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ORIGINAL PAPER

Poly(imide-amide)s and poly(imide-esters)s based on silarylene units containing (L)-alanine moiety: synthesis and characterization

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Abstract Optically active poly(imide-amide)s (PIAs) and poly(imide-ester)s (PIEs) containing two silicon atoms in the main chain and L-alanine as chiral residue, were synthesized. The former were prepared by direct polycondensation between two dicarboxilic acids and bis(4-aminophenyl)dipenylsilane according to the triphenyl phosphate method. PIEs were synthesized with bis(4-hydroxyphenyl)dimethyl o ethylmethylsilane, according to the tosyl chloride method. Monomers and polymers were characterized by IR and ^{1}H, ^{13}C, and ^{29}Si NMR spectroscopy and elemental analysis, and the results were in agreement with the proposed structures. PIAs and PIEs showed low values of η_{inh}, indicative of low molecular weight species, probably of oligomeric nature. Polymers were soluble in polar aprotic solvents and also in common solvents such as CH_2Cl_2, $CHCl_3$, and acetone, due to the effect of both, the polarity of the Si–C bond and the presence of an aliphatic residue provided by the L-alanine amino acid. The glass transition temperatures (T_g) of the PIAs were higher than those obtained for PIEs, due to the higher flexibility of the ester group. The thermal decomposition temperatures (TDT) were lower than 400 °C and dependent of the polymer structure.

Keywords Poly(imide-amide) · Poly(imide-esters) · Silicon-containing polymers · Optically active polymers · Glass transition temperature · Thermal decomposition temperature

Introduction

Poly(imides), poly(esters), and poly(amides) are important heat-resistant polymers used as high-performance engineering materials. In general, the attention has been

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focused on the synthetic methods and on the structural variances in order to upgrade the processability and solubility through the design of new monomers in order to obtain polymers with improving solubility but maintaining the thermal stability [1]. In the same sense, the modifications can include two or more functional groups and the introduction of flexible linkages of aliphatic nature or bulky substituents in the polymeric chain [2].

On the other hand, the introduction of silarylene groups in the repeating unit [–Ar–Si(R_1R_2)–Ar–], described by Bruma et al. [3], can improve the solubility maintaining the thermal stability. In this sense, the lower electronegativity of the Si atom compared to carbon, increases the thermal stability of silicon-containing polymers due to a higher ionic character of the C–Si linkage compared with the C–C bond, in spite of their similar bond energy dissociation, which permits electronic transport, thus also increasing the solubility and thermal stability.

In addition, the introduction of chiral atoms in the polymeric chain has been very attractive in recent years. Mallakpour et al. have described in a recent review about the synthesis of optically active condensation polymers that the introduction of chiral carbons in the polymeric structure is an attractive topic due to the wealthy and multifacetic architecture of chiral polymers compared with small molecules. They mention that the synthesis of chiral polymers containing amino acids is a subject of interest, because a high degree of amino acid functionality can lead to polymers with increased solubility and the ability to form secondary structures [4].

Recently, we published a work about the synthesis and characterization of poly(imide-amides) and poly(imide-esters) containing two Si atoms in the main chain and glycine as part of the diacid residue. These polymers are insoluble in common solvents, such as $CHCl_3$ and CH_2Cl_2, and show thermal properties that depend on the groups, methyl or ethyl, bonded to the Si atom of the diacid residue, for poly(imide-amides), and to the Si atom of both residues, diacid and diphenol, for poly(imide-esters) [5].

Continuing with this work, here we described the synthesis, characterization and thermal properties of analogous poly(imide-amides) and poly(imide-esters) but incorporating L-alanine as chiral amino acid in the diacid residue, and containing two Si atoms in the main chain.

Experimental

Materials

L-alanine, $(CH_3)_2SiCl_2$, $(CH_3CH_2)(CH_3)SiCl_2$, triphenyl phosphite (TPP), $CaCl_2$, *N*-methyl-2-pyrrolidone (NMP), *p*-bromophenol and 4-bromo-*N*,*N*-bis(trimethylsilyl)aniline were obtained from Aldrich Chemical (Milwaukee, WI) and used without further purification. AlfaAesar provided 4-bromo-*o*-xylene. Tetrahydrofuran (THF) and diethylether (Aldrich Chemical, Milwaukee, WI) were dried over sodium previous to use. All other reagents and solvents were purchased commercially as analytical-grade and used without further purification.

The IR spectra (KBr pellets) were recorded on a Perkin-Elmer 1310 spectrophotometer over the range 450–4000 cm^{-1}. ^{1}H, ^{13}C, and ^{29}Si NMR spectra were carried out on a 400 MHz instrument (Bruker AC-200) using DMSO-d_6, $CDCl_3$ or acetone-d_6 as solvents and TMS as the internal standard. Viscosimetric measurements were made in a Desreux-Bischof type dilution viscosimeter at 25 °C ($c = 0.3$ g/dL). T_g values were obtained with a Mettler-Toledo DSC 821 calorimetric system (20 °C min^{-1} under N_2 flow). Thermogravimetric analyses were carried out in a Mettler TA-3000 calorimetric system equipped with a TC-10A processor, and a TG-50 thermobalance with a Mettler MT5 microbalance. Samples of 6–10 mg were placed in a platinum sample holder and the thermogravimetric measurements were carried out between 30 and 800 °C with a heating rate of 20 °C min^{-1} under N_2 flow. Specific rotations were measured in an Optical Activity Automatic polarimeter, Model AA-5 at 17 °C. Elemental analyses were performed in a Fisons EA 1108-CHNS-O equipment.

Monomer synthesis

The precursors bis(3,4-dimethylphenyl)-dimethylsilane, bis(3,4-dimethylphenyl)-ethylmethylsilane, bis(3,4-dicarboxyphenyl)-dimethylsilane, bis(3,4-dicarboxyphenyl)-ethylmethylsilane, bis(3,4-dicarboxyphenyl)-dimethylsilane dianhydride and bis(3,4-dicarboxyphenyl)-ethylmethylsilane dianhydride were synthesized following procedures described previously [6–9]. The silicon-containing diphenols and the diamine bis(4-aminophenyl)-diphenylsilane were obtained according to described procedures [10, 11].

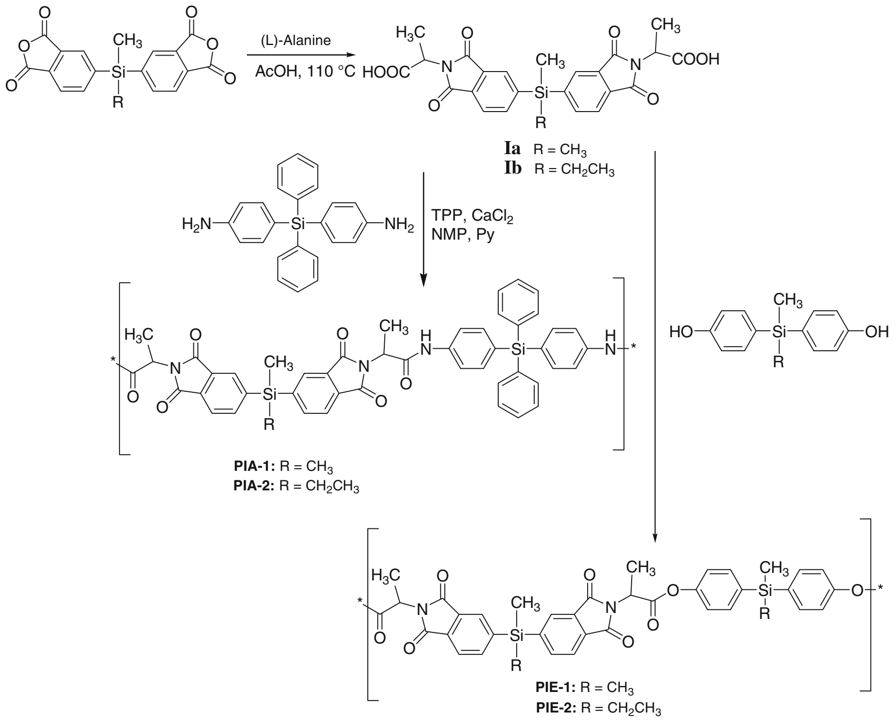
N,*N*′-(4,4′-dimethylsilylenediphthaloyl)-bis-(L)-alanine and *N*,*N*′-(4,4′-ethylmethylsilylenediphthaloyl)-bis-(L)-alanine diacids were synthesized following reported procedures (Scheme 1), in which the dianhydride derivative (2.8 mmol) and L-alanine (5.6 mmol) are refluxed 3 h in 30 mL of acetic acid. The solvent was removed by distillation under reduced pressure and the residue treated with water and drops of HCl and filtered. The obtained solid was washed several times with water for removing the L-alanine remained. Finally, the diacid was dried at 40 °C under vacuum until constant weight and characterized [12].

*N,N’-(4,4′-dimethylsilylenediphthaloyl)-bis-(L)-alanine diacid (**I-a**)*

Yield: 74%. M.p.: 199–202 °C. $[\alpha]_{589}^{17}$ (acetone) = −21.9 deg dm^{-1} g^{-1} cm^3. IR (KBr) (cm^{-1}): 3241 (OH), 3064 (CH arom.), 2961 (CH aliph.), 1775 (C=O imide), 1717 (C=O acid), 1610 1526 (C=C arom.), 1459 (CH_3), 1252 (Si–C aliph.), 837, 784 (arom. 1,2,4-subst.). ^{1}H NMR (acetone-d_6) (δ) (ppm): 0.81 (s, 6H, Si–CH_3), 1.67 (d, 6H, CH–CH_3), 4.97 (q, 2H, CH), 7.87–8.12 (m, 6H, arom.). ^{13}C NMR (acetone-d_6) (δ) (ppm): −3.86 (Si–CH_3), 14.4 (CH_3CH), 47.0 (CH), 122.4, 128.3, 131.2, 133.0, 140.3, 145.8 (C arom.) 167.1; 167.3 (C = O imide), 170.2 (C=O acid). ^{29}Si NMR (acetone-d_6) (δ) (ppm): −4.5.

*N,N′-(4,4′-ethylmethylsilylenediphthaloyl)-bis-(L)-alanine diacid (**I-b**)*

Yield: 63%. M.p.: 219–221 °C. $[\alpha]_{589}^{17}$ (acetone) = −22 deg dm^{-1} g^{-1} cm^3. IR (KBr) (cm^{-1}): 3392 (OH), 3080 (CH arom.), 2954 (CH aliph.), 1774 (C=O imide),



Scheme 1 Synthetic route for PIAs and PIEs

1715 (C=O acid), 1611 (C=C arom.), 1452, 1383 (CH_3), 1256 (Si–C aliph.), 839, 789 (arom. 1,2,4-subst.). ^{1}H NMR (DMSO-d_6) (δ) (ppm): 0.70 (s, 3H, Si–CH_3), 0.96 (t, 3H, $CH_2\mathit{CH_3}$), 1.26 (q, 2H, $\mathit{CH_2}CH_3$), 1.53 (d, 6H, CH–$\mathit{CH_3}$), 4.94 (q, 2H, CH), 7.87–8.13 (m, 6H, arom.). ^{13}C NMR (DMSO-d_6) (δ) (ppm): −5.4 (Si–CH_3), 4.77 ($\mathit{CH_2}CH_3$), 7.49 ($CH_2\mathit{CH_3}$), 15.4 ($CH\mathit{CH_3}$), 48.1 (CH), 122.9, 128.7, 131.2, 132.9, 140.9, 144.9 (C arom.), 167.7, 167.9 (C=O imide), 169.5 (C=O acid). ^{29}Si NMR (DMSO-d_6) (δ) (ppm): −2.6.

Poly(imide-amide) and poly(imide-ester) synthesis

Poly(imide-amides) (PIAs)

Poly(imide-amides) were synthesized according to a described procedure [13, 14], in which bis(4-aminophenyl)-diphenylsilane (0.5 mmol) and the diacid *N,N*′-(4,4′-dimethylsilylenediphthaloyl)-bis-(L)-alanine or *N,N*′-(4,4′-ethylmethylsilylene diphthaloyl)-bis-(L)-alanine (0.5 mmol) were mixed with triphenyl phosphite (0.366 mL, 1.39 mmol), $CaCl_2$ (0.122 g, 1.1 mmol), pyridine (0.33 mL) and *N*-methyl-2-pyrrolidone (0.62 mL), and the mixture was heated at 110–130 °C for 3 h. After this time, the mixture was poured into methanol and the PIA filtered, washed, dried until constant weight and characterized.

PIA-1 Yield: 48%. $[\alpha]_{589}^{17}$ (acetone) = -13 deg dm^{-1} g^{-1} cm^3. IR (KBr) (cm^{-1}): 3434 (NH), 3067 (CH arom.), 2955 (CH aliph.) 1744, 1718 (C=O), 1591, 1491 (C=C arom.), 1443 (Si–C arom.), 1250 (Si–C aliph.), 814 (arom. *p*-subst.), 738, 714 (arom. *mono*-subst.). ^{1}H NMR (DMSO-d_6) (δ) (ppm): 0.72 (s, 6H, CH_3–Si), 1.15 (d, 6H, CH_3CH), 4.9 (q, 2H, CH), 7.41–8.02 (m, 24H, arom.), 9.97 (s, 2H, NH). ^{13}C NMR (DMSO-d_6) (δ) (ppm): -2.9 (CH_3–Si), 15.7 (CH_3CH), 49.3 (CH), 120.6, 122.8, 128.5, 128.7, 130.2, 131.4, 132.7, 135.1, 135.4, 135.5, 136.2, 136.8, 140.9, 145.9 (C arom.), 167.8, 168 (C=O). ^{29}Si NMR (DMSO-d_6) (δ) (ppm): -4.1 [Ph–Si$(CH_3)_2$–Ph], -15.3 [Ph–Si$(Ph)_2$–Ph]. Elem. Anal. Calcd. for $[C_{48}H_{40}N_4O_6Si_2]_n$; (824): C: 69.90%, N: 6.80%, H: 4.85%. Found: C: 70.73%, N: 6.50%, H: 4.68%.

PIA-2 Yield: 98%. $[\alpha]_{589}^{17}$ (acetone) = -41.1 deg dm^{-1} g^{-1} cm^3. IR (KBr) (cm^{-1}): 3466 (NH), 3069 (CH arom.), 2957, 2875 (CH aliph.), 1774, 1717 (C=O), 1593, 1515 (C=C arom.), 1443 (Si–C arom.), 1250 (Si–C aliph.), 833 (arom. *p*-subst.), 741, 700 (arom. *mono*-subst.). ^{1}H NMR (DMSO-d_6) (δ) (ppm): 0.72 (s, 3H, CH_3–Si), 0.97 (t, 3H, CH_3CH_2), 1.26 (q, 2H, CH_2), 1.56 (d, 6H, CH_3CH), 4.92 (q, 2H, CH), 6.69–8.01 (m, 24H, arom.), 9.9 (s, 2H, NH). ^{13}C NMR (DMSO-d_6) (δ) (ppm): -5.5 (CH_3–Si), 4.6 (CH_3CH_2), 7.5 (CH_2–CH_3), 15.6 (CH_3CH), 49.3 ($\mathit{CH}CH_3$), 115.2, 119.8, 120.8, 122.8, 128.7, 129.2, 130.6, 132.7, 135.1, 135.8, 136.6, 136.8, 137.2, 145.2 (C arom.), 167.9, 168.3 (C=O). ^{29}S NMR (DMSO-d_6) (δ) (ppm): -2.6 [Ph–Si$(CH_3)(CH_2CH_3)$–Ph], -15.3 [Ph–Si$(Ph)_2$–Ph]. Elem. Anal. Calcd. for $[C_{49}H_{42}N_4O_6Si_2]_n$; (838): C: 70.17%, N: 6.68%, H: 5.01%. Found: C: 71.23%, N: 7.02%, H: 4.90%.

Poly(imide–esters) (PIEs)

Poly(imide-esters) were synthesized according to a described procedure [15], in which 1.07 mmol of tosyl chloride, 0.02 mL of pyridine, 0.06 mL of *N*,*N*-dimethylformamide and 0.05 mmol of the diacid in 0.02 mL of pyridine were mixed, and the mixture stirred at room temperature for 30 min. After this time, 0.05 mmol of the diphenol and 0.02 mL of pyridine were added and the new mixture stirred for 30 min at room temperature. Then, the mixture was heated at 120 °C for 2 h, and poured in methanol–water 1:4 mixture. The polymer was filtered, washed, dried until constant weight and characterized.

PIE-1 Yield: 53%. $[\alpha]_{589}^{17}$ (acetone) = -13.2 deg dm^{-1} g^{-1} cm^3. IR (KBr) (cm^{-1}): 3060 (CH arom.), 2977, 2939 (CH aliph.), 1772, 1713 (C = O), 1598, 1503 (C = C arom.), 1450, 1383 (CH_3), 1447 (Si–C arom.), 1248 (Si–C aliph.), 837 (arom. 1,2,4-subst.), 814 (arom. *p*-subst.). ^{1}H NMR (acetone-d_6) (δ) (ppm): 0.81 (s, 12H, CH_3–Si), 1.67 (d, 6H, $C\mathit{H}_3$–CH), 4.95–5.0 (q, 2H, CH), 7.87–8.11 (m, 14H, arom.). ^{13}C NMR (acetone-d_6) (δ) (ppm): -3.84 (CH_3–Si), 14.4 (CH_3–CH), 47.1 (CH), 122.2, 122.4, 122.5, 128.4, 131.2, 131.3, 132.6, 132.9, 134.8, 145.8 (C arom.), 167.1, 167.3 (C=O imide), 170.4 (C=O ester). ^{29}Si NMR (acetone-d_6) (δ) (ppm): -4.5. Elem. Anal. Calcd. for $[C_{37}H_{34}N_4O_8Si_2]_n$; (690): C: 64.35%, N: 4.06%, H: 4.93%. Found: C: 63.74%, N: 3.87%, H: 4.61%.

PIE-2 Yield: 54%. $[\alpha]^{17}_{589}$ (acetone) = −13 deg dm^{-1} g^{-1} cm^3. IR (KBr) (cm^{-1}): 3061 (CH arom.), 2962, 2877, 2914 (CH aliph.), 1774, 1718 (C=O), 1590, 1498 (C=C arom.), 1459, 1381 (CH aliph.), 1255 (Si–C aliph.), 907, 833 (arom. 1,2,4-subst.), 791 (arom. *p*-subst.). ^{1}H NMR (acetone-d_6) (δ) (ppm): 0.33 (s, 6H, CH_3–Si), 0.53 (q, 4H, CH_2), 0.92 (d, 6H, CH_3–CH_2), 1.35 (d, 6H, $C\mathit{H}_3$–CH), 4.96–5.01 (q, 2H, CH), 6.84–8.08 (m, 14H, arom.). ^{13}C NMR (acetone-d_6) (δ) (ppm): −5.5 (CH_3–Si), 5.6 ($\mathit{C}H_3$–CH_2), 7.4 (CH_2), 15.5 ($\mathit{C}H_3$–CH), 48.2 (CH), 121.9, 122.0, 123.3, 123.4, 129.5, 132.1, 133.8, 135.4, 136.5, 141.5 (arom.), 167.9, 168.1 (C=O imide), 169.3 (C = O ester). ^{29}Si NMR (acetone-d_6) (δ) (ppm): −2.51, −2.53. Elem. Anal. Calcd. for $[C_{39}H_{38}N_2O_8Si_2]_n$; (718): C: 65.18%, N: 3.90%, H: 5.29%. Found: C: 66.23%, N: 3.71%, H: 5.01%.

Results and discussion

The tetramethyl derivatives, tetraacids and dianhydrides were synthesized according to procedures described previously [6–9]. The diphenols and the diamine were obtained as described in the literature [10, 11].

The dianhydrides were reacted with L-alanine in acetic acid under reflux in order to obtain the silicon-containing diacids **Ia** and **Ib**. The purification of these diacids was achieved washing with water in order to eliminate the residue of L-alanine. The characterization was made using IR and NMR spectroscopy, and the results were in agreement with the proposed structures and are shown in the experimental part.

Poly(imide-amide)s were obtained by direct reaction between the diacid and the diamine, according to the method using triphenyl phosphite in *N*-methyl-2-pyrrolidone and pyridine with $CaCl_2$, shown in Scheme 1. Polymers were precipitated in methanol, dried and characterized by spectroscopic methods. The results for PIA-1 and PIA-2 are in agreement with the proposed structures.

The ^{29}Si NMR spectrum of PIA-1 showed the signals corresponding to the two Si atoms in the main chain, being the first at −4.1 ppm, bonded to aliphatic groups, and the second at −15.3 ppm, bonded to aromatics rings only. For PIA-2, the ^{29}Si NMR spectra showed the same signal for the Si atom of the diamine residue, but the other at −2.6 ppm due to the effect of the ethyl group. These results are in agreement with those obtained for other monomers and polymers containing Si in their structure [5, 16].

Poly(imide-amide)s were obtained directly from the diacids and the diphenols with tosyl chloride in *N,N*-dimethylformamide, and the polymers were precipitated in a methanol/water mixture, dried and characterized by spectroscopic methods. The ^{29}Si NMR showed one signal for PIE-1 at −4.5 ppm, which was described for us in other systems in which the Si atom is bonded to methyl groups. For PIE-2, it was possible to observe two closely similar signals for the Si atoms, at −2.51 and −2.53 ppm, related to two very similar magnetic environments. The two signals observed in the ^{29}Si NMR spectra corresponding to the two Si atoms are indicative of the polymer formation.

Table 1 shows the solubility of the polymers, which are soluble in aprotic polar solvents such as dimethylsulfoxide and *N,N*-dimethylformamide,

Table 1 Solubility of the PIAs and PIEs

Polymer	DMSO	DMF	DMAc	*m*-cresol	$CHCl_3$	CH_2Cl_2	Acetone
PIA-1	+	+	+	+	+	+	+
PIA-2	+	+	+	±	+	+	+
PIE-1	+	+	+	±	+	+	+
PIE-2	+	+	+	+	+	+	+

+, Soluble at room temperature; ±, partially insoluble at room temperature

Table 2 Yields, η_{inh}, glass transition temperatures (Tg) and thermal decomposition temperatures (TDT) of the PIAs and PIEs

	Yield (%)	η_{inh} (dL/g)[a]	T_g (°C)	TDT[b] (°C)
PIA-1	48	0.06	180	280
PIA-2	98	0.06	111	286
PIE-1	53	0.06	80	188
PIE-2	54	0.10	60	365

[a] Inherent viscosity, in $CHCl_3$ at 25 °C ($c = 0.3$ g/dL)

[b] 10% weight loss temperature

N,*N*-dimethylacetamide, partially soluble in *m*-cresol, but soluble in acetone, $CHCl_3$ and CH_2Cl_2, due probably to the effect of the amino acid and the imide group. In fact, the imide group and the aliphatic residue increase the interactions with the aprotic polar solvents and increase the solubility of the material.

Table 2 shows the yields and the η_{inh} values obtained for the four polymers. The yields were normal with the exception of PIA-2, which was practically quantitative. The η_{inh} values are similar and low, which is indicative that polymers are of low molecular weight, probably of oligomeric nature.

Table 2 also shows the T_g values of the PIAs and PIEs. PIAs showed higher T_g values than PIEs due to the fact that the former are derived from a wholly aromatic diamine, and the amide group makes the PIAs more rigid than the PIEs, which have a more flexible ester group. When PIA-1 and PIA-2 are compared, can be noted that when the ethyl group is present in the acid residue there is a decrease in the T_g value, due to an increase of the flexibility of the side groups of the polymeric chain. The same effect it is possible to see in the PIEs. The increase of the volume of the side groups increases the flexibility and the asymmetry of the chain, being a consequence a decrease of the T_g values. This behaviour is in agreement with that described for analogous silicon-containing poly(imide-esters) but derived from glycine instead of L-alanine [5]. For PIEs, the values shown in this work are lower, due to the effect of the methyl group of L-alanine in comparison with the effect of the glycine without side groups.

Table 2 also shows the thermal decomposition temperatures (TDT) for all polymers, which is the temperature at which the polymers lose 10% of weight. In general, a polymer is considered as thermostable when the weight loss at 400 °C is lower than 10%, and in this sense these polymers con not be considered as thermostables.

The TDT values of PIAs are practically the same, and also similar to those obtained for analogous PIAs and PIEs derived from diacids with glycine [5]. PIA-1 showed a TDT value of 280 °C, which is lower than that obtained for the analoge with glycine (294 °C). The methyl group of the L-alanine makes the polymer more flexible, and consequently with less thermal stability. The inverse occurs with PIA-2 with a TDT value of 286 °C respect to the analoge with glycine (275 °C), but the small difference does not allow to obtain more conclusions.

There is a great difference between PIE-1 and PIE-2, being the difference the ethyl groups bonded to both Si atoms. If we compare these polymers with those derived from glycine, the trend is the inverse, because the polymer with ethyl groups bonded to the Si atom has less stability than that with methyl group bonded to the Si atom. In fact, it seems that the methyl side group of the L-alanine and the ethyl groups bonded to the Si atoms in PIE-2, increase the interactions between the chains, as well as the asymmetry and, consequently, the thermal stability in this poly(imide-ester).

Conclusions

Silicon-containing poly(imide-amides) (PIAs) and poly(imide-esters) (PIEs) were obtained from two diacids, derived from a dianhydride and L-alanine, and bis(4-aminophenyl)diphenylsilane, for PIAs, and bis(4-hydroxyphenyl)dimetyl- or ethyl-methylsilane, for PIEs. The presence of a flexible and aliphatic residue increases the solubility in common organic solvents such as $CHCl_3$ and CH_2Cl_2. Low values of η_{inh} are indicative of low molecular weight polymers of oligomeric nature. The T_g and TDT values are influenced by the nature of the groups bonded to the Si atom of the acid residue, in the case of PIAs, and both, the acid and the diphenol, in the case of the PIEs.

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