



Assessment of optical and thermal properties of polyimide/metal oxide composites for photovoltaic uses

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Abstract. Novel polyimide/metal oxide composites are theoretically designed for analysis of their applicative potential as covers for photovoltaic devices. The polymer matrix is made of a common sulphone-containing aromatic diamine combined with certain cycloaliphatic dianhydrides, rendering new polymer structures. The polymer conformations and chain polarizability are affected by the matrix structure and filler loading. Optical and thermal properties of the proposed materials are described by the approach of connectivity index and group contribution method. The dianhydride moiety influenced the magnitude of refractive index, revealing that its bulkiness, asymmetry and flexibility lead to larger n -values, hence lowering the optical losses at the interface with ZnO layer from the photovoltaic cell. The presence of TiO₂ filler in these polyimides upgrades matching of the refractive and coefficient of thermal expansion with those of the neighbouring ZnO layer, improving the device reliability.

Keywords. Polyimide composites; molecular modelling; refractivity; thermal expansion coefficient; solar cells.

1. Introduction

A key element for photovoltaic panels is represented by the outer cover that directs the solar radiations towards the active part of the device [1]. For advanced operability in terms of conversion efficiency of light into electricity, a larger amount of light must enter in the solar cell. As a result, many researchers made considerable efforts and tried to substitute the classical cover glass with polymers owing to their easy processing and adequate flexibility [2]. There is a variety of polymer structures that are fitting these two aspects [3], but deeper requirements must be fulfilled, such as optical clarity, heat resistance, high refraction and good adhesion. Such features are often found in polyimides, which are an important category of engineering thermoplastics [4–9]. Besides their elevated temperature stability, these materials also display notable mechanical toughness [10], low coefficient of thermal expansion [11] and chemical resistance [12]. Their properties are dictated by the chemical structure of the monomers used for synthesis. Many diamines and dianhydrides are commercially accessible for polyimide preparation and might be selected to tailor the final characteristics of the material [13,14]. For attaining the targeted compromise between large transparency, raised thermo-mechanical resistance, high refractive index and good adhesion, a careful strategy must be

formulated for the chemical design of the polymer. A solution would reside in combining the main chain cycloaliphatic moieties, along with flexible bonds and/or bulky side groups, which are diminishing the co-planarity and symmetry of the macromolecules and would generate conformations that enable a balanced chain packing and polarizability [15]. This is expected to improve the optical properties without sacrificing thermal resistance [16,17].

Concerning the optical properties, aside from elevated transmittance, it is necessary to match the refractive characteristics of the polyimide with those of the neighbouring sheet of conductive oxide [18]. This is of paramount relevance, in order to avoid unwanted reflections of the sun's emerging radiations, when they are passing from the outer cover of the solar cell to the next medium. A general rule to enhance the refractive properties of a polyimide is to incorporate in its structure elements characterized by large molar refraction and small molar volumes [19]. There is a diversity of atoms or units, like sulphur, oxygen, phosphorus, heavy halogens and π -conjugated rings that are proper candidates for this purpose [19]. Furthermore, the refractive-related standards of polyimides can be additionally improved by the addition of inorganic fillers, such as ferrous sulphide, tellurium dioxide, zinc oxide, titanium dioxide, zirconium oxide, aluminium nitride, lead(II) sulphide, selenium dioxide and zinc sulphide [19]. By this approach,

hybrid polyimide composites, having both high transmittance and high refractive index, are achieved.

Another important aspect arises from the thermal features of the shielding material. The glass transition temperature (T_g) represents a parameter that reveals the point of enhanced molecular mobility that results in converting the polymer from hard to soft. A higher T_g means that the polymer displays better thermal resistance, which is compatible with the pursued applications [20]. When interfacing the shielding material with the conductive oxide layer, it is important to match also the thermal expansion coefficient (CTE) to keep proper stability among the layers of the photovoltaic cell.

In previous works [21–23], we have reported new polyimide-based covers for solar cells that contain chalcogen atoms either in the polymer or in the filler structure. The results revealed that the refractive index of the obtained materials is enhanced and by this the optical losses are smaller and positively impact the conversion efficiency of the photovoltaic system.

In this study, an assessment of the synergism of chalcogen atoms and inorganic fillers on the polarizability and refractivity of the polyimide composites is performed. Here, we chose to investigate the influence of several dianhydrides because it seems that this type of monomer has a greater effect on the polyimide chemical and physical properties than the diamine [15]. The explanation is arising from the fact that higher electrophilicity of the dianhydride (reflected by electron affinity) enhances the capacity of reaction with the diamines [24]. The reactivity of the diamines is less facile to be correlated with their nucleophilic character. In any case, their reaction rates with dianhydride are improved by larger ionization potential [24]. Hence, the rationale of this study is to analyse how subtle variations in the structure of the dianhydride would render the desired effect on the high refractivity and thermal stability of the proposed polymer materials. Molecular modelling of the imide-based polymer loaded with several sorts of particles is accomplished. Employing the connectivity index approach [25], the refractive index is evaluated. Also, the refraction matching with that of a conductive oxide used in solar cell fabrication is discussed in terms of optical losses. The results of this theoretical study could deliver fruitful insights for designing shielding covers for photovoltaics with better light trapping and therefore advanced performance in terms of conversion efficiency.

2. Experimental

The polyimide structures are envisioned by combining monomers of commercial nature, namely: 10H-dibenzo[b,e]thiopyran-3,7-diamine 5,5-dioxide (10H-TP), dicyclohexyl-3,4,3',4'-tetracarboxylic dianhydride (DCHMDA), tetrahydrofuran-2,3,4,5-tetracarboxylic dianhydride (THTDA), 4-(2,5-dioxotetrahydrofuran-3-yl)-

1,2,3,4-tetrahydronaphthalene-1,2-dicarboxylic anhydride (TDA) and bicyclo[2.2.2]octane-2,3,5,6-tetracarboxylic 2,3:5,6-dianhydride (CHTDA). The theoretical design of these structures aims to predetermine the balance between chain packing and molecular polarizability as a function of the selected diamine and dianhydride structures. Scheme 1 illustrates the polyimides analysed in this study.

These polymers are ideally reinforced with several particles of titanium dioxide, which is a compound known for its high refractive index.

Molecular modelling involved computations with the HyperChem demo version, which enables attainment of the polymer optimized geometries at the lowest energy conditions with or without the filler particles.

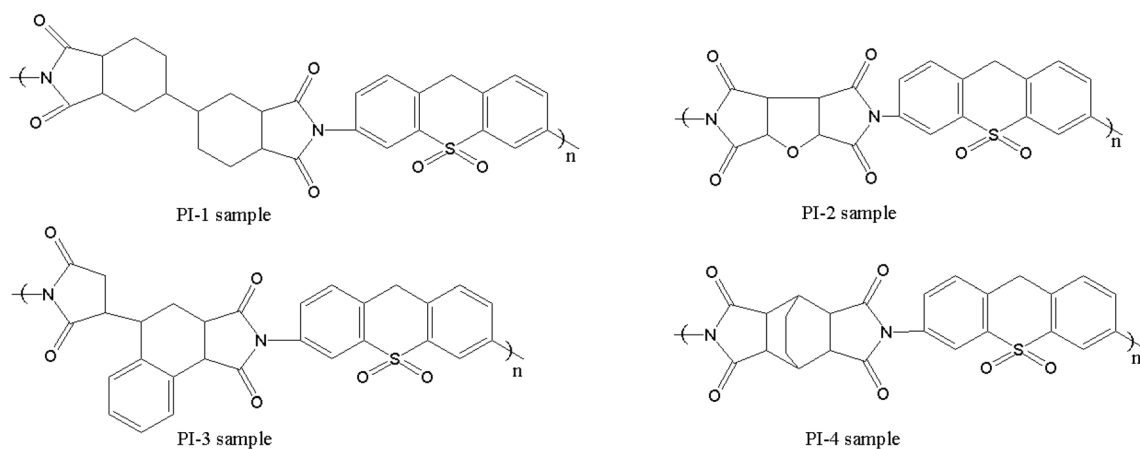
3. Results and discussion

3.1 Molecular modelling of the polyimide composites

The chemical design of the polymer structures from this study involves the use of a common diamine that contains chalcogen atoms, namely 10H-dibenzo[b,e]thiopyran-3,7-diamine 5,5-dioxide (10H-TP). In order to keep the pursued balance between the optical and thermal properties, this diamine was combined with several cycloaliphatic dianhydrides that differ in terms of rigidity and symmetry: dicyclohexyl-3,4,3',4'-tetracarboxylic dianhydride (DCHMDA), bicyclo[2.2.2]octane-2,3,5,6-tetracarboxylic 2,3:5,6-dianhydride (CHTDA), 4-(2,5-dioxotetrahydrofuran-3-yl)-1,2,3,4-tetrahydronaphthalene-1,2-dicarboxylic anhydride (TDA) and tetrahydrofuran-2,3,4,5-tetracarboxylic dianhydride (THTDA). The envisioned polyimides were computed with the help of HyperChem to achieve the conformation at the lowest energy conditions. Figure 1 displays the geometry optimization at minimum energy for five structural units of each polymer structure: PI-1 containing DCHMDA/10H-TP, PI-2 containing THTDA/10H-TP, PI-3 containing TDA/10H-TP, and PI-4 containing CHTDA/10H-TP.

The conformational features of the imide-type polymers are substantially impacting their optical and thermal properties. The chain flexibility has its origins in the peculiarities of the chemical structure. The degree of freedom, which denotes the overall shape of the macromolecule, is provided by the torsion angle produced by linkages among imide rings and phenyls and/or among the aromatic rings connected through the sulphone group. Samples PI-1 and PI-3 contain a single bond among the aliphatic rings from the dianhydride moiety, which enhance the overall degree of freedom and more twisted/bent chain conformation. In contrast to these, the polymers PI-2 and PI-4 contain rigid and symmetrical dianhydride moieties that enhance the chain rigidity, reflected in a more stretched conformation.

The selected polymer structures are designed from the common 10H-TP diamine, which in combination with the



Scheme 1. The chemical structures of the analysed imide-type polymers.

chosen dianhydrides of distinct structures might lead the imide-type polymers with tuned properties. Since both dianhydride electrophilicity (E_{Af}) and diamine ionization energy are affecting the monomers reactivity, these parameters are additionally extracted from simulation of the monomer structures based on HOMO and LUMO computed values. The obtained data are summarized in table 1.

It is known that the nucleophilic attack susceptibility is favoured by a bigger electrophilicity of the dianhydride unit, whose reactivity is connected to its E_A [24,26]. The evaluated values of this parameter are around 1, similarly to other aliphatic dianhydrides and this aspect is adequate for PI preparation [26].

The computations were continued with the analysis of the conformational aspects in the presence of six particles of an inorganic metal oxide (TiO_2), widely recognized for its high refractive index ($n = 2.7$) [19]. The modelling data are comprised in figure 2. It can be remarked that the polyimide conformation is slightly modified by the presence of the inorganic particles, which are tending to position themselves in the spaces created by chain bending or torsion.

3.2 Evaluation of the refractive index of polyimides and their composites

Theoretical analysis of the refractive index is possible by employing the theory elaborated by Bicerano [25]. In his work, a formula was developed that accounts on several structural parameters that are comprised in the zeroth-order (atomic) and first-order (bond) connectivity indices, as shown in equation (1):

$$n = 1.885312 + \frac{0.024558}{N} 17 \cdot^0 \chi^v - 20 \cdot^0 \chi - 12 \cdot^1 \chi^v - 9 \cdot N_{rot} + N_{ref} \quad (1)$$

where n is the refractive index, N the number of non-hydrogen atoms, N_{rot} is parameter related to the backbone

degrees of freedom, N_{ref} is the parameter linked to the number of F, S, Si atoms, fused rings, $^0 \chi$ and $^0 \chi^v$ are the zeroth-order (atomic) indices, while $^1 \chi$ and $^1 \chi^v$ are the first-order (bond) connectivity indices.

Table 2 contains the attained data concerning the connectivity indices and structural parameters involved in the calculation of the refractive index. It seems that besides the balance between molar refraction and small molar volumes, the dianhydride features (size, symmetry, polarity) affect the magnitude of the refraction properties of each sample.

As a general rule, the refractive index of polymers is affected by the polarizability (α) or molar refraction (R_m) of the incorporated atoms and the corresponding molar volume (V_m) of the polymer backbone [19]. Therefore, to augment the refractive index it is necessary to attain a proper balance between these two parameters, namely a high α/V_m or R_m/V_m ratio. This means that in the respective material there are numerous polarizable (high refractivity) groups in a unit volume, so that the refractive index is improved. The data regarding the polarizability was extracted from molecular modelling and the results are included in table 1. It is noted that the polarizability ranges as follows: PI-1 > PI-3 > PI-4 > PI-2. Both molar refraction and molar volume are assessed by means of group contribution theory, as indicated in the work of Groh and Zimmermann [27]. According to figure 3, the type of dianhydride moiety affects the magnitude of R_m and V_m . The molar refraction assessed by group theory ranges similarly to the polarizability values obtained via simulations. Moreover, the highest values for the R_m/V_m ratio are noticed to be larger for PI-3 and PI-1 samples. In the case of PI-4, it is remarked that R_m/V_m ratio is the lowest which is reflected in a smaller refractive index. The utilization of dianhydrides with high symmetry, like those from PI-2 and PI-4 structures, determines the cancellation of the dipoles and hence reduces the polarizability and refractive index. The highest refractive index is noted for PI-3, which contains an aromatic pendant ring in the dianhydride structure that augments the overall polarizability.

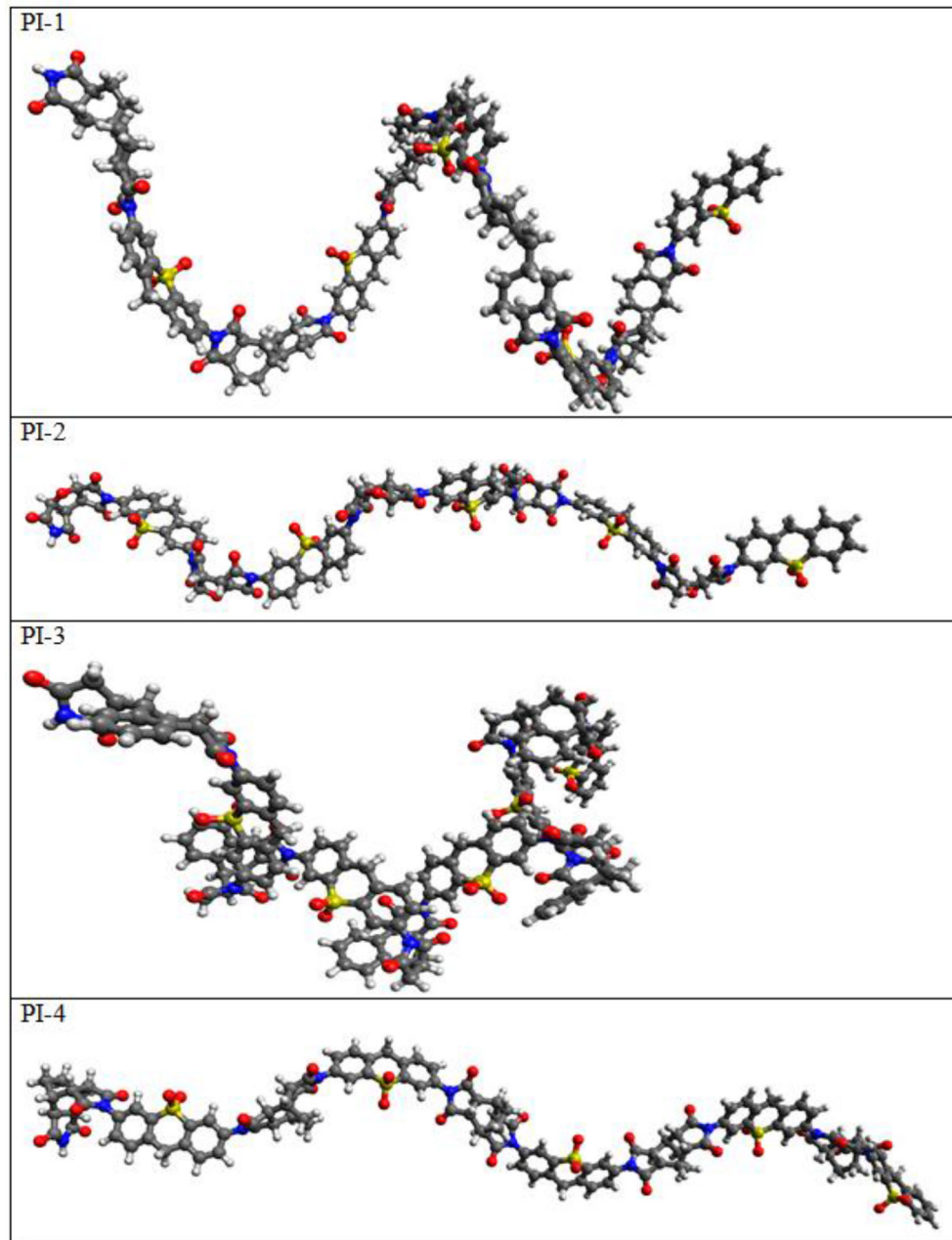


Figure 1. Three-dimensional images of the optimized structures in minimum energy for each investigated polyimide containing 5 structural units (blue colour, nitrogen; red, oxygen; light gray, hydrogen; dark gray, carbon; yellow, sulphur).

The refractive index of the composite samples is considered to be an additive function of the materials used (polymer and reinforcing agent) and the composition of the system, as shown in equation (2):

$$n_c = n_{PI}w_{PI} + n_f w_f \quad (2)$$

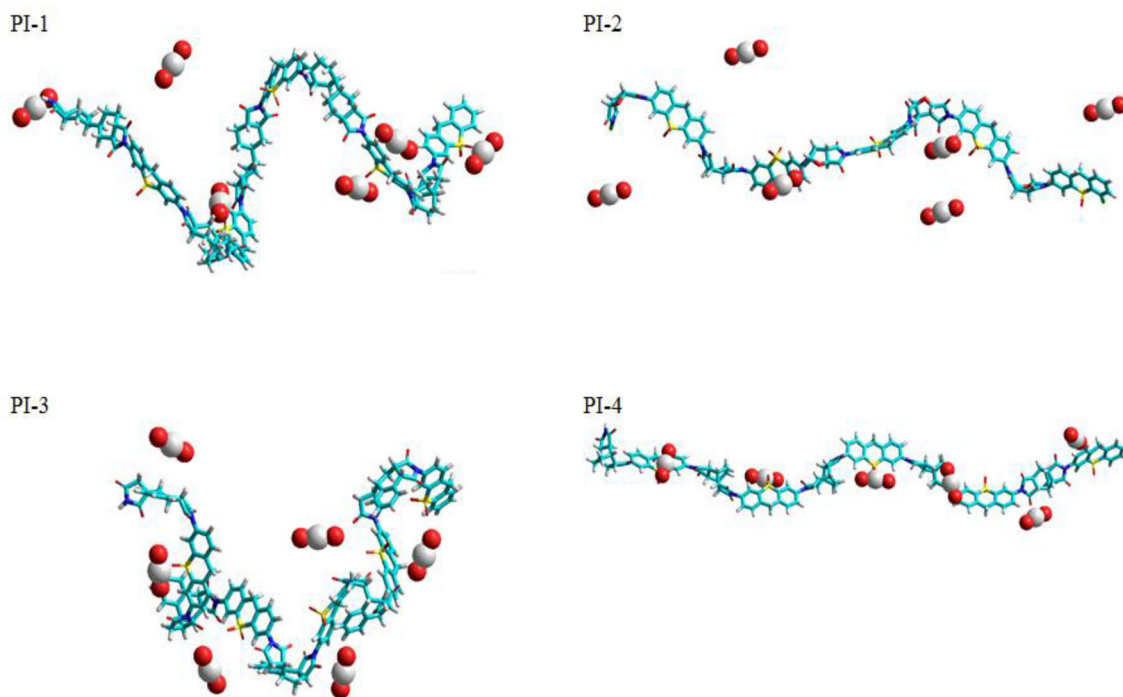
where n_c is the refractive index of the polymer composite, n_{PI} is the refractive index of the polymer matrix, w_{PI} is the fraction of the polymer in the composite, n_f is the refractive index of the filler and w_f is the fraction of the filler in the composite.

Figure 4 illustrates the variation of the refractive index with the addition of the metal oxide filler in all studied polymers. The presence of TiO_2 augments the refractive index of the composites in regard to the neat polyimides, regardless of the reinforcement percent. The largest values of n are noted for the system having PI-3 as the matrix, while the lowest values are noted for the PI-4 composite.

The refractive index properties are paramount for ensuring the propagation of a large amount of light inside the photovoltaic cell. The discrepancy between the

Table 1. Reactivity descriptors (eV) based on molecular orbital energies of the monomers used for PI design.

Sample	HOMO	LUMO	E_{Af}	IE
Dianhydride monomer used in PI design				
DCHMDA in PI-1	-11.5505	-0.9209	0.9209	
THTDA in PI-2	-12.0828	-1.1090	1.1090	
TDA in PI-3	-10.1282	-1.0331	1.0331	
CHTDA in PI-4	-11.8560	-1.0754	1.0754	
Diamine monomer used in PI design				
10H-TP	-8.803312	19.8427		8.803312

**Figure 2.** Three-dimensional images of the optimized structures of the polyimides (containing 5 structural units) in the presence of six particles of TiO_2 drawn as balls (blue colour, nitrogen; red, oxygen; light gray, hydrogen; cyan, carbon; yellow, sulphur).**Table 2.** The values of zeroth-order (atomic) and first-order (bond) connectivity indices, N_{rot} , N_{ref} , N , polarizability (\AA), and refractive index for all examined imide-type polymers.

Sample	${}^0\chi^v$	${}^0\chi$	${}^1\chi$	${}^1\chi^v$	N_{rot}	N_{ref}	N	α	n
PI-1	20.704	26.085	16.569	13.484	8	45	38	51.61	1.658
PI-2	15.845	21.394	14.452	9.856	7	45	31	38.33	1.652
PI-3	20.161	26.214	17.919	12.746	8	63	38	51.03	1.663
PI-4	18.006	30.602	15.952	11.701	7	45	34	44.26	1.576

refractive characteristics of the shielding layer and neighbouring conductive oxide must be shrunk to avoid reflection losses at the interface. For instance, conductive metal oxides exhibit a much higher refractive index (e.g., ZnO has $n = 1.952$) [28] than that of many polymers. Therefore, a

refractive index above 1.65 for the shielding layer is desirable for solar cells applications to reduce the optical losses. The results concerning the reflection and transmission at normal incidence at the composite sample/ZnO interface are depicted in figure 5a and b. These data were

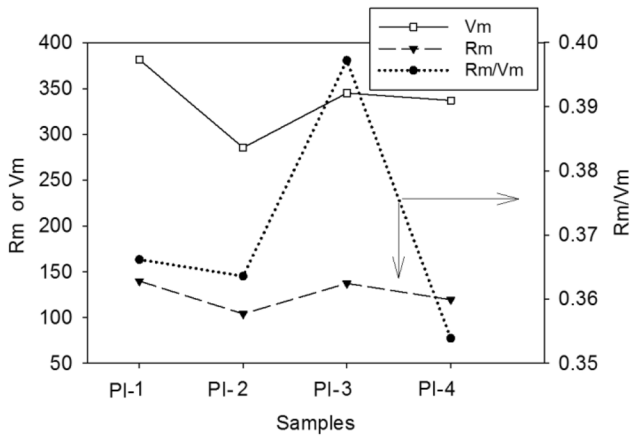


Figure 3. The variation of the molar refraction, molar volume and R_m/V_m ratio for each examined PI structure.

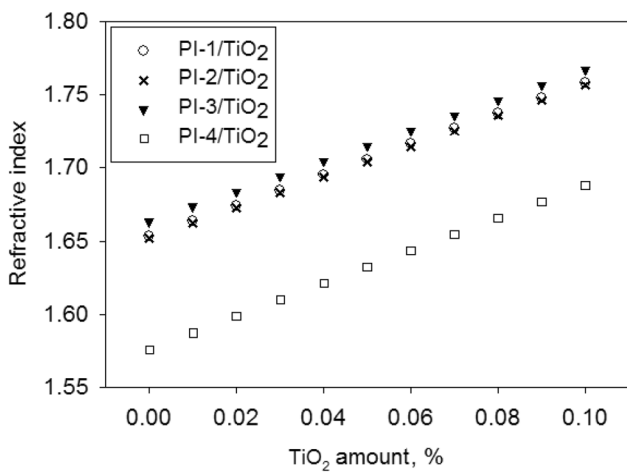


Figure 4. The variation of the refractive index with the metal oxide amount for the studied semi-aliphatic polyimides.

obtained using Fresnel relations for transmission and reflection, expressed for the situation of normal incidence [29,30]. As observed, the differences among optical properties of the polyimide-based shielding layer and ZnO are affecting the magnitude of the reflected and transmitted light at the interface. The PI-4/TiO₂ composite, which presents the lowest refractive index in regard to the metal oxide, renders the highest reflection losses allowing a smaller amount of light to pass further in the photovoltaic cell. Conversely, better matching of the refraction features of ZnO is accomplished for the PI-3/TiO₂ system. In this situation, the light is less reflected in the incident medium and more of it is transmitted towards the active zone of the solar device, increasing its conversion efficiency.

3.3 Evaluation of the coefficient of the thermal expansion

The coefficient of thermal expansion of the polyimide matrix can be determined with the Bicerano method [25], according to equation (3):

$$\alpha = \frac{1}{298 + 9.47 \cdot T_g} \quad (3)$$

where α_v is the volumetric coefficient of thermal expansion and T_g is the glass transition temperature.

The glass transition temperature (T_g) can be estimated according to Bicerano theory [25], as depicted in equation (4):

$$T_g = 351 + 5.63\delta + 31.68 \frac{N_{Tg}}{N} \quad (4)$$

where δ is the solubility parameter, N_{Tg} the structural parameter and N the number of non-hydrogen atoms.

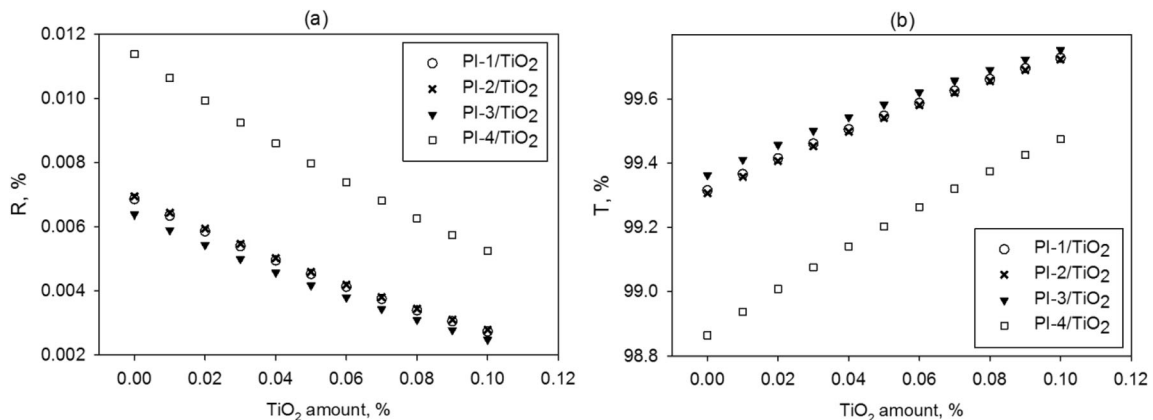


Figure 5. (a) Reflected and (b) transmitted light at the polyimide composite/ZnO interface in the solar cell.

Table 3. The values of the molar volume, cohesion energy, solubility parameter, N_{T_g} , glass transition temperature and volumetric coefficient of thermal expansion of all studied polyimide matrix.

Sample	V (cc mol ⁻¹)	E_{coh} (J mol ⁻¹)	δ (J cc ⁻¹) ^{0.5}	N_{T_g}	T_g (°C)	α_v (1/K)
PI-1	430.274	1.744×10^5	20.461	2	467	1.369×10^{-4}
PI-2	318.655	1.712×10^5	23.179	79	562	1.219×10^{-4}
PI-3	401.795	2.025×10^5	22.450	23	496	1.319×10^{-4}
PI-4	386.882	1.031×10^5	16.325	134	568	1.212×10^{-4}

The solubility parameter (δ), involved in T_g assessment, can be calculated based on cohesion energy and molar volume, as shown in equation (5) proposed by Bicerano [25]:

$$\delta = \sqrt{\frac{E_{\text{coh}}}{V}} \quad (5)$$

where E_{coh} is the cohesion energy and V the molar volume.

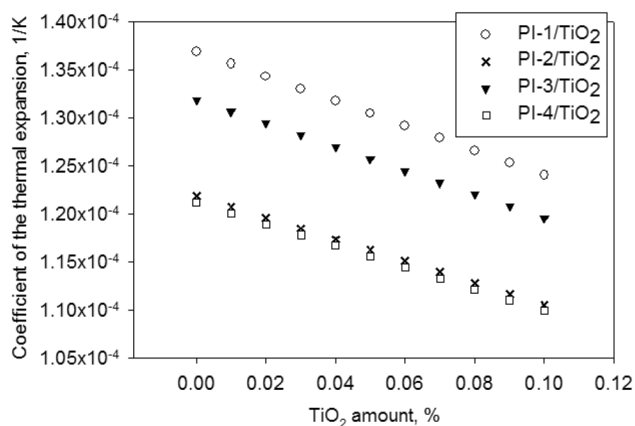
The calculations of the parameters used for the estimation of the coefficient of thermal expansion are listed in table 3. The chemical structure of the polyimide is impacting the thermal properties. The highest T_g is noticed for PI-4 and PI-2 structures, which display a larger rigidity and stronger intermolecular interactions. The obtained values of the coefficient thermal expansion are highest for PI-1 and PI-3 and smallest for PI-4 and PI-2.

In the case of the polyimide/metal oxide composites, the evaluation of the volumetric CTE can be assumed using equation (6):

$$\alpha_c = \alpha_{\text{PI}}w_{\text{PI}} + \alpha_f w_f \quad (6)$$

where α_v is the volumetric CTE of the composite, α_{PI} the volumetric CTE of the polymer, w_{PI} the fraction of the polymer in the composite and w_f the fraction of the filler in the composite.

The data regarding the variation of the sample α_v against the metal oxide loading level are illustrated in figure 6 for all composite systems.

**Figure 6.** The variation of the volumetric CTE with the metal oxide amount inserted in the studied semi-aliphatic polyimides.

It can be remarked that after the addition of TiO₂ in the polyimide, the coefficient of the thermal expansion is decreasing, regardless of the chemical structure of the matrix. Knowing that ZnO material presents a value of $1.5 \times 10^{-5} \text{ K}^{-1}$ [31], it is preferable to use polymer shielding layers with appropriate thermal features. The smallest values of α_v are noted for the system containing PI-2 and PI-4, while for the other two composites the α_v is slightly higher. However, given the refraction properties of the PI-3/TiO₂ and the fact that at the highest loading level the magnitude of α_v is considerably reduced, it can be considered that this sample is suitable for matching the thermal properties of the ZnO conductive layer from the solar cell structure.

4. Conclusions

New polymer composites were theoretically designed for use in photovoltaics as shielding layers. The polyimides were designed from the same aromatic diamine and distinct cycloaliphatic dianhydrides. These polymers were then ideally loaded with TiO₂ filler in various amounts. The balance between thermal stability and the refractive index was optimized by combining in the same molecule cycloaliphatic sequences together with aromatic units and sulphone groups. The spatial molecular conformations and chain polarizability were found to depend on the matrix structure and amount of filler, thus determining differences in the thermal and optical properties. The dianhydride segment influenced the resulted values of refractive index, showing that its bulky character, asymmetry and flexibility lead to larger n -values, which render lower optical losses at the interface with ZnO layer from the solar cell. These polyimides display high thermal stability and relatively low coefficient of thermal expansion around 10^{-4} K^{-1} . The presence of TiO₂ filler in these polyimides improves the matching of the refractive and coefficient of thermal expansion with those of the neighbouring ZnO layer from the photovoltaic cell. Hence, the device reliability and conversion efficiency are enhanced in terms of thermal expansion and the amount of transmitted light towards the junction, respectively. Particularly, the PI-3/TiO₂ is the most suited shielding material since it reduced significantly the reflection losses at the ZnO interface.

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