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Blending of Poly(methyl methacrylate) with Poly(1,3-diphenyl-1H-pyrazol-5-yl methacrylate): Investigation of its Optical Properties

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Abstract

In the present study, a new blend polymer consisting of a pyrazole-derived polymer poly(1,3-diphenyl-1H-pyrazol-5-yl methacrylate) and poly(methyl methacrylate) was prepared with solution mixing method. The blend polymer was spectrally characterized with FTIR and ^{1}H-NMR techniques. TGA analysis showed that the blend polymer was stable up to 275.4 °C. A thin film of blend polymer was prepared by using spin coating technique. The UV measurements were obtained as a function of wavelength (300 – 700 nm). The optical and dispersion parameters were reported in detail. Transmittance was increased depending on the increasing wavelength whereas the reflectance decreased. The Transmittance value at 700 nm was measured as 97.55%, meaning the blend polymer was transparent. The refractive index at 700 nm was 1.37. The Urbach energy (E_u) and the optical band gap constant (E_g) were calculated to be 0.831 eV and 3.737 eV, respectively. The type of electronic transitions for polymer was determined as indirect allowed transition.

Keywords: Blend polymer; Pyrazole; Methyl methacrylate; UV absorptions; Optical properties; Dispersion parameters.

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Poli(metil metakrilat) ile Poli(1,3-difenil-1H-pirazol-5-il metakrilat)'ın Blendleşmesi: Optik Özelliklerinin Araştırılması

Öz

Mevcut çalışmada, pirazol türevli bir polimer olan poli(1,3-difenil-1H-pirazol-5-il metakrilat) ve poli(metil metakrilat) içeren yeni bir blend polimer, çözelti karıştırma yöntemiyle hazırlandı. Hazırlanan blend polimer FTIR ve ^{1}H-NMR teknikleri ile spektral olarak karakterize edildi. TGA analizi blend polimerin 275.4 °C'ye kadar stabil olduğunu gösterdi. Spin kaplama tekniği kullanılarak blend polimer ince filmi hazırlandı. UV ölçümleri dalga boyunun (300-700 nm) bir fonksiyonu olarak kaydedildi. Optik ve dağılım parametreleri detaylı olarak rapor edildi. Artan dalga boyuna bağlı olarak geçirgenlik artarken yansıma azaldı. Geçirgenlik değeri 700 nm'de %97,55 olarak ölçüldü, bu da blend polimerin oldukça geçirgen olduğu anlamına gelmektedir. Kırılma indisi değeri 700 nm'de 1.37 olarak hesaplandı. Urbach enerjisi (E_u) ve optik bant aralığı sabiti (E_g) sırasıyla 0.831 eV ve 3.737 eV olarak belirlendi. Polimerin elektronik geçiş türünün dolaylı izinli geçiş olduğu görüldü.

Anahtar Kelimeler: Blend polimer; Pirazol; Metil metakrilat; UV absorpsiyon; Optik özellikler; Dispersiyon parametreleri.

1. Introduction

Polymer blends are one of the most important research topics in polymer chemistry. The chemical structures, properties and processing techniques of polymer blends have been investigated in scientific and technological platforms for years [1]. Polymer blends play an important role in our lives due to their high elasticity, low cost, light, transparent and easy processing. Thanks to these mentioned features; polymer blends have a wide application and usage potential in electronics, packaging, automotive, daily life materials, household appliances, etc. Therefore, polymer blends have shown a continuous development and become an ideal candidate for today's modern technologies [2]. Generally, a polymer blend is a physical mixture of at least two or more different homopolymers. By blending, a new material is obtained that differs in properties from its homopolymers. Polymer blends can be classified as plastic, thermoplastic, elastomer, etc. according to the physical properties of their components. In addition, the final properties of a polymer blend are highly dependent on the morphology, miscibility, and phase behavior of its partners [3]. If a well-defined blend with the desired properties can be achieved, this blending technique becomes a faster and more cost-effective technique without the need for the synthesis of new monomers or the use of new polymerization methods [1]. Many studies investigating PMMA-based blend polymers and their various properties can be seen widely in the literature. For example, one of these studies was reported by Bubmann et al. in which they prepared the transparent terminally phenolic OH-functionalized polycarbonate and epoxy-functionalized polymethylmethacrylate (PC/PMMA) blends with enhanced mechanical properties via reactive compounding of functionalized polymers [4]. In another study, Le and coworkers reported that the multiphase blends were prepared by melt processing of poly(L-lactide) and poly(methyl methacrylate) at various compositions to identify their morphologies, phase relationships and biomaterial compatibility for tissue engineering studies [5]. Aid and friends studied the miscibility of poly(vinylidene fluoride)-PVDF/PMMA blends at different approaches such as experimental tests using physico-chemical and rheological methods, thermodynamics using Flory-Huggins parameters and numerical simulation using Fluent Ansys software to describe the coalescence phenomenon [6].

The optical properties of polymer molecules are quite remarkable in addition to the many physical and chemical properties of these molecules such as thermal, mechanical, rheological, dielectric, etc. Among the optical properties of polymers, some parameters such as transmittance, reflectance, absorbance, refractive index, optical band energy, and dispersion parameters are widely studied [7-9]. These properties are highly dependent on the chemical structure of the polymer or copolymer, the formulation of the polymeric material (colorants, fillers, plasticizers, stabilizers and other additives), crystallinity and amorphous structure of the polymer. In addition to these, the mechanical, thermal or chemical degradation of the polymer, the mechanical conditions that the polymer is exposed to during production or processing, heat treatments, environmental conditions, solvents and chemical interactions significantly change the optical properties of polymers.

Many techniques can be used to determine the optical properties of polymers. However, among these, Ultraviolet-Visible spectroscopy is a technique that can give very effective, reliable and fast results in the optical characterization (absorbance, transmittance, reflectance) of polymers [10]. Optical measurements of polymers at different wavelengths are taken in UV-vis spectroscopy, and many different optical parameters can be easily determined by using the obtained data in the methods or equations available in the literature. There are many publications to investigate the optical properties of different chemically derived blend polymers. Some of those, Tikish and friends investigated the electrical and optical properties of polypyrrole and polyaniline blends [11]. Takahashi and coworkers reported a paper on the optical properties of ethylene–vinyl acetate copolymer and poly(methyl methacrylate) blends at different compositions [12]. In a study reported by our research group, we prepared a blend polymer based poly(ethyl methacrylate) and poly(2-hydroxy-3-phenoxypropyl methacrylate). The compatibility, thermal degradation kinetics, degradation mechanism and electro optic properties of that blend polymer was studied [13].

The synthesis of many heterocyclic polymers with different functional groups and also preparation of blend polymers of these polymers with commercial ones are seen in in the literature. However, the synthesis and characterization of the blend polymer containing pyrazole-derived poly(1,3-diphenyl-1H-pyrazol-5-yl methacrylate) polymer with poly(methyl methacrylate) and especially the optical properties of this blend polymer have not been found in the literature. Pyrazole-derived molecules have been the subject of widespread study in many fields with their small molecule forms as well as their polymeric macromolecular structures. Especially, pyrazole-derived polymers are one of the most important members of heterocyclic polymers. The pyrazole ring consists of a five-membered lactam ring containing two nitrogen atoms in its chemical structure [14]. In addition, the pyrazole molecule also has a dense pi bond structure. There has been an increasing trend of studies on pyrazole derivatives in recent years. The fact that both two pi bonds and heteroatoms are present in the structure of these molecules have made these molecules particularly important electro-optic properties in addition to their pharmacological properties [15, 16]. From this point, the main purpose of this study is to synthesize and characterize a new blend polymer consisting of a pyrazole-derived polymer poly(1,3-diphenyl-1H-pyrazol-5-yl methacrylate) and poly(methyl methacrylate). The miscibility of two polymers in terms of different chemical structures has been tested. Thus, it is aimed to develop new pyrazole-derived polymeric systems and contribute to the heterocyclic polymer class. Another aim is to determine some important optical parameters such as transmittance, reflectance, optical band gap, Urbach energy, dispersion parameters of new blend polymer thin film prepared by spin coating technique.

2. Materials and Methods

2.1. Sample preparation

Methyl methacrylate, benzoyl peroxide, tetrahydrofuran (THF), chloroform, and ethyl alcohol were purchased from Sigma-Aldrich. Methyl methacrylate (MMA) was passed through an alumina column to separate it from the inhibitors. Then, poly(MMA) was synthesized by free radical polymerization using 1.00 g of MMA, 3 ml of THF, and 0.01 g benzoyl peroxide (1 %wt of MMA) at 60 ºC for 24 h. After that, polymer was precipitated in ethyl alcohol, filtrated, and dried. Synthesis and characterization of pyrazol derived polymer poly(1,3-diphenyl-1H-pyrazol-5-yl methacrylate), poly(DPMA), was reported in our previous study [17]. Blend of poly(MMA) with poly(DPMA) was prepared by solution method in THF. In the present work, we set the weight fraction of poly(MMA) to poly(DPMA) blend as 50% : 50%, respectively. For this process, poly(DPMA) homopolymer (0.100 g) was first added to a 50 ml single necked reaction flask and dissolved by adding THF (3 mL). Then the calculated amount of poly(MMA) homopolymer (0.100 g) was added to this solution. The solution containing both homopolymers was stirred on a magnetic stirrer at room temperature for 24 hours. At the end of this period, the blend polymer solution was precipitated in ethyl alcohol, filtered and dried in a vacuum oven. A suitable schematic illustration for the preparation of the blend polymer was given in Fig. 1.

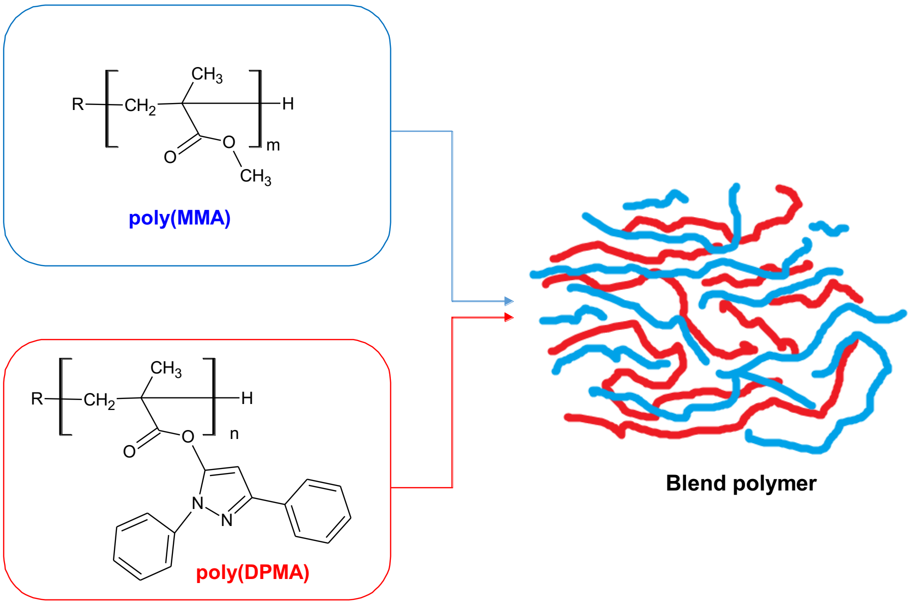

Figure 1: Preparation of the poly(DPMA)/poly(MMA) blend polymer

2.2. Characterization

At the spectral characterizations of poly(DPMA)/poly(MMA) blend polymer, a high resolution Bruker Avance III HD 300 MHz NMR Spectrometer and a Perkin Elmer Spectrum 100 FTIR Spectrometer (with ATR unit) were used. TGA analysis was performed on a Seiko SII 7300 TG/DTA thermobalance heated from ambient temperature to 500 ºC at heating rate of 15 ºC/min in nitrogen flow of 25 mL/min. Blend polymer thin film was prepared by spin coating technique. For this purpose, a Laurell WS-400-6NPP-Lite spin coater was used. In this process, blend polymer (0.015 g) was dissolved in 0,5 mL of THF and coated onto a glass at the conditions of 5 bar argon gas pressure and 1500 rpm for 50 seconds. The thickness of film was measured to be 6 µm using a digital micrometer supplied by Mitutoyo Corporation Ltd Japan. After that, the UV measurements of blend thin film was obtained by using a Perkin Elmer Lambda 25 UV/VIS spectrophotometer at wavelengths of 300 - 700 nm.

3. Results and Discussion

FTIR spectra of poly(MMA), blend polymer and poly(DPMA) were shown in Fig. 2(a-c), respectively. In the FTIR spectrum of the blend polymer (Fig. 2b), the characteristic signals for both homopolymers are seen. It is important to observe the absorbance's of aromatic C-H stretching (3160-3029 cm^{-1}) and the aromatic C=C stretching (1595 cm^{-1}) which are characteristic for poly(DPMA) in blend polymer apart from the standard poly(MMA) signals.

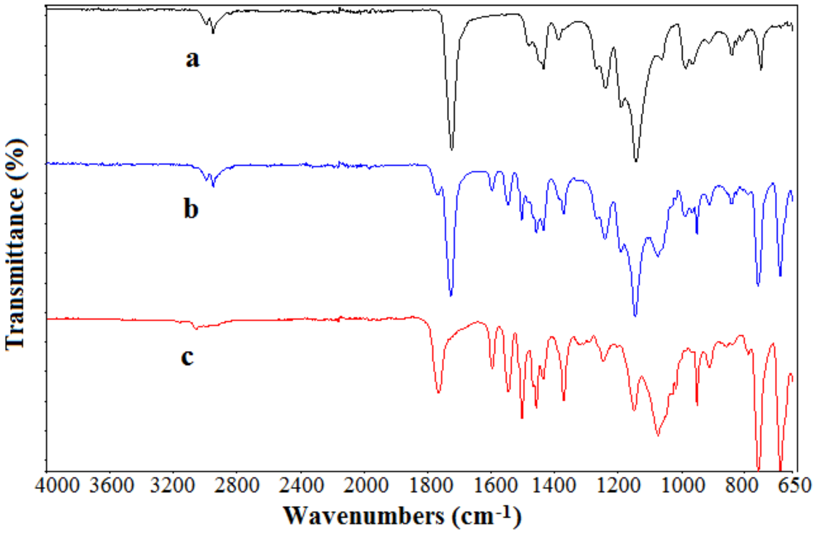

Figure 2: FTIR spectra of a) poly(MMA), b) blend polymer, c) poly(DPMA)

So, these signals clearly show that the blend polymer has accomplished and the presence of both homopolymers in the blend. A second important change is occurred in the ester carbonyl region. The methacrylic ester carbonyl stretching in the blend polymer is seemed at 1768 cm^{-1} and 1726 cm^{-1} reasoned from the ester carbonyl bands of poly(DPMA) and that of poly(MMA), respectively. In the blend polymer, the vibration energy of ester carbonyls of both polymers are relatively shifted due to possible intermolecular secondary interactions. This situation gives an important clue about the miscibility of the blend polymer.

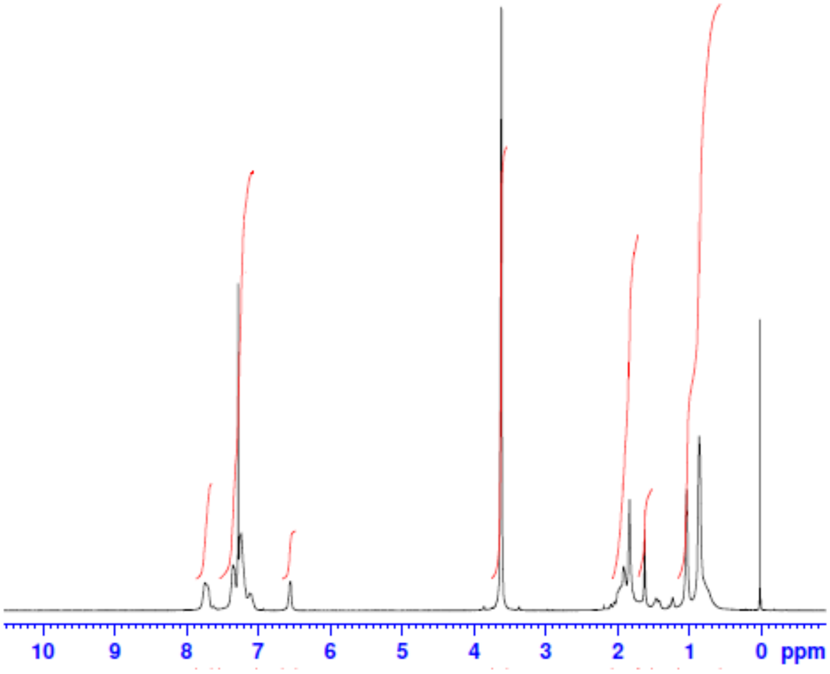

Figure 3: ^{1}H-NMR spectra of blend polymer

Another proof of the success of blend polymer synthesis is the ^{1}H-NMR spectrum. This spectrum is shown in Fig. 3 where the signals belonging to both poly(MMA) and poly(DPMA) units are seen. Accordingly, the 7.74 – 7.19 ppm signals and also 6.56 ppm signal are recorded for the aromatic =CH protons in phenyl groups and aliphatic =CH proton in the pyrazole group for poly(DPMA), respectively. The signal at 3.62 ppm is attributed to $–COOCH_3$ protons in poly(MMA). Methylene and methyl protons on the blend polymer main chains reasoned from both units are observed at 1.92 – 0.86 ppm.

Thermal analysis of blend polymer was accomplished by thermogravimetric analysis (TGA). The thermal decomposition process was performed on a temperature range of room temperature to 500 °C. Fig. 4 shows the TGA thermogram of blend polymer. As can be seen, the decomposition of blend polymer is occurred in three stages. The first one, with up to 12% weight loss at ~290 - 340 °C usually caused by volatile hydrocarbons, the second decomposition stage is about between 340 – 390 °C with up to 44% weight loss, and the third one is ~ 390 – 460 °C with up to 94% weight loss. The initial decomposition temperature of the blend polymer is measured as 275.4 °C. In our previous study, this value was determined as 238.8 °C for the poly(1,3-diphenyl-1H-pyrazol-5-yl methacrylate) homopolymer [17]. As a result of the blending of this homopolymer with poly(MMA), an increase in thermal stability occurred. In addition, the final decomposition temperature of the blend polymer was determined as 460 °C and the temperature of 50% decomposition was 401.1 °C. The % mass losses of the blend polymer at different temperatures such as 350 °C, 400 °C and 450 °C were also recorded as 15.53%, 49.90% and 90.94%, respectively.

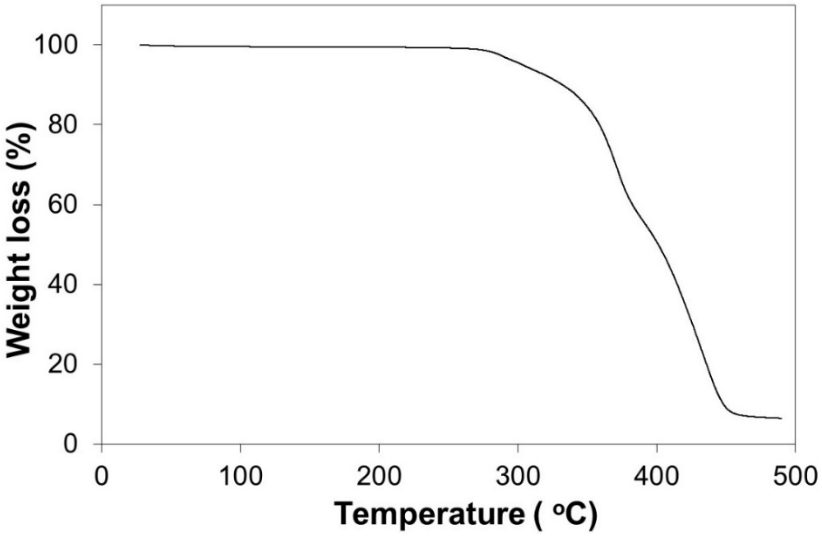

Figure 4: TGA thermogram of blend polymer

The UV measurements of poly(DPMA)/poly(MMA) blend polymer were recorded at the wavelengths of 300 - 700 nm for determining the optical properties. Therefore, firstly, the absorbance (A) and transmittance (T) measurements were directly measured by UV and then the reflectance (R) values were calculated by the equation of (R = 1 - T - A) [18]. Transmittance and reflectance spectra of blend polymer were shown in Fig. 5 and Fig. 6, respectively. The transmittance values showed a general increase depending on the increase in wavelength. While this increasing trend is quite sharp in the 300-330 nm range, the increase in the next wavelengths gradually decreases and reaches almost constant value.

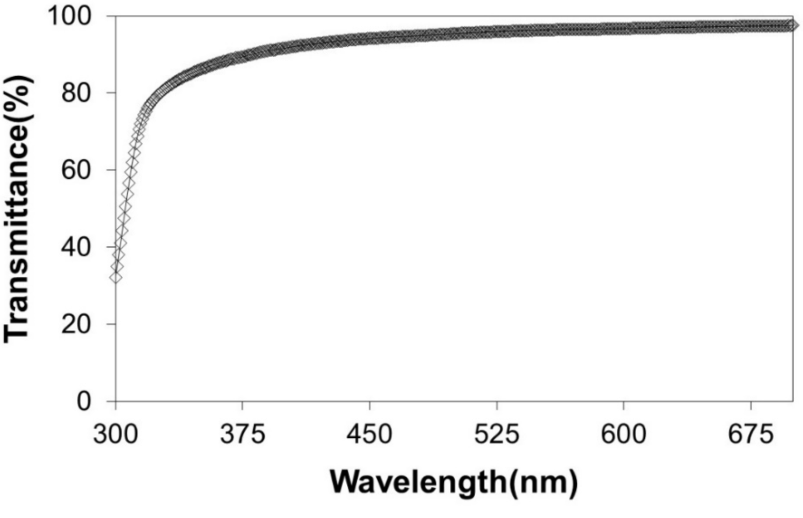

Figure 5: Transmittance spectra of blend polymer thin film

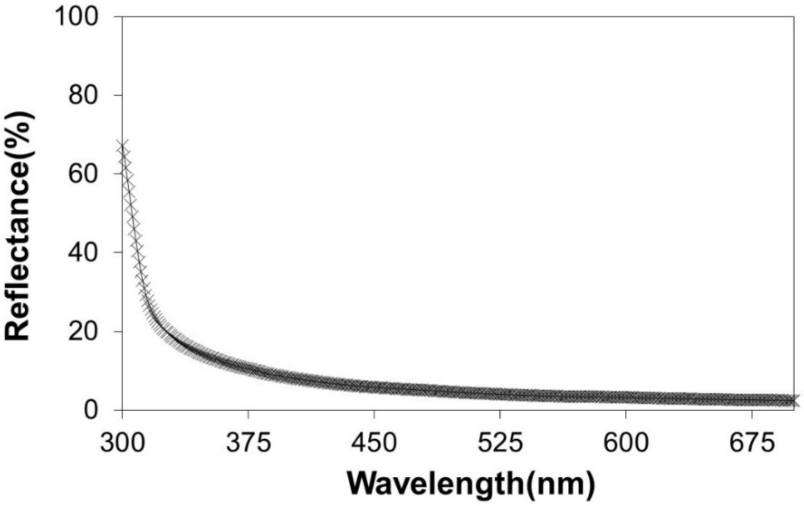

Figure 6: Reflectance spectra of blend polymer thin film

An opposite behavior of the transmittance values was observed in the reflectance values. A rather drastic decrease in reflectance values was observed, especially at low wavelengths (up to 320 nm). However, depending on the increase in wavelength, a slowdown occurs in this decrease rate and an almost constant value is obtained towards 700 nm. The transmittance and reflectance values at 700 nm were measured as 97.55% and 2.43%, respectively. The presence of pyrazole groups in the structure of the prepared blend polymer and also the presence of intense pi-bonds in these groups cause the absorption at low wavelengths to be higher [19].

The refractive index (n) is very important for the optical characterization of polymers. It is possible to determine this parameter with the following formula using the transmittance and reflectance values [20]:

$$n = \left[\frac{1+R}{1-R}\right] + \left[\frac{4R}{(1-R)^2} - k^2\right]^{1/2} \tag{1}$$

where k is the extinction coefficient and equal to, $k = \alpha\lambda/4\pi$. In this formula, α is the absorption coefficient which is obtained as: $\alpha = 2.303A/d$ where A is the absorbance and d is the film thickness. Fig. 7 shows the relationship between the refractive index and the wavelength. There is a significant decrease in the refractive index depending on the increase in wavelength. While this decrease is quite evident especially at low wavelengths, the downward trend towards the next wavelengths gets lighter [21]. The refractive index value at 700 nm is measured as 1.37. This value is the expected value for methacrylate-derived polymers [22].

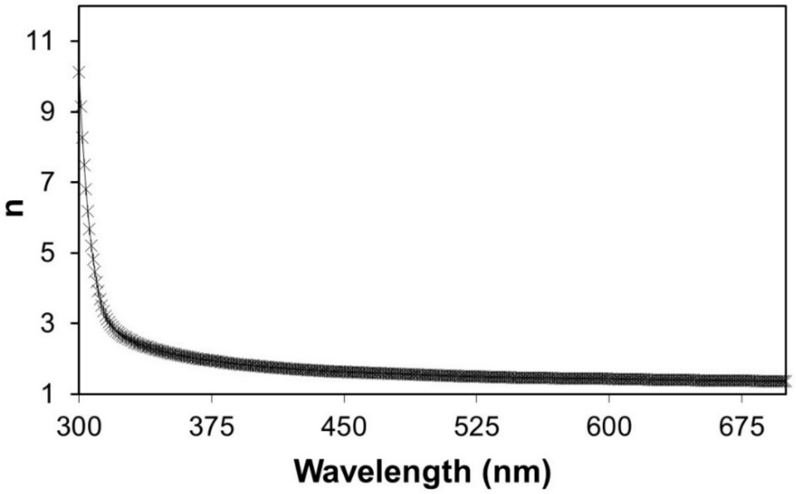

Figure 7: Refractive index dispersion of blend polymer thin film

The dependence of the dispersion energy parameters on the refractive index were well defined by Wemple and DrDomenico with the following equation [23].

$$n^2(h\nu) = 1 + \frac{E_o E_d}{E_o^2 - (h\nu)} \qquad (2)$$

These parameters are E_o, the single – oscillator energy and E_d, the dispersion energy. From these parameters, E_o responsible from the electronic excitations in the molecules and illustrates the average of the optical band gap whereas E_d shows the average strength of inter-band optical transition [24]. Figure 8 shows the $(n^2-1)^{-1}$ vs. $(h\nu)^2$ plots. The linear fit of this figure yields a slope corresponds to $(E_oE_d)^{-1}$ and intercept is E_o/E_d. Finally, the single – oscillator energy (E_o) and the dispersion energy (E_d) values for blend polymer are calculated to be 3.982 eV and 3.488 eV, respectively. These values are given in Table 1.

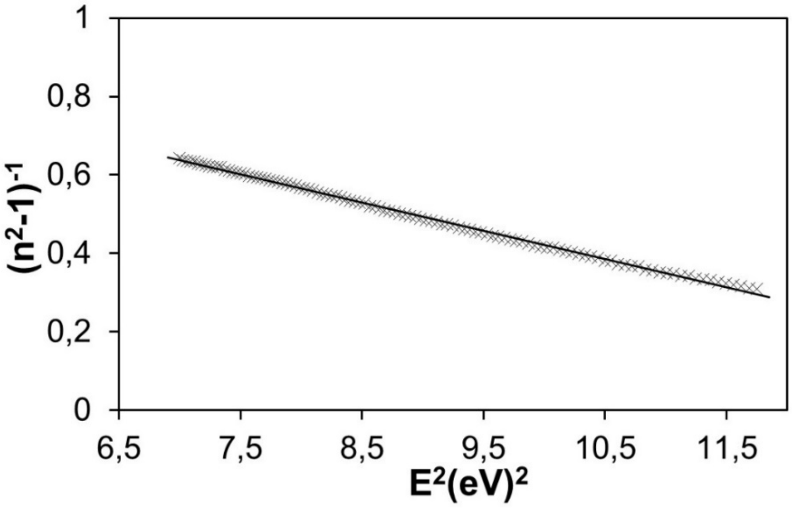

Figure 8: Variation of $(n^2-1)^{-1}$ as a function of E^2 of blend polymer thin film

Wemple and DrDomenico have defined the moments of imaginary part of the complex dielectric constant (M_{-1} and M_{-3}) with the single oscillator energy parameters (E_o and E_d). These moments can be determined from following relation [23, 25]:

$$E_o^2 = \frac{M_{-1}}{M_{-3}} \qquad E_d^2 = \frac{(M_{-1})^3}{M_{-3}} \tag{3}$$

Table 1 includes the moments M_{-1} and M_{-3} are calculated as 0.876 and 0.055, respectively. These parameters are also given in Table 1.

The average inter-band oscillator wavelength (λ_o) and oscillator strength (S_o) may be obtained from the single-term Sellmeier equation [26]. This is expressed as:

$$n^2(\lambda) - 1 = \frac{S_0.\lambda_0^2}{1 - (\lambda_0/\lambda)^2} \tag{4}$$

Figure 9 shows the $(n^2-1)^{-1}$ vs. λ^{-2} plots. From the slope and intercept of this figure, the average inter-band oscillator wavelength (λ_o) and oscillator strength (S_o) is calculated as 311.7 nm and 9.02×10^{12} m^{-2}. Table 1 also gives these values.

Table 1: Optical results of blend polymer thin film

E_u (eV)	**E_g (eV)**	**E_0 (eV)**	**E_d (eV)**	**M_{-1}**	**M_{-3} (eV)$^{-2}$**
0,831	3,737	3,982	3,488	0,876	0,055
$S_0.10^{12}$(m^{-2})	**λ_0(nm)**	**σ**	**α_0**	**n (700 nm)**	**T% (700 nm)**
9,02	311,7	0,0309	$3{,}46.10^{-7}$	1,37	97,55

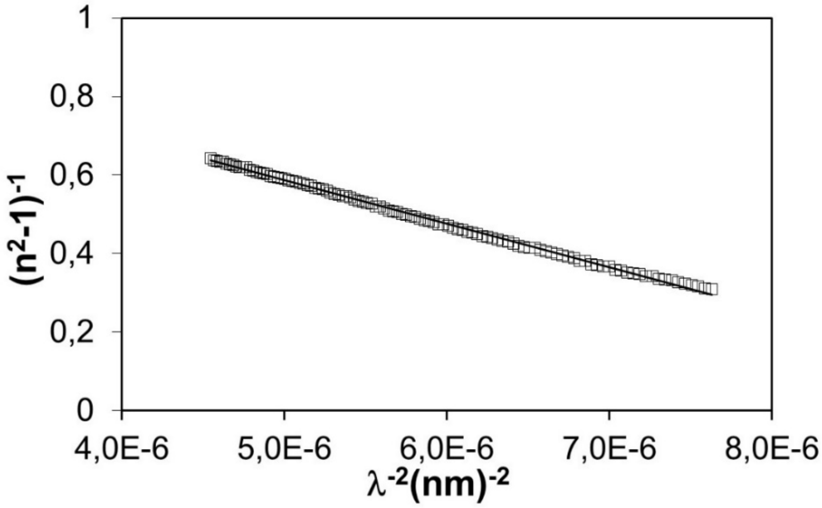

Figure 9: Variation of $(n^2-1)^{-1}$ as a function of $(\lambda)^{-2}$ of blend polymer thin film

The interband transitions of polymers may be obtained by analyzing the absorption data at the fundamental edge by [27]:

$$(\alpha h\nu) = B(h\nu - E_g)^n \tag{5}$$

where E_g is the optical band gap, B is a constant, and n is an index that shows the electronic transition types [24]. For the prepared blend polymer, the electronic transition type is the indirect allowed transition (n=2). So, the $(\alpha h\nu)^{1/2}$ vs $(h\nu)$ values are plotted as shown in Fig. 10. The slope of this line gives the B constant, which is 0.0189, whereas the intercept corresponds *(-B.E$_g$)*. When the value of B is written in the intercept, the optical band gap (E_g) is calculated as 3.737 for blend polymer.

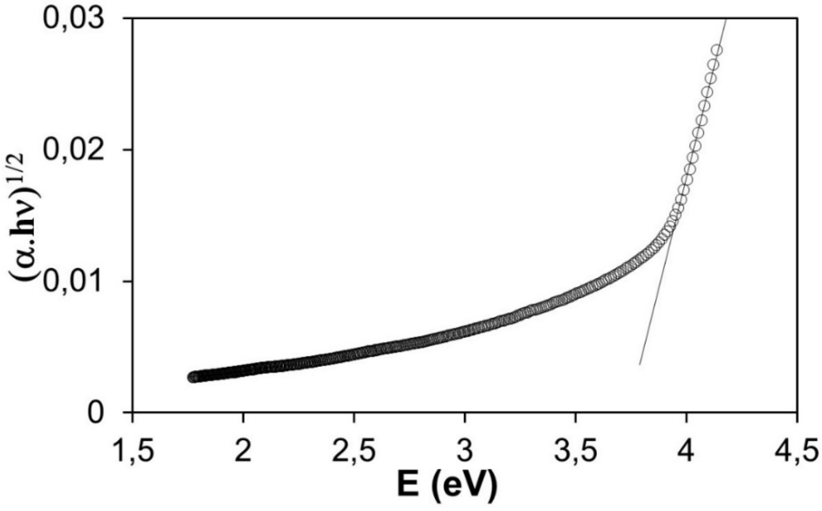

Figure 10: Plots of $(\alpha h\nu)^{1/2}$ vs: E of blend polymer thin film

On the other hand, some local defects may occur in polymeric materials when exposed to UV radiation. One effect of these defects is to trap electrons and prevent their direct transition to the conduction band [28]. Urbach energy is defined as a measure of the size of these defects and expressed [29]:

$$\alpha = \alpha_0 \exp(E/E_u) \tag{6}$$

where α_0 is a constant and E_u is the Urbach energy. Fig. 11 shows the (lnα) vs E plots. The slope of this figure equal to $1/E_u$ and from this Urbach energy value of blend polymer is found to be 0.831 eV. Also, α_0 constant is $3.46x10^{-7}$.

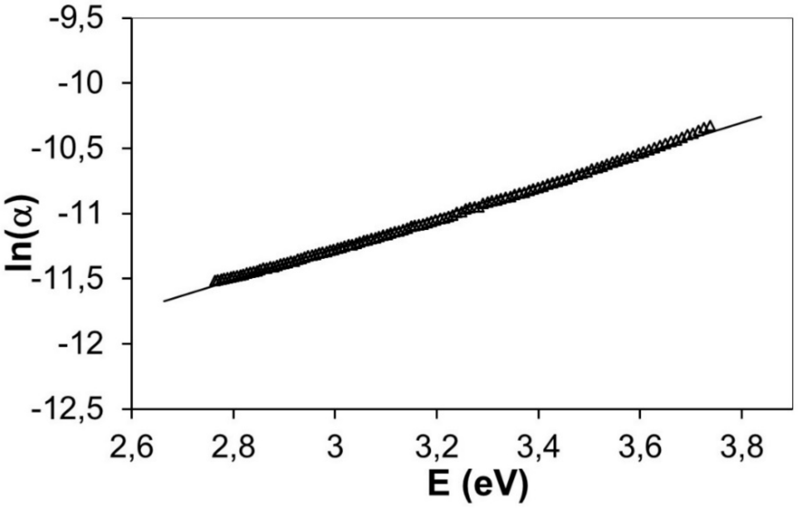

Figure 11: Plots of ln(α) vs: E of blend polymer thin film

Steepness parameter which is a constant corresponds the broadening of absorption edge and characterizes the sates usually caused by electron-phonon interactions [30] in the Urbach spacing. The slope of line in Fig. 11 also gives σ/kT at a constant temperature. Where k is the Boltzmann constant, T is the temperature, σ is the steepness parameter. When the Boltzmann constant are taken as $8.167x10^{-5}$ $eV.K^{-1}$ and the temperature is 298 K, the steepness parameter of blend polymer is determined as 0.0309.

4. Conclusion

UV/VIS measurements of newly prepared blend polymer [poly(DPMA)/poly(MMA)] thin film were obtained to determine the electro optic parameters. Transmittance was increased depending on the increase in wavelength whereas the reflectance values were decreased. The refractive index at 700 nm was 1.37. The optical band gap (E_g) and the Urbach energy (E_u) were determined as 3.737 eV and 0.831 eV, respectively. The electronic transition type was indirect allowed transition. The average inter-band oscillator wavelength (λ_o) and oscillator strength (S_o) were found to be 311.7 nm and 9.02×10^{12} m^{-2}, respectively. The oscillator energy (E_o) was 3.982 eV and the dispersion energy (E_d) was 3.488 eV. TGA analysis showed that the blend polymer was stable up to 275.4 °C.

Acknowledgments

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