline	157	140
2,3-Butylene carbonate	149	130	*N*,*N*,*N'*,*N'*-Tetramethyldiglycol-		
Trimethylene oxide phenyl ether	—	130	amide	150	140
Ethyl phosphate	—	130	Terpineol	—	140
Quinoline	148	131	1,6-Hexanediol	—	140
N,*N*-Dimethylbenzamide	149	131	Cyclohexanol	150	140
Methyl *p*-hydroxybenzoate	—	131	Dicyclopentyl alcohol	150	140
β-Methoxyadiponitrile	153	131	*N*,*N*-Di-(β-cyanoethyl)-*N'*,*N'*-		
Dimethyl trioxymethylene ether	140	131	dimethyl urea	150	140
Isophorone	—	132	2-(*p*-Tolyl)-dioxolane	143	140
N-(2-Cyanoethyl)-methylform-			Fenchone	—	141
amide	154	132	Isofenchone	—	140
β-Methoxyethyl methoxyacetate	151	132	Isofenchyl alcohol	—	141
Ethyl acetoacetate	—	132	7-Bromo-1-cyanoheptane	149	141
Dimethylphthalate	—	132	Vinyl-2-thenoate	153	142
β,β'-Iminodipropionitrile	161	132	Methoxytriglycol	158	142
Di-2-mercaptoethyl ether	155	132	Diphenyl ether	160	142
Di-(β-cyanoethyl)ether	150	132	3,7-Dimethyl-2,6-octadien-1-ol		
α-Toluenethiol	153	133	(Geraniol)	—	142
1-Allyl-3,4-methylenedioxyben-			Allyl dimethyldithiocarbamate	158	142
zene (Safrole)	158	133	Isobutyl levulinate	153	142
1,3-Butylene carbonate	150	133	Methyl methacrylate adduct of		
Benzyl acetone	150	134	di-2-mercaptoethyl sulfide	170	142
Terpene B alcohol	—	134	Dimethoxytetraethylene glycol	162	142
Diethyleneglycol diacetate	155	134	Allyl phenoxypropene oxide	155	142
Diethyl oxalate	—	134	Diethyleneglycol dibenzoate	153	143
Methoxytetraglycol acetate	154	134	Diethylethoxymethylene malonate	160	143
Nitrobenzene	148	134	Propionic anhydride	155	144
1,1-Dimethoxyethoxymethane	155	135	2-Methoxymethyl-2,4-dimethyl		
Cyanoethylcyclohexanone	150	135	pentanediol-1,5	156	144
Benzil	—	135	*p*-*tert*-Amylphenoxy ethanol	153	144
2-Thenoyltrifluoroacetone	156	135	Dichlorocyclohexane	150	145
Benzophenone	—	135	Benzyl benzoate	160	145
N-Ethylformamide	—	135	1,3-Propanediol	161	145
5-Nitro-4,4-dimethylpentanone-2	155	136	Diethyl itaconate	153	146
6-Butyl hydroxyacetate	145	136	(β-Cyanoethyl)sulfide	—	147
ε-Caprolactam	145	136	Triethyl aconitate	155	148
Diphenyl carbonate	155	136	Methoxypolyethylene glycol	170	148
Trichlorobenzene	—	137	Triglycol formal	157	148
β-Bromostyrene	147	137	Phenyl isothiocyanate	166	148
Glycerol triacetate	151	137	3,5-Dimethylmethoxybenzene	159	150
2-Butene-1,4-diacetate	150	137	Tetracarboethoxypropane	166	150
Dimethyl adipate	156	137	Butylcarbitol diglycollate	163	151
β-Methoxyethyl-*N*-ethyl car-			Trimethallyl carballylate	163	153
bamate	154	137	Di-2-mercaptoethyl sulfide	165	153
Adiponitrile	152	137	Nonaethylene glycol	167	154
Trichlorobenzene	—	137	Butyl acetonylphthalate	155	154
5,5,5-Trichloropentane-1-thiol	175	137	2,5-Hexanediol	—	155
Bis-(carbomethoxyethyl)-sulfone	145	137	Dimethallyl carbonate	160	155
β,β'-Oxydipropionitrile	153	137	Diethylene glycol bis-(*n*-butyl		
1,1-Di-(β-hydroxyethoxy)methane	160	150	carbonate)	174	156
Fenchyl alcohol	—	138	Diethoxydiethyl adipate	158	156
Biacetylcyanohydrin diacetate	150	138	3,5-Dimethoxy-1-hexanol	160	157
Di-β-hydroxyethyl glutarate	—	138	Arochlor-1242	—	160
1,2-Bis-(2-cyanoethoxy)ethane	152	138	Butyl benzyl phthalate	178	174
N-Methoxymethylacetamide	157	139	Butyl carbitol glutarate adipate	181	178
Diethyl fumarate	—	139	2-Thenylethyl xanthate	188	183

RESULTS AND DISCUSSION

A. Polymer in Solvents

No solvent for high molecular weight (>40,000) oxymethylene polymer at room temperature has been found. Of the 406 compounds, representing 27 different classes of compounds, tried as solvents at the 1% polymer concentration level, none produced a solution at temperatures less than 50°C. At elevated temperatures, two effects were observed: in some cases solvation occurred, and in others degradation occurred. A list of the solvents for the polymer, showing the dissolving temperature and the gel (reprecipitation) temperature at 1% concentration is shown in Table I.

Phenolic compounds were found to have the greatest solvent power; i.e., lowest gel temperature, for the oxymethylene polymer. Although many phenols gave solutions with gel temperature in the range of 50° to 100°C., the actual dissolving temperatures were usually 40° to 60° higher. Generally, the temperature at which the acetal polymer became soluble was 10° to 40° higher than its gel temperature. Barkdall and MacDonald[7] have shown that the molecular weight of the polymer is not altered in solutions of m-chlorophenol, p-chlorophenol, or benzyl alcohol and have reported gel temperatures at a 2% concentration of polyoxymethylene. A great many solvents were found that were effective in the range of 100 to 150°C. Most of the classes of compounds tested had at least one member which was a solvent in this range. In no case was a mixture of solvents found to be more effective than the best of the individual components. Compounds having structures similar to that of the polymer, such as ethers, Cellosolve, Carbitol, acetals, etc., were generally poor solvents. Strongly acidic and strongly basic substances degraded the polymer.

B. Solvent Absorption

Most polymers absorb solvents to varying amounts, and this sorption generally results in changes in weight, dimensions, and mechanical properties. Solvent absorption studies were undertaken to develop methods for predicting the solvent-polymer interaction from the properties of the solvent and polymer.

Injection molded test bars, 5 in. × $\frac{1}{2}$ in. × $\frac{1}{8}$ in., and tensile bars were exposed to solvents at 60°C. for periods varying from 3 months to one year. At the end of this time, the bars were assumed to be at equilibrium. Studies on the rate of absorption showed that at this temperature equilibrium was usually established within 10 days. Table II summarizes the results of these tests.

These data show a direct relation between the amount of solvent sorbed on the polymer and the change in properties. These same data are shown graphically in Figures 1-3. Obviously a relationship exists between the swelling; i.e., per cent

TABLE II
The Effect of Equilibrium Solvent Absorption at 60°C. on the Properties of Oxymethylene Polymers[a]

Solvent	Maximum weight gain, %	Gain in length, %	Tensile modulus[b]	Tensile strength, psi[b]	Elongation, %[b]
Aniline	8.27		150,000	9,065	33.7
Dioxane	7.15	3.09	203,500	9,405	30.2
Pyridine	6.12	2.89	213,500	9,480	30.3
Carbon tetrachloride	5.70			9,600	
Perclene	5.58	1.57	290,000	9,840	24.6
Benzene	4.17	2.18	254,500	9,575	24.8
Ethyl acetate	3.80			9,510	
Butyraldehyde	3.79	2.02	241,000	9,535	29.5
Acetone	3.30			9,610	
Toluene	2.80			9,480	
Methanol	2.61	1.84	264,000	9,600	23.8
Ethanol	2.10			9,520	
Water	1.54	0.52	369,500	10,515	23.8
Hexane	0.74	0.63	410,500	10,135	20.7
Triethylamine	0	0	397,500	10,200	18.9
Control	0	0	450,000	10,200	15

[a] Physical properties were measured at room temperature.
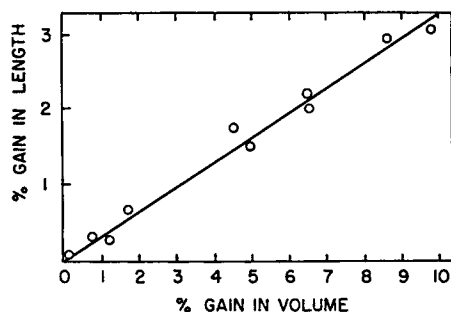[b] ASTM D-638.

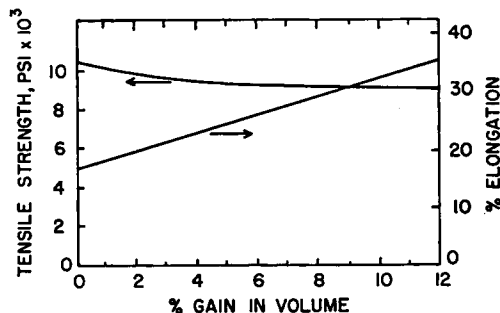Fig. 1. Dimensional changes in swollen bars of polyoxymethylene diacetate.

Fig. 2. Tensile strength and elongation of swollen polyoxymethylene tensile bars.
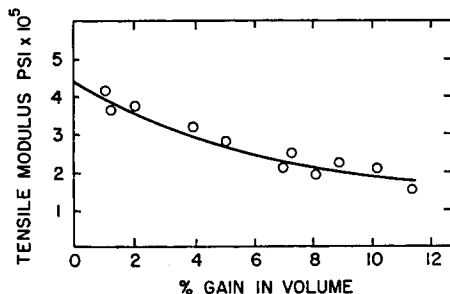

Fig. 3. Tensile modulus of swollen polyoxymethylene bars.

where ΔH is the molar heat of vaporization at temperature T, R is the thermodynamic gas constant, and V is the molar volume of solvent at temperature T. For solvents, this can be calculated easily from the heat of vaporization and density of the material. Table III lists the CED's

TABLE III
Cohesive Energy Densities of Organic Compounds
Evaluated from Vapor Pressure Data

Compound	Temperature °C.	CED, cal./cc.
Diethyl ether	25	59.3
n-Butyl acetate	25	65.9
Isopropyl acetate	25	67.9
n-Amyl acetate	25	69.8
Ethyl chloride	25	76.3
Chloral	25	81.5
Methyl ethyl ketone	25	82.0
Chloroform	25	86.0
Methyl isobutyl ketone	25	90.2
Tetrachloroethane	25	91.2
Methyl acetate	25	91.7
Ethylene dichloride	25	97.8
Ethylene dibromide	25	100
Methylene chloride	25	103
n-Butanol	25	125
n-Propanol	25	147
Acetic acid	25	159
Hexane	60	52.6
Triethylamine	60	53.9
Carbon tetrachloride	60	67.9
Perclene	60	75.0
Toluene	60	79.1
Butyraldehyde	60	81.8
Benzene	60	83.4
Ethyl acetate	60	85.0
Acetone	60	88.8
Isoamyl alcohol	60	91.8
Pyridine	60	98.3
Dioxane	60	100
m-Cresol	60	130
Aniline	60	142
Dimethylformamide	60	146
Ethanol	60	163
Methanol	60	192
Water	60	552

change in volume, and the mechanical properties of the polymer.

A method of predicting the swelling of a polymer in various solvents has been reported by Scatchard,[8] Walker,[9] and Gee.[10] Essentially, the theory is based on the observation that like dissolves like. In this case, features of solute and solvent that must be similar are the cohesive energy densities (CED). The theory has been applied successfully to the swelling of rubber.

The cohesive energy density of a substance is the amount of heat required to vaporize a unit volume of the substance, given by

$$CED = \frac{\Delta H - RT}{V}$$

of several organic compounds calculated from such data. Hildebrand[11] also gives several methods for the direct estimation of the CED of a compound. For polyoxymethylenes, the cohesive energy density can be estimated from group contributions as determined by Dunkel.[12] On the basis of the Dunkel value of 2620 cal./g.-mole for the —CH_2O— unit, 30 for the molecular weight of the unit, and 1.42 g./cc. for the density, the CED for polyoxymethylene is
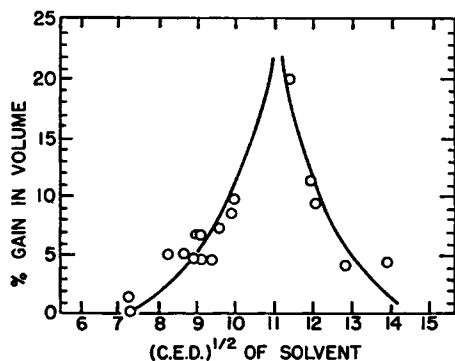
Fig. 4. Equilibrium swelling of polyoxy-
methylene bars as a function of the cohesive
energy density of the solvent.

Fig. 5. Relationship between swelling equilib-
rium and the density of polyoxymethylene.

$$CED = \frac{(2620 \text{ cal./g.-mole})(1.42 \text{ g./cc.})}{30 \text{ g./g.-mole}}$$

$$= 124 \text{ cal./cc.}$$

The error involved in ignoring the end groups on a polymer of greater than 40,000 molecular weight is negligible, probably less than 0.5%.

A graphical presentation of the swelling of polyoxymethylenes at 60°C. as a function of the square root of the CED of the solvent is shown in Figure 4. This shows the maximum swelling in a solvent whose CED is nearly the same as that of the polymer. As the CED of the solvent becomes progressively different from that of the polymer, the amount of swelling decreases. Water is outside the limits of the plot, since it has a $(CED)^{1/2}$ of 23.5. Water also gives low swelling, about 2%.

Undoubtedly the amorphous content of the polymer contributed to the amount of absorption of solvent. To show this, bars were prepared under conditions of both rapid cooling of the polymer melt and, in some cases, annealed at 160°C. to alter the crystallinity. Density measurements of these bar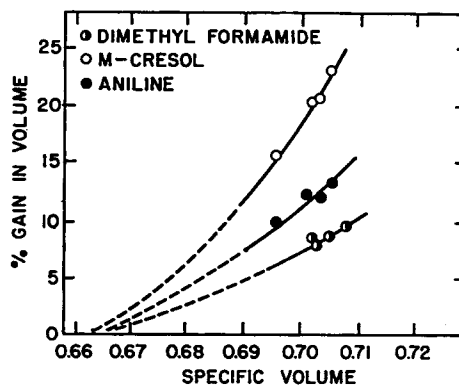s indicated the amount of crystalline and amorphous phases in the polymer. The data given in Table IV and Figure 5 show the dependence of sorption and swelling on the amorphous content of the polyoxymethylene polymer in three solvents. These data show two important features of the sorption of solvents by polyoxymethylenes.

1. As the density of the polymer increases (increasing crystallinity and decreasing amorphous content), the amount of solvent absorbed and the amount of swelling decreases. This indicates that the solvent is absorbed mostly in the amorphous region of the polymer.

2. A decrease in the amorphous content of the polymer gives a proportional decrease in the amount of solvent absorbed. In the sorption of aniline, a decrease in amorphous content of the polymer of 27%, i.e., from 35.0 to 25.5% (Table III), resulted in a decrease in sorption of 28.2% (from 9.60% to 6.90%). This shows that the crystalline portion of the polymer is relatively unaffected by the sorbed solvent. More evidence for this conclusion is obtained by extrapolating the curves shown in Figure 5 to the region of no absorption of solvents. The graph indicates that polyoxymethylene having

TABLE IV

Swelling Equilibrium Data for Polyoxymethylene Diacetate in Aniline,
Dimethylformamide and *m*-Cresol at 60°C.

Aniline				Dimethylformamide				*m*-Cresol			
Density of polymer, g./cc.	Amorphous content, %	Sorbed at equilibrium, wt.-%	Gain in vol., %	Density of polymer, g./cc.	Amorphous content, %	Sorbed at equilibrium, wt.-%	Gain in vol., %	Density of polymer, g./cc.	Amorphous content, %	Sorbed at equilibrium, wt.-%	Gain in vol., %
.418	35.0	9.60	13.7	1.413	36.0	6.35	9.5	1.418	35.0	16.48	23.0
1.422	33.0	8.66	12.1	1.419	34.0	5.73	8.6	1.422	33.0	14.56	20.6
1.427	30.5	8.71	12.3	1.425	32.0	5.62	8.5	1.425	32.0	14.60	20.4
1.438	25.5	6.90	9.8					1.440	25.0	10.50	15.7

a specific volume of about 0.664, or a density of about 1.51, should not absorb any solvent. A density of 1.51 is very close to the value of 1.506 calculated for an oxymethylene polymer of 100% crystallinity.[13]

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Synopsis

High molecular weight polyoxymethylenes are not soluble at room temperature in any of the hundreds of solvents or solvent systems tested. However, solutions can be obtained at elevated temperatures with a wide variety of solvents. Phenolic compounds are the most effective at temperatures of 50–100°C. At the other extreme of solubility, solvents are absorbed by the acetal polymer in varying amounts, depending on the crystallinity of the polymer and the cohesive energy density of the solvent. The absorption is concentrated entirely in the noncrystalline region of the polymer.

Résumé

Les polyoxyméthylènes de poids moléculaire élevé ne sont pas solubles à température de chambre, bien qu'une centaine de solvants et systèmes de solvants aient été essayés. Toutefois, des solution peuvent être obtenues à températures élevées avec une grande variété de solvants. Les dérivés phénoliques sont les plus efficaces aux températures de 50–100°C. Par ailleurs, les solvants sont absorbés par le polymère acétalique en quantités variables suivant la cristallinité du polymère et la densité d'énergie de cohésion du solvant. L'absorption est entièrement concentrée dans la région non-cristalline du polymère.

Zusammenfassung

Hochmolekulare Polyoxymethylene sind bei Raumtemperatur in keinem der nach hunderten zählenden, untersuchten Lösungsmittel oder Lösungsmittelsystemen löslich. Es können jedoch bei erhöhten Temperaturen mit einer grossen Vielfalt von Lösungsmitteln Lösungen erhalten werden. Phenolische Verbindungen sind bei Temperaturen von 50–100°C die wirksamsten Lösungsmittel. Das andere Löslichkeitsextrem bilden Lösungsmittel, die von dem Acetalpolymeren je nach seiner Kristallinität und je nach der Kohäsionsenergiedichte des Lösungsmittels in verschiedenem Ausmass absorbiert werden. Die Absorption spielt sich ausschliesslich im nichtkristallinen Bereich des Polymeren ab.