Electrical Conduction and Dielectric Properties of Poly(methyl methacrylate)/Perylene Solar Concentrators

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ABSTRACT: Poly(methyl methacrylate) doped with fluorescent perylene dye was prepared by both radical polymerization of methyl methacrylate and solvent casting from polymer solutions. The samples were characterized by differential scanning calorimetry, Fourier transform infrared spectroscopy, electrical conductivity, and dielectric properties. Both conductivity and dielectric properties were measured in the temperature range 303–433 K and the frequency range 10^3 to 5×10^6 Hz. The results show that the direct-current electrical conductivity increased by increasing dye content in solvent-cast samples, whereas it decreased radically polymerized samples. The results of alternating-current conductivity suggest electron hopping between filled and empty localized states. The study of dielectric properties showed two relaxation peaks corresponding to the dipole segmental and dipole group losses. Explanations based on the polymer free volume and acid–base interactions were proposed to examine the influence of the sample preparation and perylene dye concentration on the glass-transition temperature and dielectric relaxation of the samples. The obtained results recommend the thermal and molecular stability of luminescent solar concentrator (LSC) matrices prepared by radical polymerization over those prepared by solvent casting. © 2003 Wiley Periodicals, Inc. J Appl Polym Sci 88: 793–805, 2003

Key words: poly(methyl methacrylate); dyes; pigments; fluorescence; FT-IR; activation energy

INTRODUCTION

Organic dyes play an important role in the development of optical systems such as luminescent solar concentrators (LSCs),[1] laser dyes,[2] materials for phase conjugation,[3] and solid-state indicators for environmental and biological impurities.[4] LSCs have attracted attention for almost 2 decades, since the first proposal by Weber and Lambe.[5] The operation of LSCs is based on the absorption of solar radiation in a transparent material [e.g., poly(methyl methacrylate) (PMMA)] impregnated with guest fluorescent absorbers (e.g., organic dye molecules). The fluorescent radiation is trapped by total internal reflection and concentrated at the plate material edges, where it is converted to electricity with a solar cell facing such edges.[6] In fact, research activity in this field decreased for some years ago due to the poor photostability of organic laser dyes. The development of a new class of perylene dyes with excellent photostability and high quantum efficiency has encouraged the research in this area.[7–9] The physical properties of PMMA are usually influenced by the presence of relaxation transitions, which are directly related to polymer structure, are low-energy transitions, and often occur over a narrow temperature range. Therefore, analyzing these transitions requires a technique that offers high sensitivity and good resolution. Dielectric analysis provides both of these required capabilities.[10] In addition, a necessary requirement for an LSC system is a large stokes shift, which is known to be a function of the dielectric constant (ϵ') and the refractive index of the substrate material.[11]

In this study, we examined, by means of differential scanning calorimetry (DSC), Fourier transform infrared spectroscopy (FTIR), conductivity, and dielectric properties, the influence of sample preparation and perylene dye concentration (C) on the stability of PMMA to optimize the use of such matrices as LSCs.

EXPERIMENTAL

Pure methyl methacrylate (MMA) with a molecular weight of 100.12 g/mol, obtained from Merck (Germany), was used in this study. Pure-grade benzoyl peroxide (BP), supplied by BDH (England), was used as initiator after dissolution in chloroform and reprecipitation by the addition of excess methyl alcohol, filtering, and drying. Both PMMA and the perylene dye used in this study were obtained from Aldrich (England) and were reported to have molecular weights of 996,000 and 252.32 g/mol, respectively.

TABLE I
Values of T_g and T_s for PMMA/Perylene Samples

C (mol %)	T_g (K)	T_s (K)
Radically polymerized sample		
0.00	395	—
5.28×10^{-5}	396	—
6.33×10^{-5}	399	—
7.92×10^{-5}	402	—
1.06×10^{-4}	395	—
1.37×10^{-4}	392	—
2.11×10^{-4}	391	—
Solvent-cast sample		
0.0	347	416
1.78×10^{-4}	347	427
5.00×10^{-4}	348	435

PMMA/perylene samples were prepared by both radical polymerization and casting methods. In the radical method, the polymerization of MMA and different amounts of perylene was performed with BP as an initiator (1 wt %), and the mixture was poured into a glass mold. Then, the mold was immersed in water bath at 60°C for 72 h, followed by a final drying at 95°C for 6 h. In the casting method, grains of both PMMA and perylene were dissolved in chloroform and mixed with a magnetic stirrer. The homogenous mixture was poured into a glass container to evaporate the solvent off.

The number-average molecular weight (M_n) of the prepared samples was estimated by measurement of the intrinsic viscosity ($[\eta]$) of the polymer solution with an Ostwald viscometer and with the relation:[12]

$$[\eta] = a\ \overline{M}_n{}^b$$

where a and b are constants equal to 0.7 and 7.7×10^{-5}, respectively, for the used solvent (acetone) at 30°C. The calculated value of average molecular weight was 1,115,000 g/mol.

X-ray diffraction of all the investigated samples was carried out with a Phillips KW 1840 diffractometer (Holland), with a $K\alpha1$ filter and a Cu target source. An amorphous (atactic) structure was confirmed for all the prepared samples.
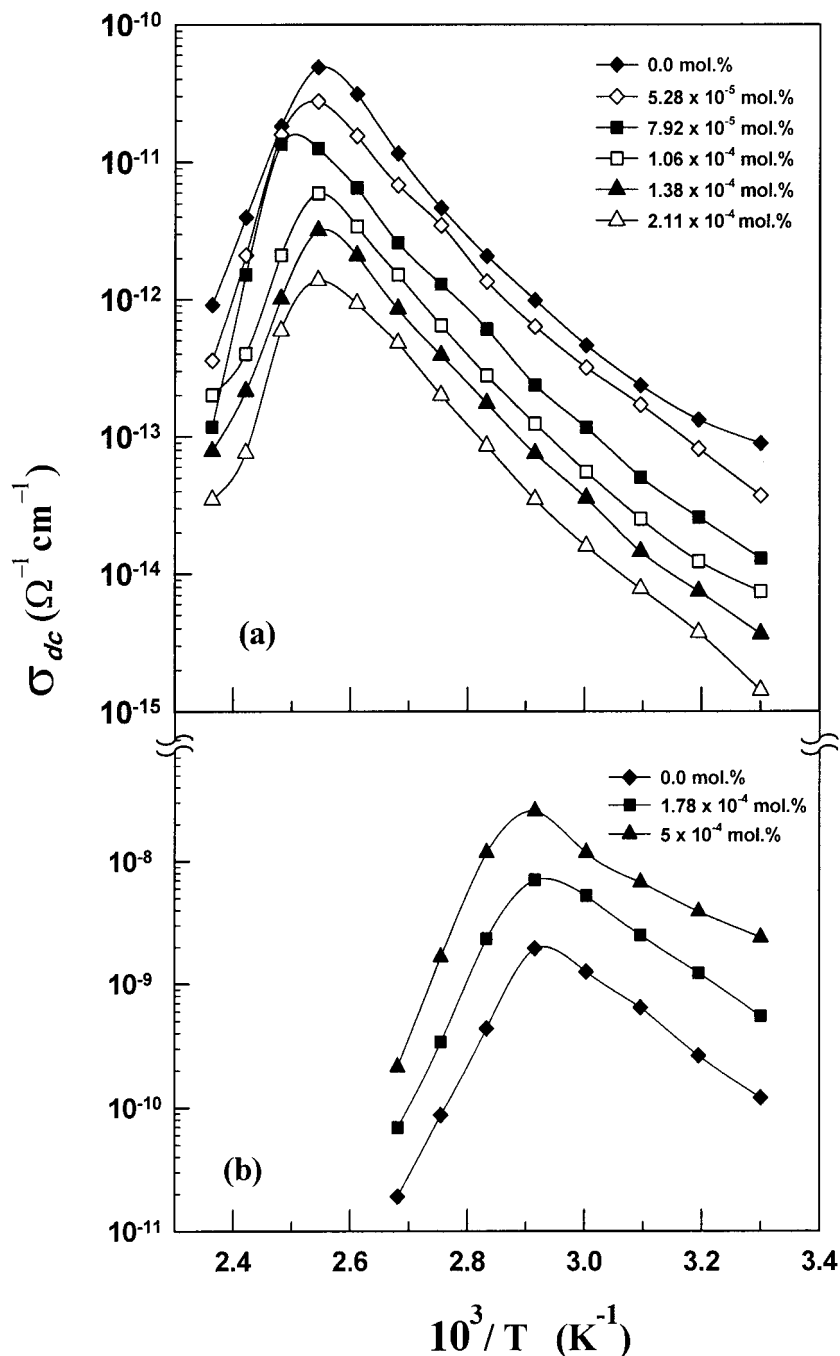
DSC investigation of the samples was carried out with a thermal analyzer (model Setaram 131) (France) at a heating rate of 20°C/min.

FTIR spectra for the pure and doped samples were recorded in the wave number range $400-4000$ cm^{-1} with an FTIR Genesis Series spectrophotometer (USA).

The electrical properties, direct-current and alternating-current conductivities (σ_{dc} and σ_{ac}, respectively), dielectric constant (ϵ'), and dielectric loss (ϵ''), were measured on discs 1.6 cm^2 in area and 0.3–0.5 mm thick. The two parallel surfaces of each disc were coated with silver paint and checked for good conduction. All samples were thermally aged before measurement in an electric oven at 333 K for 24 h to get reasonable stability and reproducibility of results. For all samples, the electrical measurements were carried out in the temperature range 303–433 K. σ_{dc} was measured with the two-probe method with a picoammeter (Keithley model 485, USA) as a current meter. Measurements under the ac field were performed at various frequencies (Fs) ranging from 10^3 to 5×10^6 kHz

TABLE II
Wavenumber (cm^{-1}) of the Vibrational Transmission Peaks of the Characteristic Groups in the PMMA/Perylene Samples

C (mol %)	—C—CH3 bending	—C—CH2—C— bending	C=O stretching (ester)	C—O symm. stretch. (ester)	C—H stretching (aromatic)	C=C stretching (aromatic)	C—CI
Radically polymerized sample							
0.00	1375	1445	1734	1117	—	—	—
5.28×10^{-5}	1361	1451	1737	1108	3083	1648	—
						1509	—
6.33×10^{-5}	1371	1481	1742	1108	3076	1639	—
				1229			—
7.92×10^{-5}	1360	1455	1746	1258	3022	1648	—
						1509	—
1.06×10^{-4}	1364	1431	1737	1105	3082	1648	—
1.37×10^{-4}	1361	1449	1734	1227	3027	1509	—
2.11×10^{-4}	1371	1446	1731	1113	3023	1647	—
				1235		1508	—
Solvent-cast sample							
0.00	2953	2844	1734	1069	—	—	656
				1081			
1.78×10^{-4}	2969	2938	1738	1099	3090	1509	665
		2846				1575	
5.00×10^{-4}	2961	2926	1742	1070	3082	1509	725
		2853				1648	

Figure 1 Temperature dependence of σ_{dc} for PMMA and PMMA/perylene samples prepared by (a) radical polymerization and (b) solvent casting.

with a computerized LCR Hi-Tester (model Hoiki 3532, Japan).

RESULTS AND DISCUSSION

DSC measurements performed for both radically polymerized and solvent-cast PMMA/perylene samples showed a glass-transition temperature (T_g), whereas an endothermic peak appeared for the solvent evapo-

ration temperature (T_s) of solvent-cast samples. The values of T_g and T_s are listed in Table I. The value of T_g for PMMA prepared by radical polymerization was a little bigger than those published in the literature;[13–15] this could be due to the heating-rate dependence of T_g for atactic PMMA.[15] This was expected because there was no evidence for other contributing parameters affecting T_g such as residual monomer or initiator, as was confirmed by the FTIR results. The increase in T_g

TABLE III
Calculated Values of ΔE_{dc}, Peak Conductivity (σ_p), T_p, and σ_o for the PMMA/Perylene Samples

C (mol %)	ΔE_{dc} (eV)	σ_p (Ω^{-1} cm^{-1})	T_p (K)	σ_o (Ω^{-1} cm^{-1})
Radically polymerized sample				
0.00	0.76	6.76×10^{-11}	395	2.39×10^{-1}
6.33×10^{-5}	0.76	2.75×10^{-11}	396	1.31×10^{-1}
7.92×10^{-5}	0.79	1.35×10^{-11}	402	1.12×10^{-1}
1.06×10^{-4}	0.80	5.93×10^{-12}	394	7.80×10^{-2}
1.37×10^{-4}	0.80	3.19×10^{-12}	392	5.50×10^{-2}
2.11×10^{-4}	0.81	1.38×10^{-12}	391	3.30×10^{-2}
Solvent-cast sample				
0.00	0.64	1.95×10^{-9}	341	58.3×10^{-1}
1.78×10^{-4}	0.59	7.08×10^{-9}	343	38.3×10^{-1}
5.00×10^{-4}	0.52	2.56×10^{-8}	347	10.1×10^{-1}

of radically polymerized samples rather than solvent-cast samples was due to two cooperating effects; the first was the high molecular weight of radically polymerized samples, and the second was due to the retained solvent molecules in the solvent-cast samples inducing a decrease in T_g.[16] The apparent increase in T_s by increasing dye content refers to the aggregation of dye molecules compared to that of the side group of the polymer, which tends to hinder the total release of confined solvent molecules between polymer chains.[17] The T_g variation with C in the case of radically polymerized samples could be attributed to the irregular variation of the polymer matrix free volume.

FTIR spectroscopy was used to study PMMA and PMMA/perylene samples prepared by radical and
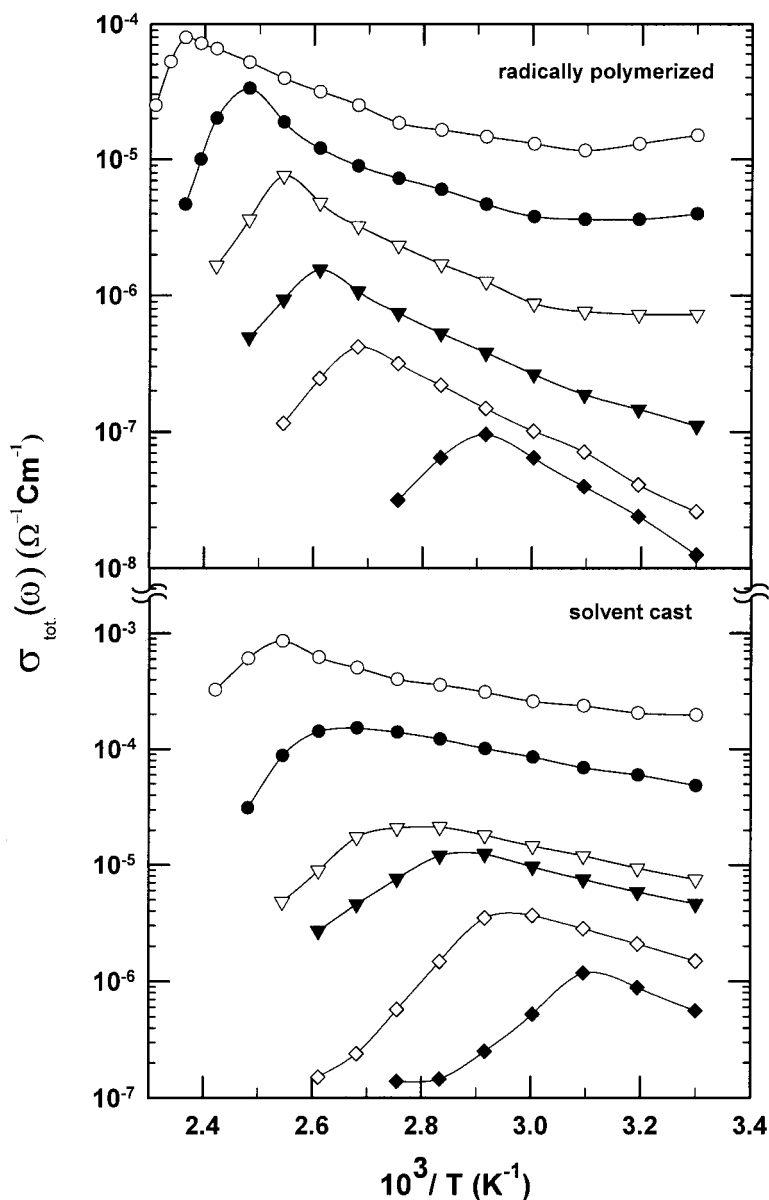
Figure 2 Temperature dependence of $\sigma_{tot}(\omega)$ for pure PMMA samples at field Fs of ($\blacklozenge$) 1 kHz, ($\lozenge$) 10 kHz, ($\blacktriangledown$) 50 kHz, ($\triangledown$) 100 kHz, ($\bullet$) 500 kHz, and ($\circ$) 1 MHz.
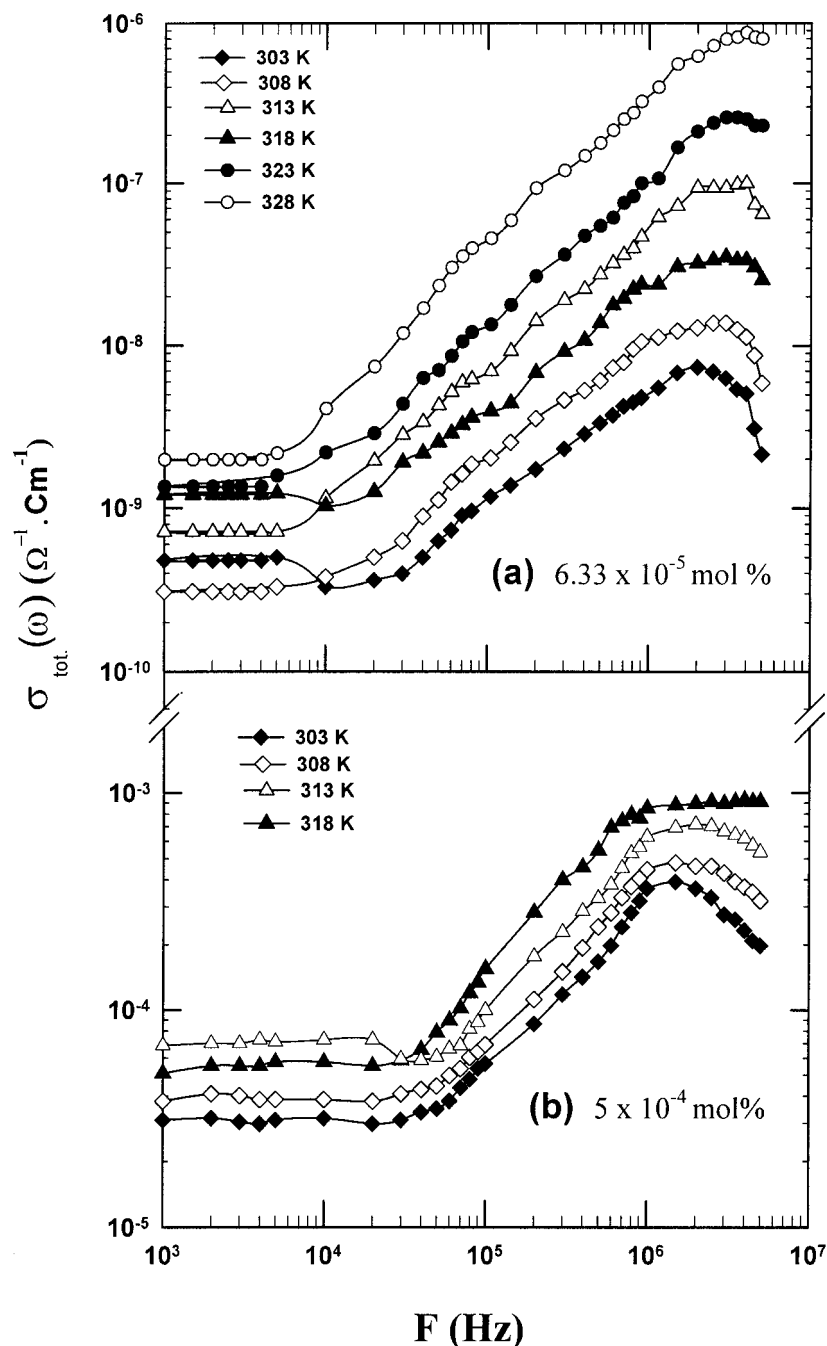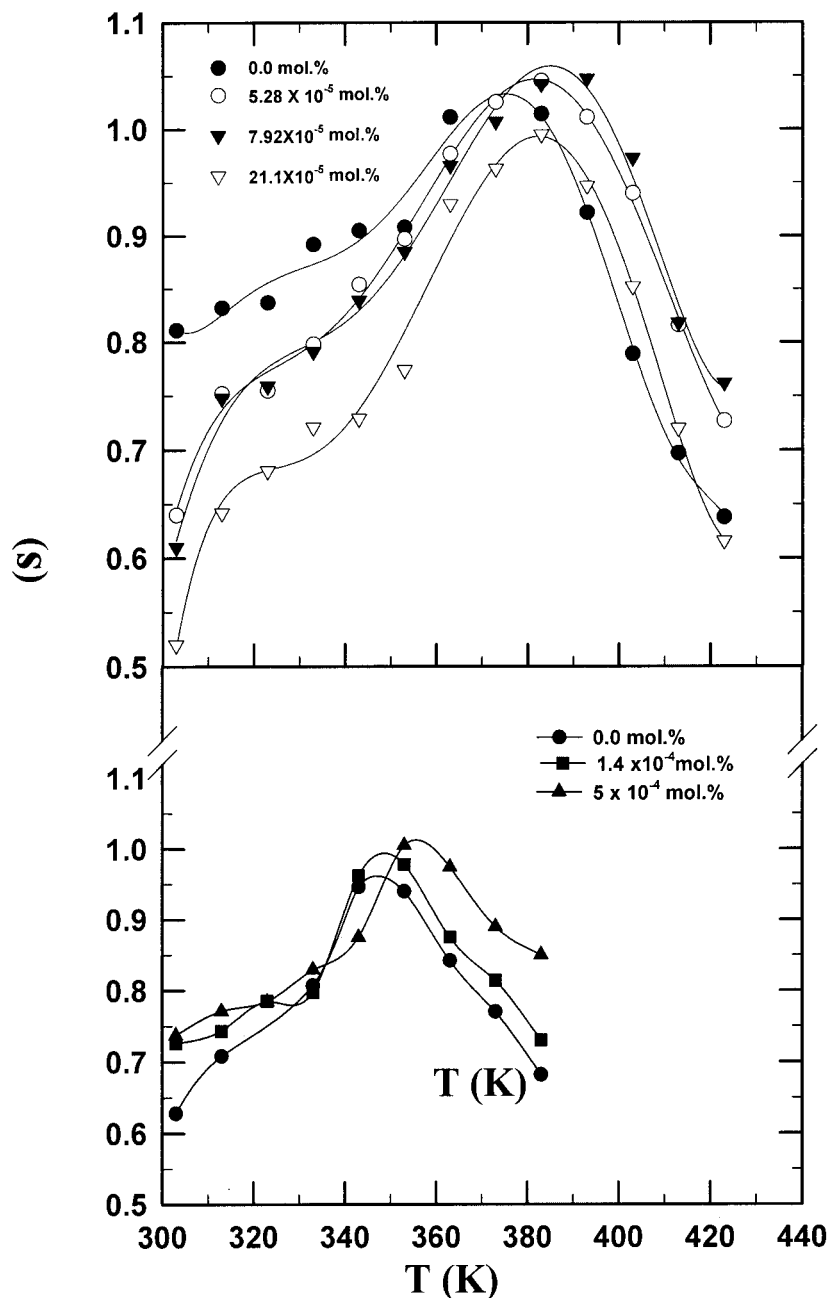
Figure 3 *F* dependence of $\sigma_{tot}(\omega)$ ($\Omega^{-1} \cdot cm^{-1}$) for PMMA/perylene samples prepared by (a) thermal polymerization and (b) solvent casting.

casting methods. The main vibrational transmission peaks observed are listed in Table II, from which it can be seen that

1. The disappearance of the aliphatic C=C, characterizing MMA,[18] indicated the complete polymerization of the samples.
2. The wave number of the main-chain groups of radically polymerized samples appeared at a shorter wave number than that of the solvent-

cast samples, which indicated the stability of radically polymerized samples because the vibrational energies of the main-chain bonds (phonon energies) were lower.
3. The appearance of the C—Cl peak, characterizing chloroform,[18,19] indicated the presence of solvent molecules.
4. The bands at 1600–1475 cm^{-1}, due to the aromatic C=C group of perylene, was not affected by the preparation method, which indicated that

Figure 4 Temperature dependence of the exponent *s* for PMMA and PMMA/perylene samples prepared by (a) radical polymerization and (b) solvent casting.

PMMA is a stable matrix for laser dyes and acts as inert medium.

5. The wave number of the C=O group shifted gradually to higher values by increasing *C* up to 7.92×10^{-5} mol % and decreased due to the aggregation of dye molecules; this anomaly behavior was also observed in the DSC measurements.

The temperature dependence of σ_{dc} for PMMA and PMMA/perylene is shown in Figure 1 in the temperature range 303–433 K. The electrical conductivity was

thermally activated up to a peak value (σ_p) at certain peak temperature (T_p), which was in the same range of the T_g. This activated part could be explained on the basis that PMMA is described as a basic polymer containing an electron-donor atom. According to the Lewis concept,[20,21] the presence of the ester functional group, with the carbonyl oxygen atom being a basic site, leads to PMMA exhibiting electron-donor ability. So, the conduction process occurs by electron jumping (hopping and/or tunneling) between donor atoms and empty sites located in the energy band gap; the conduction mechanism was confirmed in this study
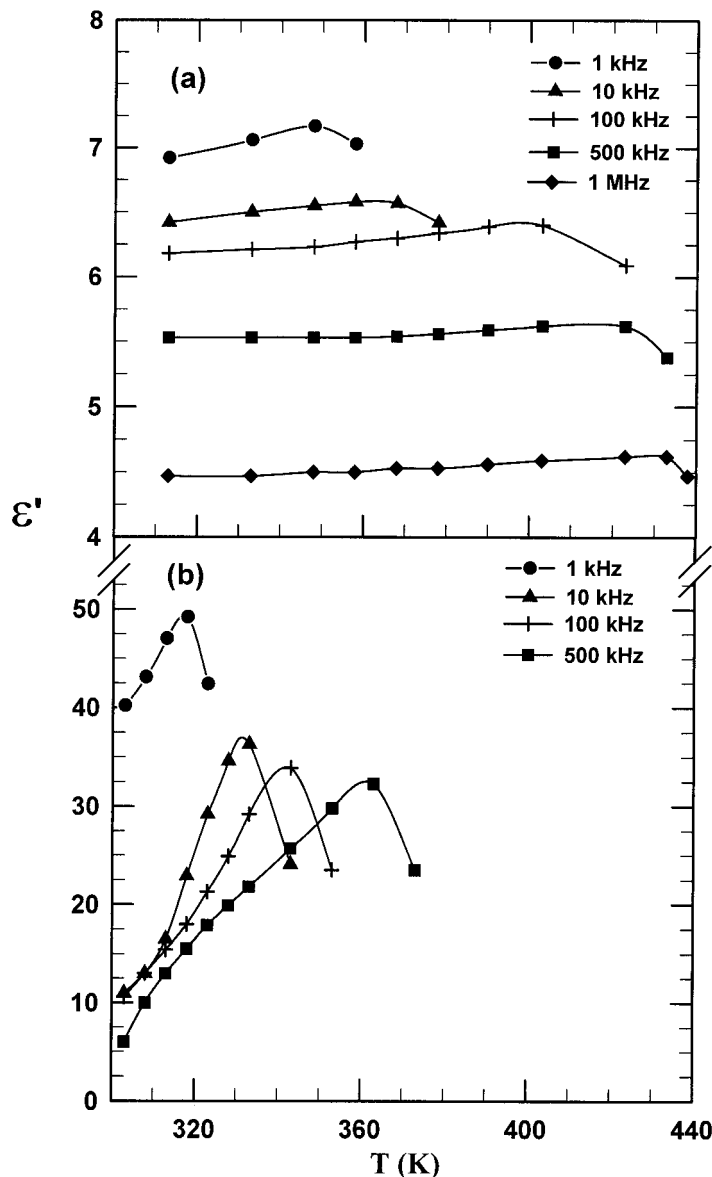
Figure 5 Temperature dependence of ϵ' for pure PMMA samples prepared by (a) radical polymerization and (b) solvent casting.

with σ_{ac} measurements. The thermal activated part of conductivity (σ_{dc}) could be described according to the following Arrhenius relation:[22]

$$\sigma_{dc} = \sigma_o \exp(-\Delta E_{dc}/k_B T) \qquad (1)$$

where σ_o is the temperature-independent parameter, ΔE_{dc} is the activation energy of conduction, k_B is Boltzmann's constant, and T is the temperature. The values of ΔE_{dc}, σ_o, σ_p, and T_p were obtained and are listed in Table III. However, the attenuated part in the conduction process above T_p could be explained by the fact that the conduction process was opposed by the large deformations characteristic of the viscoelastic state permitted by segmental motions.[23] In other

words, beyond the T_g, the electron–phonon coupling became more pronounced, which resulted in charge carrier scattering and, consequently, a reduction in the charge carrier mobility. The σ_{dc} data show that the conductivity values obtained for solvent-cast samples were higher than those of the radically prepared samples. This could be explained because PMMA and chloroform can form acid–base complexes resulting from the interactions between the ester basic group of PMMA and the acid site of hydrogen atom in chloroform.[20,21] So, the confined chloroform molecules surrounding the main chain of the polymer would lead to an increase in the density of localized states, which would enhance the probability of electron jumping.
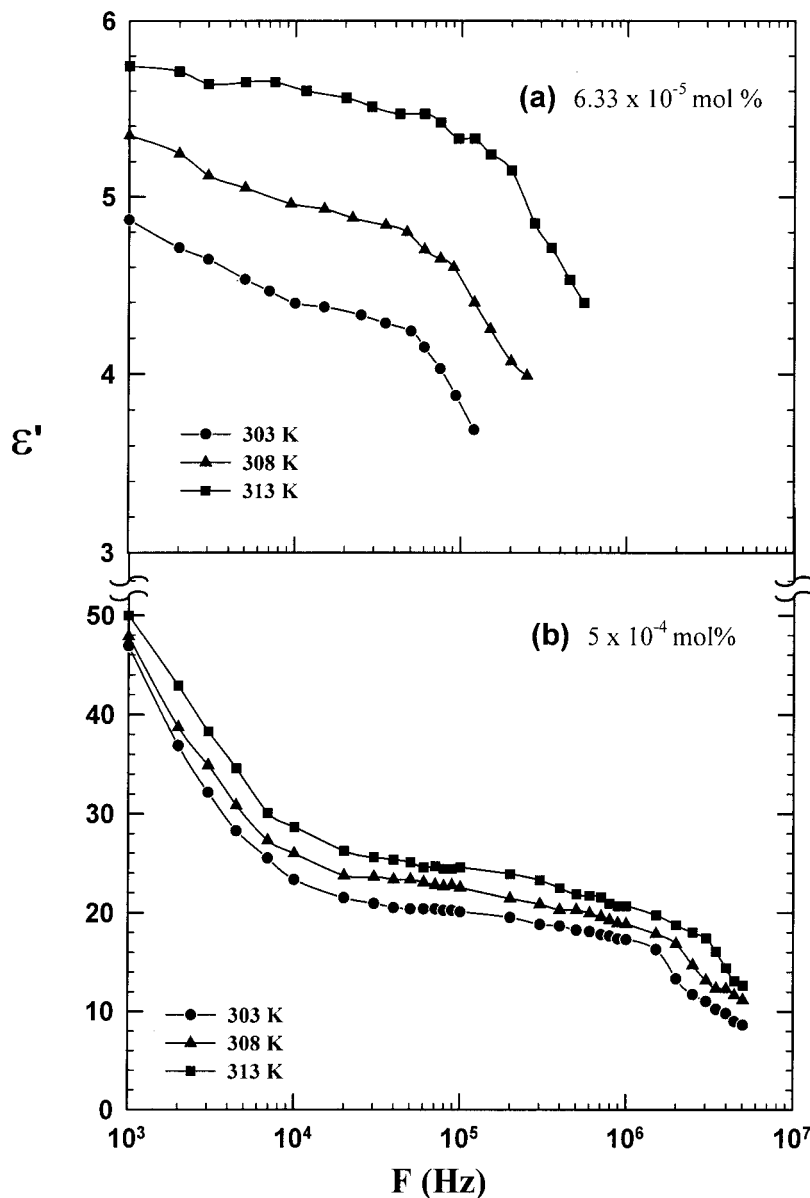
Figure 6 *F* dependence of ϵ' for PMMA/perylene samples prepared by (a) thermal polymerization and (b) solvent casting.

The effect of C on σ_{dc} was found to obey the following empirical relation:

$$\sigma = \sigma^* e^{\pm C/C_o}$$

where σ^* is a concentration-independent parameter equal to σ at $C = 0$ and C_o is a characteristic concentration for perylene in the PMMA matrix. The positive or negative sign of the exponent in the empirical equation depended on the preparation method. In the case of radically prepared samples, σ attenuated by increasing dye content. This was attributed to the increase in charge carrier scattering because the attachment of large-size dye molecules hindered the conduction process. In the case of solvent-cast samples, the

conductivity increased by increasing concentration of the dye. This could be attributed to the confined solvent molecules, which acted as carrier acceptors in the polymer matrix. The temperature dependence of total conductivity [$\sigma_{tot}(\omega)$ ($\Omega^{-1} \cdot cm^{-1}$) (where Ω is the angular frequency equal to $2\pi F$)] was measured in the temperature range 303–433 K and in the F range 10^3 to 5×10^6 Hz; for PMMA and PMMA/perylene samples, a typical plot is shown in Figure 2. $\sigma_{tot}(\omega)$ was thermally activated up to a certain value (σ_p) and then attenuated with increasing temperature in agreement with the behavior observed for σ_{dc} measurements. Also, the values of $\sigma_{tot}(\omega)$ were higher than σ_{dc}, and the activation energies of $\sigma_{tot}(\omega)$ were lower than those of σ_{dc}. This was attributed to the fact that the increase
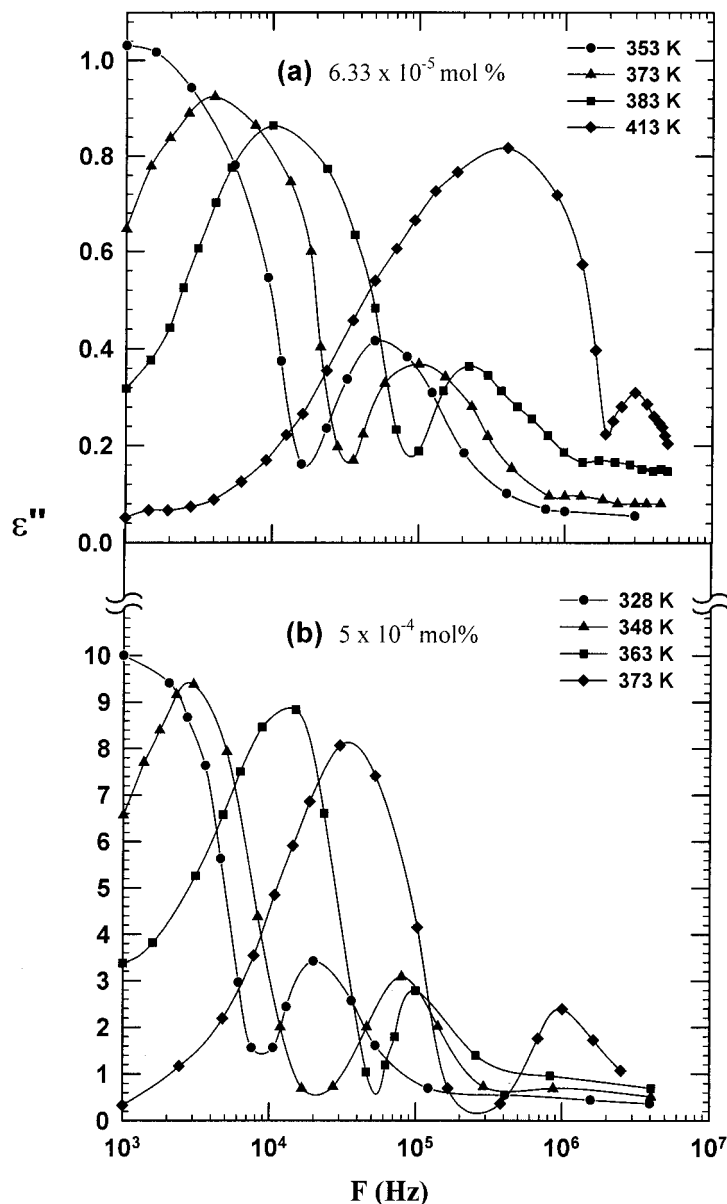
Figure 7 F dependence of ϵ'' for PMMA/perylene samples prepared by (a) thermal polymerization and (b) solvent casting.

of the applied field F enhanced the carrier jumping and, subsequently, the conductivity value. The plots of $\sigma_{tot}(\omega)$ versus F at different fixed temperatures showed similar behavior for all samples; typical plots are given in Figure 3. These showed that although the conductivity was independent on F at in a low-F range, it became F dependent in a higher F range. Therefore, the F dependence of $\sigma_{tot}(\omega)$ obeyed the relation:[24]

$$\sigma_{tot}(\omega) = \sigma_{dc} + A\,\omega^s$$

where A is the F-independent factor, and s is the frequency power. The value of s was calculated and found to depend on the temperature, as shown in

Figure 4. T_p, which was observed for both radically polymerized and solvent-cast samples, was significantly correlated to the T_g's of the samples. At $T < T_p$, s increased with the temperature, which referred to transport the charge carriers, according to the quantum mechanical tunneling model.[25-27] Although at $T > T_p$, s was found to decrease with the temperature, this was attributed to a change occurring in the conduction mechanism, where the correlated barrier hopping model[28] dominates.

To study the polarization in our samples, the temperature dependence of ϵ' at different Fs for all the prepared samples was investigated. Figure 5 shows that the trend of $\epsilon'(T)$ for pure PMMA or PMMA/perylene samples was typical for a polar dielectric.[23,29]
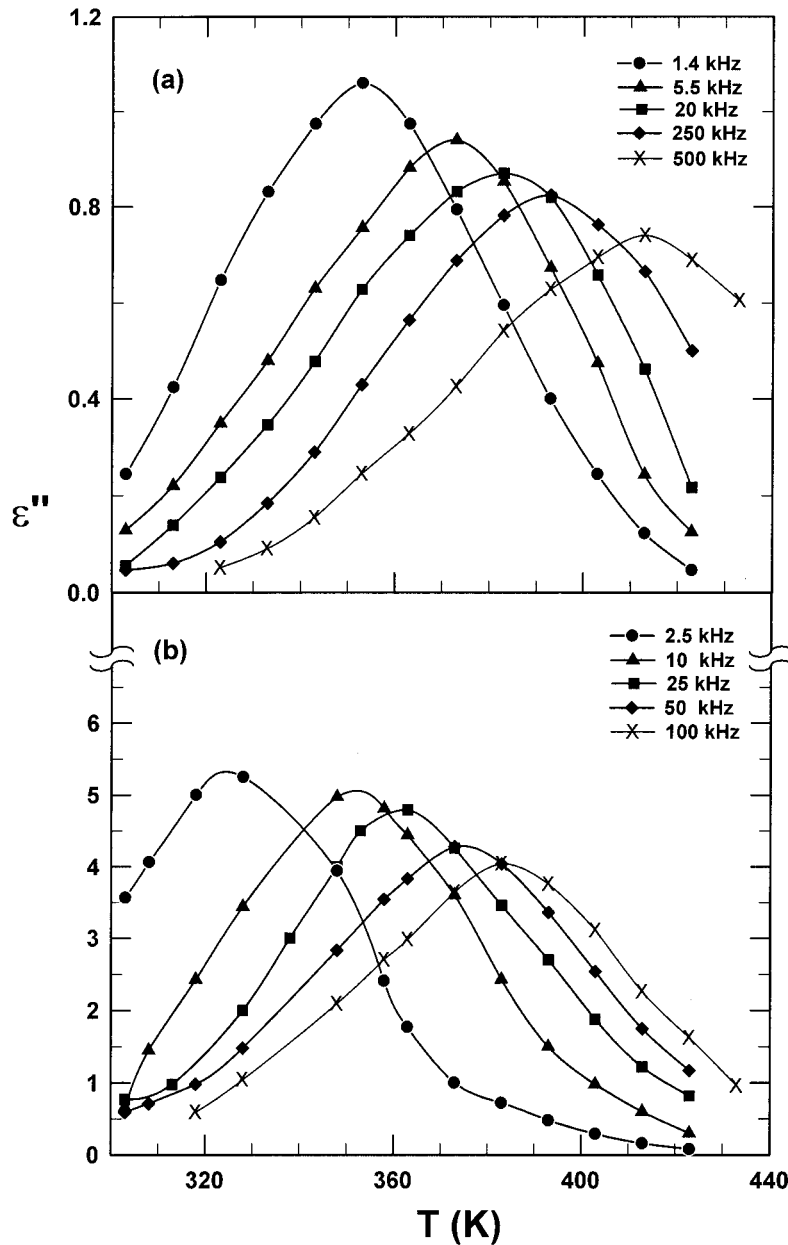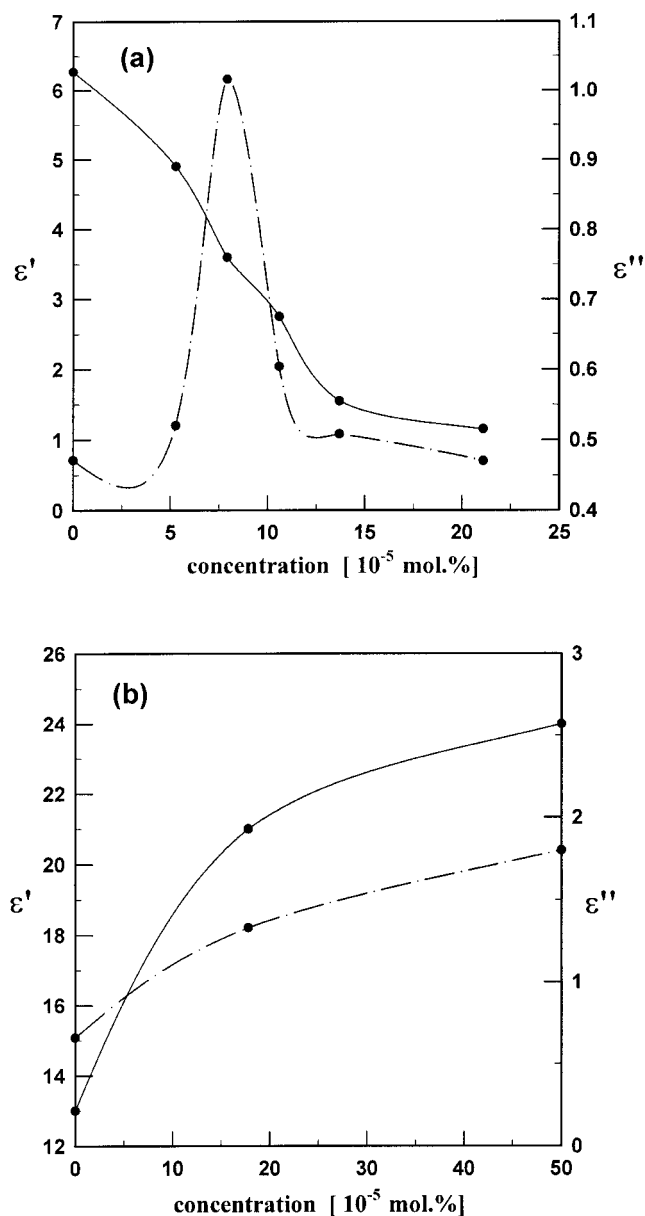
Figure 8 Temperature dependence of ϵ'' for pure PMMA samples prepared by (a) radical polymerization and (b) solvent casting.

It is clear that the solvent-cast samples had higher values of ϵ' and higher temperature gradients $(\partial\epsilon'/\partial T)$ compared with those of radically polymerized samples, which reflected the strong polar nature of the solvent-cast samples.

The F dependence of ϵ' for PMMA/perylene samples is shown in Figure 6 as a representative figure. For solvent-cast polymers, a reduction in ϵ' by increasing F occurred in three stages, whereas it occurred in two stages for the radically polymerized sample. For solvent-cast samples, in the first stage, high values of ϵ' could be attributed to the interfacial polarization effects due to discontinuity of the dielectric medium as a result of the presence of solvent.[29,30] In the second

stage, attenuation of ϵ' by increasing F for both cases (radically polymerized and solvent-cast samples) referred to the orientation polarization of the main-chain segments. In the third stage, decreased in ϵ' in the high-F range could be attributed to the lag of dipole orientational by increasing F.

Figure 7 shows the F dependence of ϵ'' at a fixed temperature for PMMA/perylene samples as a representative figure. Two peaks in the dispersion relation were observed; the first appeared at low F and was associated with the orientation of chain sections consisting of large number of monomeric units (segments).[31] The second peak appeared in the high-F range and was associated with the rotation of the ester

Figure 9 Effect of perylene concentration on (—) ϵ' and (– – –) ϵ'' for PMMA/perylene samples prepared by (a) radical polymerization and (b) solvent casting at 300 K and 500 Hz.

side group about the C—C bond, which linked it to the main chain. It was clear that $\epsilon''_{max}(F)$ was much higher for dipole segmental loss than for dipole group loss. This could be attributed to the fact that the ester side groups were more mobile kinetic units than the acrylate group loss.

The temperature dependence of ϵ'' at different Fs for PMMA samples is shown in Figure 8 as a typical plot for all of the investigated samples. Only a single peak in the $\epsilon''(T)$ curve was observed; its position depended on F. This peak could be attributed to the fact that the β-process (due to the rotation of acrylate groups around the C—C bonds, which linked them to the

main chain[32]) was much broader than α-process (due to rotation of lateral side groups around the main-chain axis[33]). α- and β-processes are only well separated below 1 kHz.[25,26] So, $\epsilon''_{max}(T)$ could be ascribed to an $\alpha\beta$-process in which the side groups cooperated with the backbone motions in a micro-Brownian motion, which had properties similar to a pure α-process.[34–36]

Figure 9 shows the effect of C on both ϵ' and ϵ'' for radically polymerized and solvent-cast samples. ϵ' decreased by increasing C for radically prepared samples, whereas it increased by increasing C for solvent-cast samples. These results were in agreement with the conductivity measurements because the conductivity of a real dielectric is directly related to the dielectric permittivity.[37] Also, ϵ'' increased by increasing C for low Cs in radically prepared samples and in all of the solvent-cast samples. The increase in ϵ'' could be explained by the following:

1. The nonpolar nature of the dye molecules reduced the polarization process and caused the shift of the dipole segmental and dipole group relaxations to lower Fs.
2. The large size and bulky nature of the dye molecules increased the steric hindrance to the motion of the dipoles (for the radically prepared polymer). For solvent-cast samples, this effect caused the trapping of solvent between chains and, subsequently, increased the intermolecular interactions discussed previously.
3. For Cs higher than 7.92×10^{-5} mol % in the radically polymerized samples, these factors were opposed by the increase of the free volume and chain mobility caused by the aggregation of dye molecules.

The relaxation times (τs) of the orientational moment in the α and β relaxation were calculated with the following relation:[38]

$$\omega_{max} \, \tau = 2 \, \pi \, f_{max} \, \tau = 1$$

where ω_{max} is the angular field frequency at which ϵ'' passes through a maximum. The obtained values of τ (τ_1 and τ_2) corresponding to the dipole segment and dipole group losses are plotted against C as shown in Figure 10. The τs for radically polymerized samples increased by increasing C, passed through a maximum at a concentration of 7.92×10^{-5} mol %, and then dropped, although they increased by increasing C for solvent-cast samples.

τ ($\tau = 1/\omega_{max}$), which showed an attenuation with increasing temperature, obeyed an exponential relation of the form:[39,40]

$$\tau = (h/kT) \, \exp \, (\Delta G/RT)$$
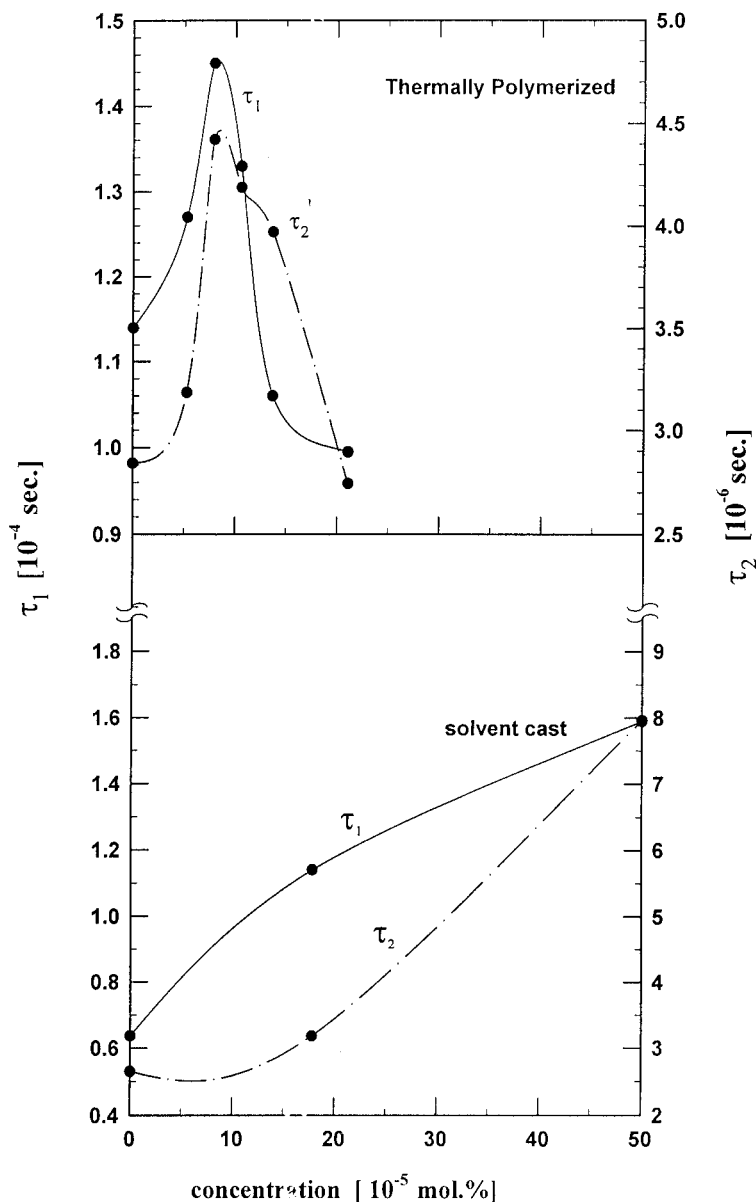
Figure 10 Effect of perylene concentration on the τs (τ_1 and τ_2) corresponding to the dipole segmental and dipole group relaxations in PMMA/perylene samples at 353 K for radically polymerized samples and at 328 K for solvent-cast samples.

$$\Delta G = RT \ln (kT\tau/h)$$

where ΔG is the free energy of activation for dipole relaxation, which is related to the enthalpy (ΔH) and entropy (ΔS) of activation by the relation:

$$\Delta G = \Delta H - T\Delta S$$

TABLE IV
Calculated Values of ΔG, ΔH, and ΔS Corresponding to the Dipole Segmental and Dipole Group Losses for Radically Polymerized PMMA/Perylene Samples

C (mol %)	ΔG (353 K; kJ/mol)	ΔH (kJ/mol)	ΔS (JK^{-1}/mol)
0.00	65.69 (54.87)	126.63 (85.55)	173.00 (86.91)
6.33×10^{-5}	66.68 (55.20)	124.50 (84.45)	164.00 (82.86)
7.92×10^{-5}	66.40 (56.16)	109.10 (66.32)	121.00 (28.78)
1.06×10^{-4}	66.14 (56.00)	117.46 (68.28)	145.00 (34.79)
1.37×10^{-4}	65.50 (55.85)	129.33 (81.46)	181.00 (72.55)
2.11×10^{-4}	65.30 (54.75)	137.85 (88.85)	206.00 (96.60)

Dipole group loss values appear in parentheses.

The values of ΔG, ΔH, and ΔS were obtained for all of the investigated samples and are listed in Table IV. For the radically prepared samples, both ΔH and ΔS showed minimum values at a concentration of 7.92 $\times 10^{-5}$ mol % and then increased by increasing C. This could be understood on the basis that the dye filled the

TABLE V
Calculated Values of ΔG, ΔH, and ΔS Corresponding to the Dipole Segmental and Dipole Group Losses for Solvent-Cast PMMA/Perylene Samples

C (mol %)	ΔG (328K; kJ/mol)	ΔH (kJ/mol)	ΔS (JK^{-1}/mol)
0.00	50.59 (59.26)	82.08 (66.32)	96.01 (21.52)
1.78×10^{-4}	51.09 (60.84)	77.32 (73.04)	79.97 (37.20)
5.00×10^{-4}	53.59 (61.76)	79.69 (79.38)	79.57 (53.72)

Dipole group loss values appear in parentheses.

voids in the polymer structure (for low content). For higher C, the dye played an important role for restricting the rearrangement of the polymer structure (see DSC results). However, Table V shows that ΔG, ΔH, and ΔS, for solvent-cast samples, increased by increasing C. This indicated that the confined solvent and dye had an effective influence on the thermodynamic parameters. The slight change observed in the thermodynamic parameters indicated that the mechanism of relaxation was the same for all samples. It meant that the dye did not contribute to the relaxation process due to the nonpolar nature of the perylene molecule, as mentioned in the FTIR results.

CONCLUSIONS

From the obtained results and discussions, we concluded the following:

1. The perylene doping of radically polymerized PMMA did not illustrate a remarkable change in the activation energy values, whereas an increase in the insulation property was recorded by increasing C, which meant less fluorescence quenching and, consequently, more optical efficiency of the solar concentrator.[8]
2. The perylene doping of solvent-cast samples showed a decrease in T_g and an increase in both electrical conduction and ϵ' due to the presence of confined solvent molecules. This reflected the instability of the optical properties for the samples prepared by this procedure for outdoor applications.[9]

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