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REVIEW PAPER

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GLASS TRANSITION TEMPERATURES AND SOME PROPERTIES OF POLY(ITACONIC ACID) DIESTERS

Different homologous series of poly(itaconic acid) diesters have been synthesized and characterized in our laboratory: n–alkyl–, n–alkylphenyl–, n–alkylcyclohexyl–, tolyl– and xylenyl itaconates. Experimentally determined data for solubility parameters, steric hindrance to free rotation, glass transition temperatures and densities were analysed in order to relate the influence of the gradual change of chemical structure on material properties of the members of the families. The glass transition temperature dependance on the parameter derived from molecular structure –mass per flexible bond of monomeric unit have been analyzed as well. Available data for similar polymeric series –poly(acrylates) and poly(methacrylates) were also taken into consideration.

Itaconic acid and its derivatives are very interesting in polymer chemistry because a great variety of different polymers can be synthesized starting from a material which is not of petrochemical origin. Among other derivatives, different homologous series of poly (itaconic acid diesters) have been synthesized and characterized in our laboratory [1–5].

Important parameters sucg as the glass transition temperature (Tg), polymer solubility parameter (δ), polymer density (d) and the value of steric hindrance to free rotation (σ), were determined experimentally for all the polymer samples obtained. It was, therefore, of interest to analyse the change of these parameters with the chemical structure of the polymers, and to correlate some of these parameters to enable the prediction of the corresponding values for the new polymers. The experimentally determined glass transition temperature values of different itaconic diesters were correlated with the calculated parameter of the mass per flexible bond of monomeric unit (w/γ), introduced by Schneider and Di Marzio [6]. The same dependences were established for polyacrylates and methacrylates, polymers of similar structure, for comparison.

Similar analyses for various polymers are numerous in the literature. Lee and Sewell [7] presented a study of the influence of cohesive forces, expressed as solubility parameter values, on Tg. They found that there is a linear dependence between Tg and cohesive energy density (CED = δ) only for polymers with Tg < 25°C.

The dependence of Tg versus the chain flexibility has been examined by Privalko and Lipatov [8] using the σ parameter. Their analysis of the literature data for 32 polymers resulted in four linear dependences, originating from the same point, corresponding to polyethylene. Since σ for polyethylene is 1.63, they assigned the Tg value of PE to be 160 K.

One of the most extensive studies was undertaken by Bicerano [9]. He collected a great deal of experimental data for a great number of polymers, and compared them with the calculated values obtained from correlations based on so called topological techniques. His calculations cover a large number of fundamental properties (van der Waals volume, cohesive energy, heat capacity, molar refraction...), as well as of derived properties (Tg, density, solubility parameter...).

EXPERIMENTAL

All details concerning the synthesis of monomers, by esterification of itaconic acid with the corresponding alcohols, and polymers, polymerized in bulk at 60°C with AIBN as the initiator, were published earlier [1–5]. The glass transition temperatures were measured applying the DSC method, using a Perkin Elmer DSC–2 thermoanalyser with a heating rate of 20 K/min.

The solubility parameters values were obtained applying the viscosimetric method, and the results were compared with calculated values obtained by group contribution methods [10–13]. Polymer densities were determined by the method described by Huglin [14] and by floatation experiments of polymer particles in fluid mixtures of known density at 20°C.

All the investigated polymers were fractionated on a column of the Backer Williams type. The intrinsic viscosity values, measured in toluene at 25°C in an Ubbelohde viscometer, and the molecular mass values for the corresponding fractions, calculated from light scattering data in n–butanone measured on a Brice–Phoenix–2000 instrument, were used to calculate Mark Houwink relations, unperturbed dimensions and σ values [1–5].

RESULTS AND DISCUSSION

The experimentally determined and the calculated parameters taken into consideration are :

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– solubility parameters (δ), cohesive energy densities (CED) and cohesive energies (Ecoh),

– steric hindrance parameters (σ) and characteristic ratios (C∞), and

– specific mass per flexible bond of monomeric unit (w/γ).

THE SOLUBILITY PARAMETERS, COHESIVE ENERGY DENSITIES AND COHESIVE ENERGIES

The solubility parameter is a measure of the strength of physical links between the structural units of a material. It is used for the characterisation of amorphous polymers and for the amorphous phases in semicrystalline polymers, especially for the estimation of the polymer solubility and the compatibility of different polymers in polymer blends [15]. The cohesive energy (Ecoh) of a material is the increase of the internal energy per mole of the material if all of its intermolecular forces are eliminated [16,17].

The cohesive energy density (CED), defined by equation (1), is the energy required to break all intermolecular physical links in a unit volume of the material. In polymers these cohesive forces are mainly interchain interactions of various types, holding different components together.

The relevant parameters are conected by the following equation:

$$\delta \equiv \sqrt{CED} = \sqrt{Ecoh}/V = \sqrt{Ecoh.M}/d \qquad (1)$$

where – V is the molar volume of the polymer, (cm^3/mol), M is the molar mass of the repeat unit, (g/mol) and d is the density of the polymer, (g/cm^3).

The data of experimentally determined (solubility parameters and densities) and the calculated values of CED, Ecoh and V for several homologous series of itaconate diester polymers are presented in Tables 1 – 5.

All solubility parameters were determined from limiting viscosity numbers in various organic solvents and solvent mixtures, differing in structure, polarity and hydrogen bonding capacity and are close to the values calculated from the polymer densities and molar attraction constants.

Numerical values for poly(d–n– alkyl itaconates), given in Table 1, decrease sharply going from the first to the second member of the series: from 19.6 (PDMI) to 18.5 (PDEI), and after that approach asymptotically a limit close to 16 $(J/cm^3)^{1/2}$, which can be attributed to the monotonously increasing paraffinic chain in the ester substituent. Phenyl groups in the substituents (Table 2) lead to slightly higher values than cyclohexyl rings (Table 3) and the gradually increasing number of methylene groups in both series results in a progresive decrease of the solubility parameter, in a similar manner as for the poly(di–n–alkyl itaconate) series. The solubility parameters for poly(di–tolyl itaconates) (Table 4) do not depend on the position of the methyl group, probably due to equally strong solvation of the substituents with

Table 1. Values of δ, d, CED, Ecoh and V of poly(di–n– alkyl itaconates).

Polymer	Tg(K)	δ $(J/cm^3)^{1/2}$	CED (J/cm^3)	Ecoh (J/mol)	V (cm^3/mol)	d (g/cm^3)
Poly(di-methyl itaconate) PDMI	368	19.6	383.4	46311	120.8	1.3082
Poly(di-ethyl–) PDEI	331	18.5	342.2	52918	154.6	1.2027
Poly(di-propyl–) PDPI	307	18.3	334.7	62357	186.3	1.1485
Poly(di-butyl–) PDBI	285	18.2	330.9	74803	226.0	1.0706
Poly(di-amyl–) PDAI	278	17.7	312.6	80038	256.5	1.0525
Poly(di-hexyl–) PDHxI	255	17.3	299.7	91575	286.6	1.0398
Poly(di-heptyl–) PDHpI	188	16.9	284.4	99668	–	1.0123
Poly(di-octyl– PDOI	191	16.8	280.9	108293	–	0.9977
Poly(di-nonyl– PDNI	197	16.7	278.9	114468	–	0.9837
Poly(di-decyl–) PDDI	210	16.6	274.1	–	–	0.9818
Poly(di-dodecyl–) PDdDI	225	–	–	–	–	0.9792

Table 2. Values of δ, d, CED, Ecoh and V of poly(di–n–alkyl-phenyl itaconates).

Polymer	Tg(K)	δ $(J/cm^3)^{1/2}$	CED (J/cm^3)	Ecoh (J/mol)	V (cm^3/mol)	d (g/cm^3)
Poly(di-phenyl itaconate) PDPhI	406	19.1	364.8	82965	227.4	1.240
Poly(di-methylphenyl–) PDMPhI	328	18.6	346.0	88635	256.2	1.210
Poly(di-ethyl-phenyl–) PDEPhI	308	18.4	338.6	96486	285.0	1.186
Poly(di-propyl-phenyl–) PDPPhI	265	18.2	331.2	104963	316.9	1.155

Table 3. Values of δ, d, CED, Ecoh and V of poly(di–n– alkylcyclohexyl itaconates).

Polymer	Tg(K)	δ $(J/cm^3)^{1/2}$	CED (J/cm^3)	Ecoh (J/mol)	V (cm^3/mol)	d (g/cm^3)
Poly(di-cyclohexyl itaconate) PDCHI	417	19.0	361.0	91730	254.1	1.157
Poly(di-methylcyclo–hexyl) PDMCHI	356	18.6	345,9	98933	285.9	1.126
Poly(di-ethylcy-clo–hexyl) PDECHI	321	17.2	295.8	94162	318.2	1.100
Poly(di-propylcyc-lo–hexyl) PDPCHI	314	16.6	275.4	96895	352.6	1.075

Table 4. Values of δ, d, CED, Ecoh and V of poly(di–tolyl itaconates).

Polymer	Tg(K)	δ $(J/cm^3)^{1/2}$	CED (J/cm^3)	Ecoh (J/mol)	V (cm^3/mol)	d (g/cm^3)
Poly(di-o-tolylitaconate) PDoTI	403	18.6	346	95125	274.3	1.130
Poly(di–m–tolylitaconate) PDmTI	381	18.6	346	113488	328.0	0.945
Poly(di–p–tolylitaconate) PDpTI	409	18.7	350	90866	295.8	1.193

solvent molecules. The same was concluded for poly (di–tolyl methacrylates) [12].

Poly(di–xylenyl itaconates) (Table 5) have slightly lower solubility parameters than poly(di–tolyl itaconates), but again the position of the two methyl substituents does not effect the δ values.

Table 5. Values of δ, d, CED, Ecoh and V of poly(di xylenyl itaconates).

Polymer	Tg(K)	δ $(J/cm^3)^{1/2}$	CED (J/cm^3)	Ecoh (J/mol)	V (cm^3/mol)	d (g/cm^3)
Poly(di 2,4xylenyl-itaconate) PD2,4XI	414	18.4	338.5	102631	303.1	1.115
Poly(di 3,4xylenyli-taconate) PD3,4XI	399	18.5	342.2	101295	296.0	1.142
Poly(di 3,5xylenyl-itaconate) PD3,5XI	414	18.5	342.2	103359	302.0	1.119

It is interesting to note that all the determined solubility parameter values change in a rather narrow range from 19.6 to 16.6 $(J/cm^3)^{1/2}$, and that there is no substantial change in δ with the side group in the polymer going from n–alkyl– to n–alkylphenyl –, n–alkylcyclohexyl–, tolyl– and xylenyl– itaconic diesters. The most noticeable change of the solubility parameter in the itaconic diester families occurs when the first methylene group is introduced in the side chain, as in the case of poly(di–n–alkyl itaconates), poly(di–n–alkylcyclohexyl itaconates) and the same trend is less pronounced in the case of poly(di–n–alkylphenyl itaconates).

In the case of poly(di–n–alkyl itaconates), poly(di–n–alkylphenyl itaconates) and poly(di–n–alkylcyclohexyl itaconates), the solubility parameters decrease with decreasing Tg values.

THE STERIC HINDRANCE PARAMETERS AND CHARACTERISTIC RATIOS

Conformational properties of the polymer chains were determined in dilute polymer solutions. The simplest parameter determining the conformations of a polymer chain in the "unperturbed state" is the steric hindrance parameter, extrapolated to give a measure of the polymer conformations at θ conditions. The steric hindrance parameter is presented by the following relation:

$$\sigma = (\bar{r}_0^2)^{1/2} / (\bar{r}_{0f}^2)^{1/2} \quad (2)$$

where $(\bar{r}_0^2)^{1/2}$ is the mean square end–to–end distance of a real chain in the unperturbed state, and $(\bar{r}_{0f}^2)^{1/2}$ is the same quantity for a freely rotating chain. It is assumed that the conformations of polymer chains in solution under θ–conditions are essentially the same as the random coil conformations of chains in glassy polymers [18,19].

The characteristic ratio C∞ is defined by the relation:

$$C_\infty = (K_\theta/\phi)^{3/2}\, m/l^2 \quad (3)$$

where K_θ is the Kuhn–Mark–Houwink–Sakurada constant at θ–conditions, φ the universal Flory constant, equal to 2.7 10^{23} mol^{-1}, l is the length of the carbon–to–carbon valence bond and m is the average molecular weight per unit skeletal link of the backbone chain. C∞ is proportional to σ^2.

The data for Tg, steric hindrance parameter, chatacteristic ratio and mass per flexible bond of monomeric unit for poly(di–n–alkyl itaconates) are listed in Tables 6–9.

The hindrance to free rotation, expressed as σ or C∞, for poly(di–itaconates) rises constantly, except in the case of poly(di–n–cyclohexyl itaconates), with increasing length of the side chain attached to the

Table 6. Values for Tg, steric hindrance parameter σ, chatacteristic ratio C∞, monomer molecular mass M_0 and mass per flexible bond of monomeric unit w/γ of poly(di–n–alkyl itaconates).

Polymer	Tg (K)	σ	C∞	M_0 (g/mol)	w/γ
PDMI	368	2.02	7.76	158	31.6
PDEI	331	1.98	7.82	186	26.6
PDPI	307	2.27	10.3	214	23.8
PDBI	285	2.60	13.1	242	22.0
PDAI	278	–	–	270	20.8
PDHxI	255	2.80	16,2	298	19.9
PDHpI	188	3.0	–	326	19.2
PDOI	191	3.02	18.3	354	18.6
PDNI	197	3.20	–	382	18.2
PDDI	210	3.30	21.9	410	17.8
PDC12I	225	3.58	24.5	466	17.2
PDC14I	(183)*	3.70	27.4	522	16.8
PDC16I	(186)*	3.89	30.3	578	16.5
PDC18I	(195)*	3.96	31.4	634	16.2
PDC20I	(212)*	3.94	31.1	690	16.0

* Tg values extrapolated from the Tg –density relation for n–alkyl itaconates [20].

Table 7. Values for Tg, steric hindrance parameter σ, chatacteristic ratio C∞, monomer molecular mass M_0 and mass per flexible bond of monomeric unit w/γ of poly(di–n–alkylphenyl itaconates).

Polymer	Tg (K)	σ	C∞	M_0 (g/mol)	w/γ
PDPhI	406	2.60	13.1	282	56.4
PDMPhI	328	2.69	14.6	310	44.3
PDEPhI	308	2.72	14.8	338	37.5
PDPPhI	265	2.78	15.6	366	33.3

Table 8. Values for Tg, steric hindrance parameter σ, chatacteristic ratio C∞, monomer molecular mass M_0 and mass per flexible bond of monomeric unit w/γ of poly(di–n–cyclohexyl itaconates).

Polymer	Tg (K)	σ	C∞	M_0 (g/mol)	w/γ
PDCHI	417	2.99	16.81	294	58.8
PDMCHI	356	2.75	15.13	322	46.0
PDECHI	321	2.83	16.06	350	38.9
PDPCHI	314	2.84	16.22	378	34.4

backbone chain. This is generally the case for series of vinyl polymers [21].

Rather high σ values, ranging from 3.00 to 3.99, obtained for poly(di–n–alkyl itaconates) (Table 6) with 8 to 20 methylene groups in the side chain are one to two

Table 9. Values for Tg, steric hindrance parameter σ, chatacteristic ratio C∞, monomer molecular mass M_0 and mass per flexible bond of monomeric unit w/γ of poly(di-tolyl itaconates).

Polymer	Tg (K)	σ	C∞	M_0 (g/mol)	w/γ
PDoTI	403	2.70	14.9	310	44.3
PDmTI	381	2.65	14.2	310	44.3
PDpTI	409	2.80	15.8	310	44.3

units higher than those usually observed for vinyl polymers [21]. If the polymer chain conformations are expressed through the chatacteristic ratio, which is considered to be a better criterion of the unperturbed state since it is not subject to the uncertanities of bond angles [18], higher poly(di–n–alkyl itaconates) reach the values of C∞ from 20 – 31, while most vinyl polymers quoted in the literature have values ranging from 5–15 [21]. The change of σ or C∞ with the molecular mass of the repeat unit fit a slightly curved line, where the first value corresponds to poly(itaconic acid) (σ = 1.53, C∞ = 4.63; M_0 = 130) [22].

Comparing the steric hindrance values for poly(di–n–alkylphenyl itaconates) (Table 7) with those for poly(di–n–cyclohexyl itaconates)(Table 8), it is evident that the more voluminous cyclohexyl residue gives rise to greater short range interaction than the smaller planar phenyl ring. This effect is less pronounced when the ring is distanced from the main chain, by gradually introducing methylene groups (alkyl spacers) in the side chain.

Poly(di–tolyl itaconates) (Table 9) have σ values comparable with those of poly(di–n–alkylphenyl itaconates).

THE GLASS TRANSITON TEMPERATURE

It is well known that the presence of long flexible n–alkyl side chains has a marked effect on the glass transition temperature. In the series of polyolefins, polyacrylates and polymethacrylates the Tg decreases as the number of methylene groups in n–alkyl side chain increases. The data from Table 1 show the same effect in the n–alkyl itaconate polymer series. The first member has a rigid methylene group which makes a barrier to rotation about the backbone bonds. As the n–alkyl side chain becomes longer it is more flexible, its interference with the backbone bonds rotation diminishes and the Tg value is lower. This is the case for the first seven members of the n–alkyl diesters. Members of the n–alkyl series with eight and more carbon atoms in the side chain show a small but constant increase of Tg. The explanation of this anomalous behavior is the consequence of partial crystalization of the long side chains [20]. Similar phenomenon has been noticed for polymethacrylates and polyacrylates.

Rigid substituents, such as phenyl rings, increase the barrier to internal rotation and therefore the Tg values are higher, i.e. the Tg value of PDPhI is 37 K higher than that for PDMI. Data for PDMPhI, PDEPhI and PDPPhI indicate lower Tg values for longer alkyl chains, positioned between the backbone and the rigid phenyl ring [23]. These alkyl chains have the same plastiscizing effect as in the case of n–alkyl itaconates.

The cyclohexyl diester has a higher Tg value than the diphenyl derivative, probably due to greater steric hindrance to rotation in the backbone chain. Alicyclic groups are not rigid but their cyclic structure moves the center of mass of the side group closer to the polymer backbone and the bulkiness of cyclic group shifts the Tg to higher values. The alkyl spacers in alkylcyclohexyl members have the same trend of lowering the Tg as the alkyl group changes from methyl to propyl.

Polyxylenyl– and polytolyl – itaconic acid diesters have high values of Tg, similar to those of phenyl esters, and their solubility parameter change within a very narrow interval, with the exeption of PmTI, having a Tg value 20 K lower than the other members of the same polymer series.

Schneider and Di Marzio [6] resently extended the Gibbs–Di Marzio model of glass transition by introducing the proportionality of Tg with the mass of monomer structural unit divided by the number of flexible bonds (w/γ). Since the possibilities of rotation around flexible bonds in the monomer structural units change the conformational structure of the polymer, the glass transition temperature is determined by conformational entropy changes. The energy difference between the rotational isomers in the chain is higher as the steric hindrance to internal rotation around single bonds increases. This postulate is confirmed for a great number of polymers and polymer blends [24].

The dependence of mass per flexible bond of monomeric unit parameter (w/γ) on the glass transition temperature was analysed for different families of itaconic acid polymeric diesters.

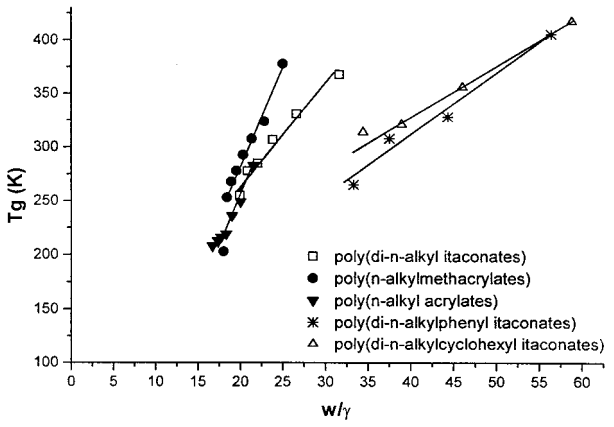

Fig 1. The dependence of Tg on mass per flexible bond parameter, w/γ for itaconate diesters homologous series.

The Tg values for polymeric derivatives of itaconic acid versus the parameter mass per flexible bond are presented in Figure 1. The same dependences for poly (methacrylates) and poly (acrylates), polymer series with similar structures, are presented as well for comparison.

It is evident that the Tg values increase with increasing w/γ and fit a linear relation for each family, but the values for different series of polyitaconates, do not fit the same line, revealing the influence of specific structural features. The rotation of different groups around flexible bonds in the monomer shows that larger groups in n–alkyl series contribute more to the increase of the free volume, i.e. to the decrease of Tg values. This is characteristic for polyitaconates, having a bulky second ester groups, compared to a H – in acrylates or the CH_3–group in methacrylates.

The very rigid phenyl group is responsible for the high Tg and w/γ values for the series of n–alkylphenyl itaconates. The highest w/γ and Tg values are in the case of cyclohexyl derivatives, owing to their bulky side groups positioned close to the polymer backbone.

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IZVOD

TEMPERATURA PRELAZA U STAKLASTO STANJE I NEKA SVOJSTVA DIESTARA POLI(ITAKONSKE KISELINE)

(Pregledni rad)

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Homologi nizovi polimernih diestrara itakonske kiseline, n–alkil–,n–alkilfenil–, n–alkilcikloheksil–, tolil– i ksilenil–, sintetizovani su i karakterisani u našoj laboratoriji. Za sve polimere su eksperimentalno određene vrednosti parametara rastvorljivosti iz rezultata merenja viskoznosti polimera u nizu različitih rastvarača. Ovi rezultati su pokazali dobro slaganje sa računskim vrednostima dobijenim iz doprinosa pojedinih grupa. Parametri koji definišu konformaciju polimera, kao parametar sternog ometanja slobodne rotacije i karakteristični odnos, određeni su iz rezultata merenja viskoznosti i molske mase, na frakcijama polimera. Temperature prelaza u staklasto stanje su određene iz DSC termograma.

Za pojedine familije polimera diskutovana je zavisnost promene pojedinih parametara sa promenom strukture polimera. Eksperimentalno odeđene vrednosti temperatura prelaza u staklasto stanje su povezane sa vrednostima parametara mase po fleksibilnoj vezi za osnovnu strukturnu jedinicu, da bi se potvrdila njihova linearna zavisnost, koja bi omogućila procenu temperature prelaza u staklasto stanje za nove polimere.

Ključne reči: diestri poli(itakonske kiseline) • svojstva • staklasto stanje • rastvorljivost • DSC analiza •

Key words: Poli(itaconic acid) diesters • Properties • Glass transition • Solubility • DSC analysis •