

Photoluminescent Enhancement of Polyesters via Photocrosslinking

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ABSTRACT: A homologous series of soluble photoluminescent polyesters, namely poly{bis(4-hydroxybenzylidene)acetone-5-(*m*-alkylox-isophthalate)}, PAII-PAI5 were designed and synthesized via polycondensation reaction. The branches of these polyesters consisted of flexible alkyloxy chains with different chain lengths in order to investigate their influence toward thermal stability as well as the photocrosslinking property. It was found that increasing length of the flexible chain would reduce the thermal stability of the polyesters. Irradiation of these polyesters under UV light induced photocrosslinking and showed fascinate enhancement in their photoluminescence property with greater emission intensity. This was evidenced visually from the transformation of a weak greenish blue emission to highly intense cloudy blue emission. The photocrosslinking system was further confirmed using spectroscopic methods. Morphology analysis using SEM showed that the photocrosslinked polyesters have a fiber-like or thread-like texture with more voids as compared to the virgin polyesters. This was because of the formation of cyclobutanes through photodimerization involving $2\pi + 2\pi$ cycloaddition. © 2014 Wiley Periodicals, Inc. *J. Appl. Polym. Sci.* **2015**, *132*, 41504.

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INTRODUCTION

The development of photosensitive polymers has gained great interest in recent years owing to their wide variety and well-proven technological applications in the field of photoresist, photolithography, printing materials, liquid crystalline, and energy exchange materials besides being employed in various optical applications such as second harmonic generation materials in nonlinear optics materials, photorefractive polymers, holographic recording materials, and fluorescent probes for sensing of metal ions, biological macromolecules, and microenvironment in micelles.^{1–6} In fact, many of these polymers have been used in manufacturing of industrial products such as integrated circuits, compact discs, cathode ray tubes, and printed circuit boards.⁷

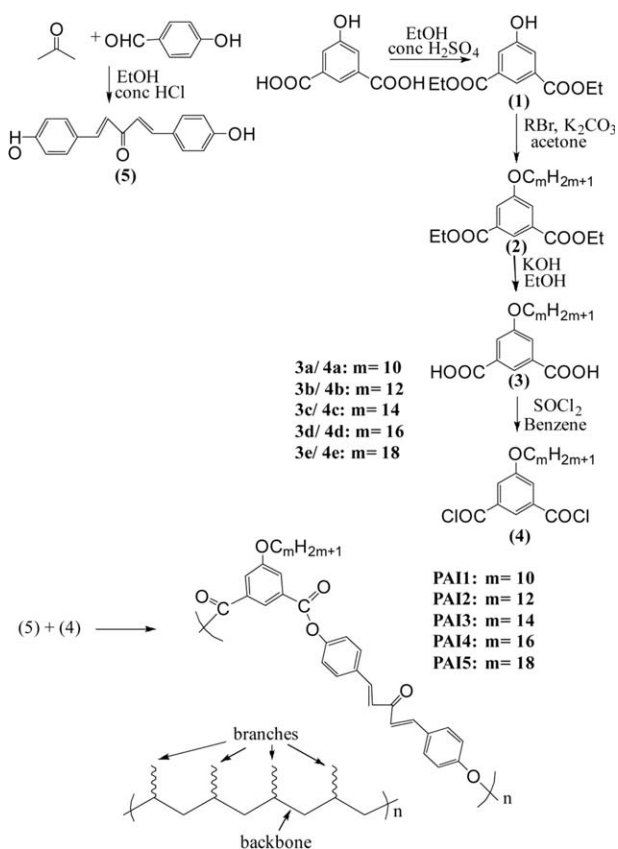
Photosensitive polymers are well defined as having photosensitive groups attributing to the π electron density of the photoactive chromophore in the main chain or pendant groups which will cross-link or cleave after irradiation with UV light.^{8,9} Beneficially, UV irradiation in forming highly cross-linked polymers are environment-friendly technology that can be generated within seconds just by the presence of UV source at ambient

temperature. These highly cross-linked polymer networks can be achieved by the photocrosslinking of functionalized polymers.^{10,11} A series of photocrosslinkable polymers have been synthesized with arylidene keto moiety in this work as this moiety is a versatile photo-active molecule bearing special features like high thermal stability, photo-curing ability (for adhesives, photo-resists, holography), electronic conductivity, fluorescence and able to undergo thermal cross-linking. Besides, these polymers merit attention for the usage in nonlinear optics because of their large electro-optic coefficients. Polymers with arylidene keto moiety in fact give other useful potential applications in fluorescent sensors, photoresist technology as well as in medicinal prospects.¹²

To date, most of the main chain polymers exhibit solubility defect because of their rigid-rod nature. The insolubility of polymers in organic solvents as well as their high melting and softening temperatures are the major drawbacks in applications.¹³ However, in this study, soluble polymers were designed and successfully synthesized. The feasibility of processing the polymers is the utmost important key for industrial applications which is depending on the solubility, flexibility and softening or melting temperatures of the polymers.¹⁴

Additional Supporting Information may be found in the online version of this article.

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Scheme 1. Synthetic routes toward formation of monomers 3a–3e, monomer 5 and their respective polyesters, PAI1–PAI5.

Photocrosslinkable polyesters with flexible alkoxy chains have yet to be explored. The present study involves the synthesis and characterization of new photocrosslinkable polyesters bearing arylidene keto moiety with the branches of flexible alkoxy chains. The structures of synthesized polyesters were elucidated using various spectroscopic techniques and their thermal behaviors and photocrosslinking properties with respect to the hydrophobic flexible alkoxy chains were discussed. The photocrosslinking studies of the synthesized polyesters were carried out under ultraviolet (UV) light to induce the $2\pi + 2\pi$ cycloaddition of the arylidene ketone and the photoreaction was studied in detail by spectroscopic methods. The effect of photocrosslinking toward the photoluminescence properties of the polyesters prior to and after cross-link was also discussed in this study.

EXPERIMENTAL

Materials

Chemicals used were potassium hydroxide, potassium iodide, 5-hydroxyisophthalic acid, 4-hydroxybenzaldehyde, triethylamine, potassium carbonate, 1-bromodecane, 1-bromododecane, 1-bromotetradecane, 1-bromohexadecane, 1-bromooctadecane, and thionyl chloride. All the chemicals were used without further purification while solvents like acetone, ethanol, benzene, triethylamine, tetrahydrofuran, glacial acetic acid, hydrochloric acid, and sulphuric acid were used as received.

Characterization Techniques

IR spectra were recorded for the monomers and polyesters using Perkin Elmer 2000-FTIR spectrophotometer in the frequency range of $4000\text{--}400\text{ cm}^{-1}$ with sample prepared in KBr discs. The Bruker Avance 500 MHz ultrashield spectrometer equipped with ultrashield magnets was used to record the ^1H NMR and ^{13}C NMR spectra of the synthesized monomers and polyesters. Deuterated chloroform (CDCl_3) and dimethylsulphoxide (DMSO-d_6) were used as the solvents and TMS as internal standard. ^1H and ^{13}C NMR assignments of the polyesters were substantiated by 2D NMR such as $^1\text{H}\text{--}^1\text{H}$ correlation (COSY), $^1\text{H}\text{--}^{13}\text{C}$ heteronuclear multiple bond correlation (HMBC) and $^1\text{H}\text{--}^{13}\text{C}$ heteronuclear multiple quantum correlation (HMQC) spectroscopy. Thin layer chromatography (TLC) was performed with TLC sheets coated with silica and spots were visualized under UV light to monitor the progress of the reaction. Elemental analysis was carried out using a Perkin Elmer 2400 LS Series CHNS/O analyzer. Thermal behavior of the polyesters were evaluated via thermogravimetric analyses (TGA) which were performed with Mettler 851e TGA under nitrogen atmosphere with the heating rate of $10^\circ\text{C}/\text{min}$ while the differential scanning calorimetry (DSC) analyses of the polyesters were carried out using a Seiko DSC6200R calorimeter with the heating and cooling rate of $10^\circ\text{C}/\text{min}$. The average molecular weights of the polyesters were determined by using Waters Gel Permeation Chromatography, GPC instrument with refractive index detector. Calibration was done by using polystyrene standards and THF as the eluent. UV absorption spectra were recorded on Shimadzu UV-2600 UV-VIS spectrophotometer. Photocrosslinking studies were carried out using 6 W high-pressure Hg lamp (Vilber Lourmat, 365 nm) at a distance of 10 cm from the light source for different time intervals before recording the spectra with UV-Vis spectrophotometer. Fluorescence measurements were performed using an LS-55 fluorescence spectrophotometer (Perkin Elmer) equipped with a plotter unit and a quartz cell ($1\text{ cm} \times 1\text{ cm}$). The morphological studies of the virgin and the photocrosslinked polyesters were carried out using Scanning Electron Microscope (SEM) with the model of Leo Supra 50vp.

Synthesis of Monomers and Polyesters

The synthesis of all the monomers and polyesters were as described in Scheme 1. The synthesis steps, the percentage yields, elemental data, and spectral data of the monomers and polyesters were illustrated in the Supporting Information.

RESULTS AND DISCUSSION

Characterization of PAI1–PAI5

As PAI1–PAI5 showed similar trend of infrared absorption bands, hence a representative FT-IR spectrum of PAI2 [Figure 1(a)] was used in the discussion. A strong band at 1741 cm^{-1} was attributed to the $\text{C}=\text{O}$ of ester, confirmed the polyesterification in the formation of the polymers.¹⁵ The conjugated $\text{C}=\text{O}$ of ketone from the photochromic monomer 5 was observed at 1650 cm^{-1} .^{5,16} A strong band at $2924\text{--}2853\text{ cm}^{-1}$ can be attributed to the stretching vibration of C–H group from the alkoxy chain. A medium band at 1599 cm^{-1} can be ascribed to the $\text{C}=\text{C}$ of the phenyl ring while the $\text{C}=\text{C}$ stretching

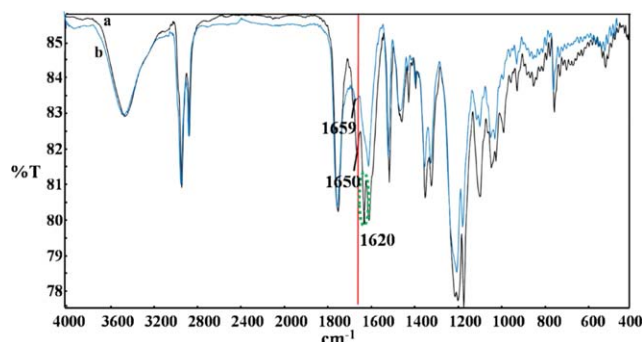


Figure 1. FT-IR spectra of photocrosslinkable PAI2 (a) before and (b) after 0.5 h of UV irradiation. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

frequency of the olefinic double bond was assigned at 1620 cm^{-1} .¹⁷

Figures 2 and 3 depicted the ^1H NMR and ^{13}C NMR spectra of PAI2, respectively. The chemical shifts of the aromatic protons were resonated around 7.07–8.61 ppm while the alkoxy $-\text{OCH}_2-$ proton was appeared to be at 4.16–4.12 ppm. The broadening of the proton signals and their complexity further substantiated

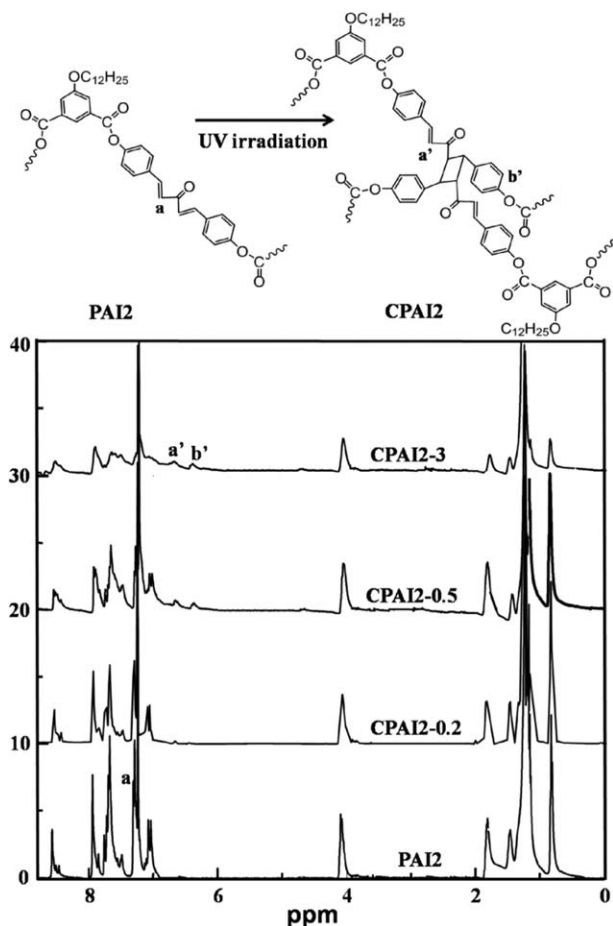


Figure 2. ^1H NMR spectra of polyester, PAI2 before UV irradiation, CPAI2-0.2 after 0.2 h UV irradiation, CPAI2-0.5 after 0.5 h UV irradiation and CPAI2-3 after 3 h UV irradiation.

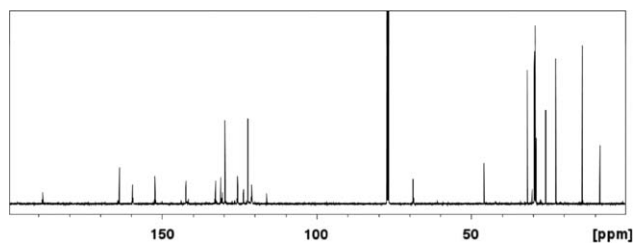


Figure 3. ^{13}C NMR spectrum of PAI2.

that the polymerization reaction was succeeded.¹⁸ In ^{13}C NMR spectrum, the chemical shifts of $\text{C}=\text{O}$ for ester and ketone appeared at 163.91 and 188.66 ppm, respectively. All the aromatic carbon signals were observed at around 121.13–152.36 ppm while the chemical shift of alkoxy $-\text{OCH}_2-$ carbon linked to the phenyl ring was at 69.32 ppm.

Besides that, the synthesized polyesters, PAI1-PAI5 were investigated for their solubility and it was found that they were soluble in various organic solvents such as chloroform, tetrahydrofuran, dimethylformamide, dimethylsulfoxide, and dichloromethane but not in alcoholic solvents and some nonpolar solvents for instance, benzene and toluene. The designed bent core polyesters as well as the long flexible alkoxy chains on the polyesters induced their solubility. This was because of the inclusion of less linear (V-shape/ bent) and more flexible core in the branches of the polymers. Their solubility properties make them more processable and feasible for various applications.

The average molecular weights, M_w of PAI1-PAI5 were determined via gel permeation chromatography (GPC). The M_w were found to be in the range of 3870–7407 and these synthesized polyesters were found to be in moderately good yield with the percentage yield ranging from 65% to 72% (Table I). From the M_w of PAI1-PAI5, it was found that degree of polymerization (DP) was depending on the length of alkoxy chains. Relatively, polyesters with shorter alkoxy chains ($m = 10$ and 12) had degree of polymerization of approximately 11–12 while those with longer alkoxy chains ($m = 14$ – 18) were found to have slightly lower degree of polymerization of approximately 6–8. This illustrated that shorter flexible alkoxy side chains may provide less steric effect during the polymerization reaction hence gave a higher degree of polymerization.

Thermal Properties of PAI1-PAI5

The thermal properties of PAI1-PAI5 were examined using TGA at the temperature ranges from 30°C to 900°C . The thermal stabilities were evaluated by the 50% weight loss and the percentage char yield. Their respective data were compiled in Table I and Figure 4. The temperature of the 50% weight loss and the percentage char yield of PAI1-PAI5 were in descending order with the increasing length of the alkoxy chains ($m = 10$ to 18) or decreasing of DP.^{19–21} The 50% weight losses for PAI1-PAI5 were recorded at 500°C , 490°C , 485°C , 480°C and 470°C , respectively. The percentage char yields at 900°C for these polyesters were 20%, 20%, 19%, 18%, and 13%, respectively. The increasing of flexibility of the molecules may induce the dilution of the main core and hence lowering their thermal stability.^{22,23}

Table I. Yields, Thermal Decomposition Values, and DSC Data of Polymers PAI1-PAI5

Polymers	Yield (%)	M_w	Weight loss (°C) 50%	Char yield at 900°C (%)	DSC T_g (°C)
PAI1	67	6259	500	20	95
PAI2	70	7407	490	20	91
PAI3	65	3870	485	19	91
PAI4	72	5192	480	18	85
PAI5	70	5066	470	13	78

DSC analysis was carried out to determine the glass transition temperatures, T_g for the polyesters. According to the data in Table I, the glass transition temperatures of PAI1-PAI5 were in decreasing order: 95°C, 91°C, 91°C, 85°C, 78°C, respectively. This showed that as the DP of the polyesters decreased (length of the alkyloxy chains increased), the T_g decreased. Again, the increment in the flexibility of the polyesters because of longer alkyloxy chains in the branches resulted in lower thermal stability.²² These observations were well corresponded to the work reported by Báez and co-workers.²⁴

PhotocrossLinking Studies of PAI1-PAI5

A representative UV-Vis spectrum in Figure 5 illustrated the changes in the UV absorbance of PAI2 ($1 \times 10^{-5} M$) with respect to the different time intervals of UV irradiation at $\lambda = 365$ nm. A decrease in the absorption intensity at $\lambda = 333.5$ nm which correspond to $\pi-\pi^*$ transition of benzyldiene chromophores and an increase of absorption band at 238 nm with an isobestic point at 286.5 nm were observed.^{25,26} This behavior clearly indicated that dimerization occurred in the olefinic double bond of the polymeric chain which involved $2\pi+2\pi$ cycloaddition in forming a cyclobutane ring. This ring formation destroyed the conjugation of the π -electron system thus giving a decrease in the absorption maxima of $\pi-\pi^*$ transition with a concomitant in the absorbance at a shorter wavelength which was because of the single bond formation from the cyclobutane ring.^{27,28} PAI1-PAI5 reacted photochemically similar to cinnamic acid and its derivatives where a cyclobutane structure was formed after the cross-linking.²⁵ The intensity of the absorption band was gradually decreased until 1300 s of irradiation time in which maximum photocrosslinking was

achieved. Beyond that no further decrease in the intensity of the absorption band was observed. Similar observation was noticed for PAI1, and PAI3-PAI5. Therefore it showed that the time taken for the maximum photocrosslinking of PAI1-PAI5 was found to be independent of the flexible alkyloxy chain lengths.

The rate of photocrosslinking in PAI1-PAI5 was determined in terms of the rate of disappearance of the $-C=C-$ group which was ascribed in Figure 6 and the percentage of photocrosslinking of the photoreactive olefinic chromophore was evaluated using the following equation:

$$\text{Percentage of photocrosslinking} = A_0 - A_t / A_0 \times 100\% \quad (1)$$

where A_0 is the absorption before irradiation and A_t is the absorption after irradiation at time t .

At the initial exposure to UV light (Figure 6), PAI1 experienced fastest rate of photocrosslinking followed by PAI2, PAI3, PAI4, and PAI5. Polyesters with shorter alkyloxy chains showed slightly higher percentage of photocrosslinking compared to those with longer alkyloxy chains. All of these polyesters showed steady increase in the percentage of photocrosslinking followed by a plateau. PAI1-PAI5 achieved maximum percentage of photocrosslinking (70–72%) at 1300 s indicated that the formation of photocrosslinked network resisted further subsequent exposure to UV light after 1300 s.^{27,28} The formation of cyclobutane ring in the polymer backbones causes steric hindrance because

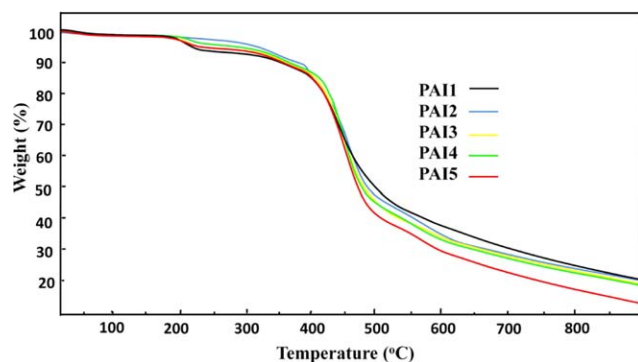


Figure 4. TGA thermograms of polyesters PAI1-PAI5. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

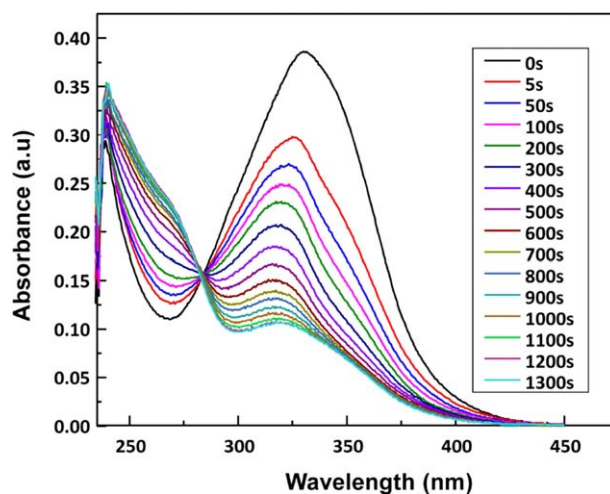


Figure 5. UV-Vis spectra of PAI2 in chloroform upon UV irradiation at various time intervals. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

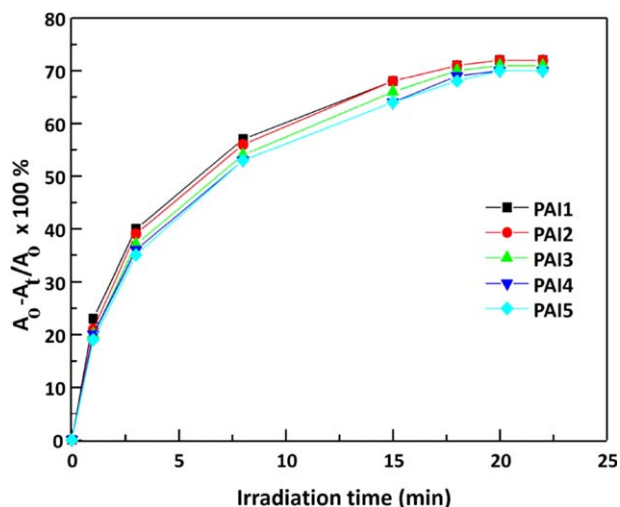


Figure 6. Rate of photocrosslinking of PAI1-PAI5 upon UV irradiation at various time intervals. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

of the bulkiness of the structure and thus hinders the ability of the $-C=C-$ moieties to cross-link completely.⁸

In order to obtain a better idea on the photocrosslinked system, FT-IR analysis was conducted. As according to the comparative FT-IR spectra in Figure 1(a,b), after 0.5 h of UV exposure, the intensity of the absorption band of the olefinic double bond ($-C=C-$) at 1620 cm^{-1} was found to be decreased drastically. The significant change in the α,β -unsaturated carbonyl group confirmed the success of $2\pi+2\pi$ cycloaddition. In addition, the absorption band of the $C=O$ at 1650 cm^{-1} was shifted to higher frequency at 1659 cm^{-1} suggested the formation of cyclobutane ring attributed to the opening of the unsaturated

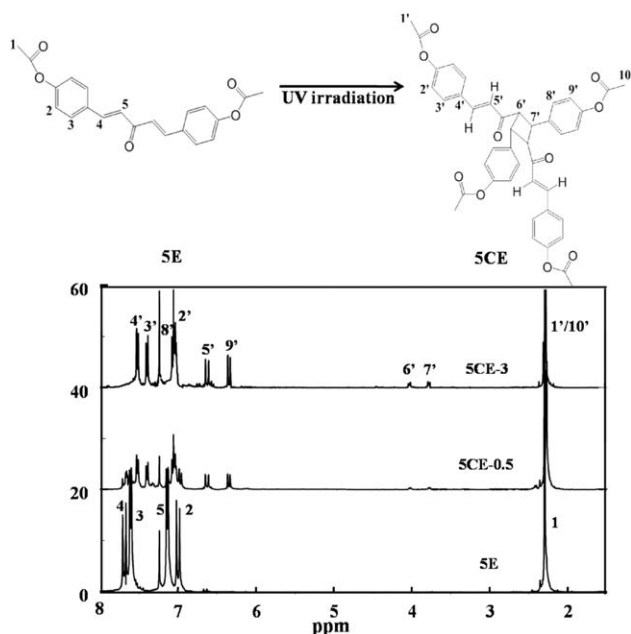


Figure 7. ^1H NMR spectra of the chromophore monomer before (SE) and after UV irradiation at 0.5 h (SCE-0.5) and 3 h (SCE-3), respectively.

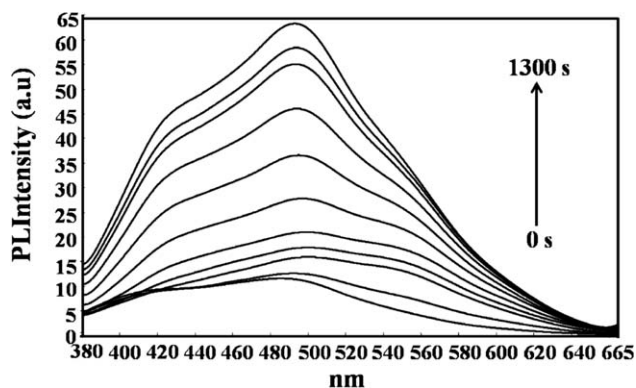


Figure 8. The changes of emission maxima of PAI2 in chloroform upon UV irradiation at various time intervals.

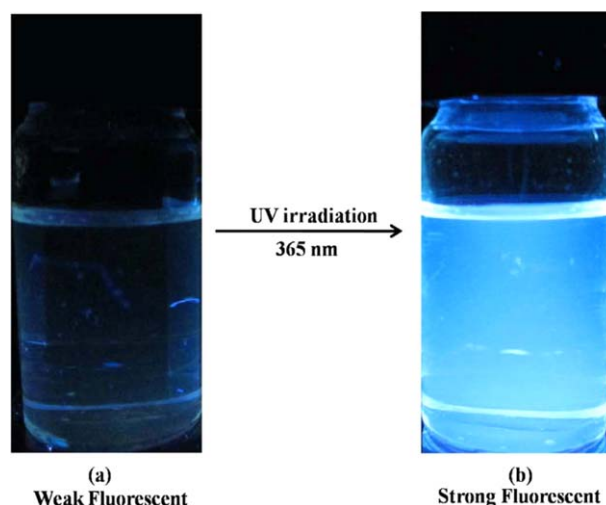


Figure 9. Images for PAI2 in chloroform with the (a) absence and (b) presence of UV source. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

bond of the olefinic functional group.²⁹ The shifting of $C=O$ band to higher frequency was because of the change from the fully conjugated $-C=C(\text{CO})-C=C-$ into partially conjugated $-C-C(\text{CO})-C=C-$ group.

Further analysis on ^1H NMR spectrum (Figure 2) of PAI2 prior to and after UV irradiation was carried out. The

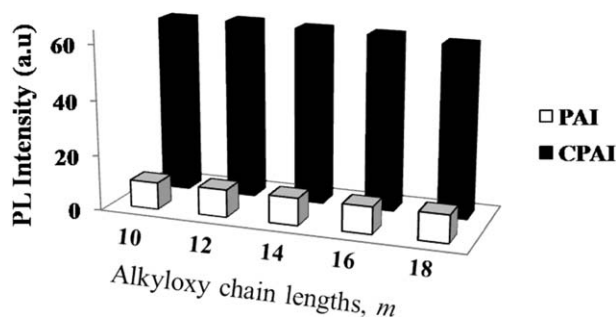


Figure 10. Fluorescence intensities of polyesters, PAI1-PAI5 before UV irradiation (PAI) and after UV irradiation (CPAI), respectively, to their alkyloxy chain lengths ($m = 10-18$).

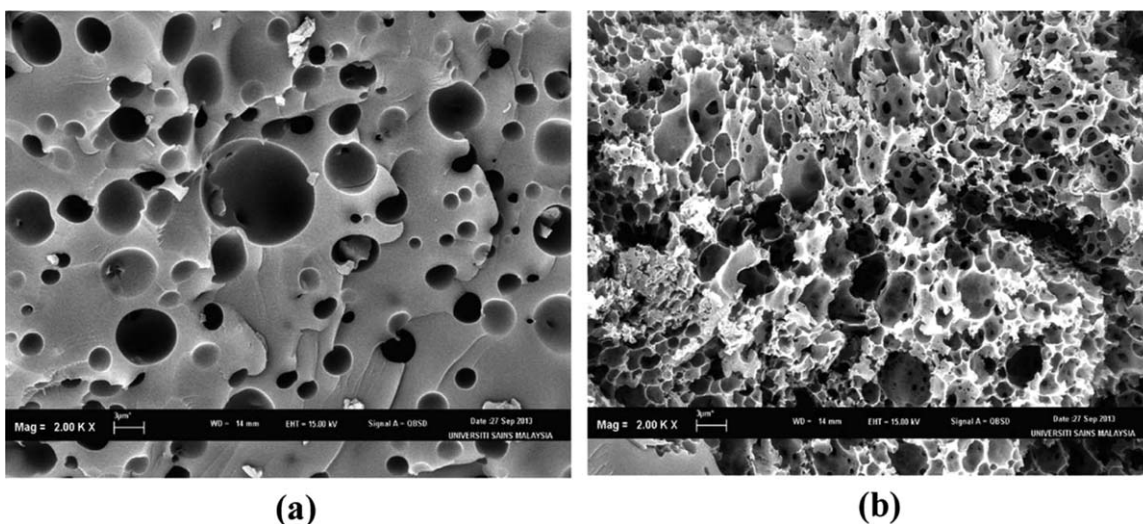


Figure 11. Scanning electron microscope (SEM) images of (a) virgin polyester, PAI2 and (b) photocrosslinked polyester, CPAI2.

photocrosslinked PAI2s were designated as CPAI2-0.2, CPAI2-0.5 and CPAI2-3 representing the time of UV irradiation: 0.2 hours, 0.5 hours and 3 hours, respectively. The spectra of CPAI2-0.5 and CPAI2-3 were observed to be much broader and the peaks were shifted toward much upfield as compared to that of PAI2. Comparing the spectra of both CPAI2-0.5 and CPAI2-3, the peaks of CPAI2-3 were even broader and shifted more upfield. In order to understand the photocrosslinked behavior of the polyesters, UV irradiation was done on the monomer, 5E bearing the benzylidene group involved in the photocrosslinking. Upon UV irradiation at different time intervals, 5E was photocrosslinked to form 5CE-0.5 (0.5 hours) and 5CE-3 (3 hours) as shown in Figure 7. The asymmetric 5CE showed 10 proton signals after complete photocrosslinking as compared to the symmetric 5E with 5 proton signals. This observation suggested that only one of the olefinic groups of benzylidene was involved in the photocrosslinking reaction. Similarly to the spectrum of CPAI2-3, after cross-linking, the proton peaks of 5CE-3 shifted to upfield. H5 (7.14 ppm) from 5E was shifted to 6.63 ppm (H5') in 5CE-3. The peaks at 7.05 and 6.34 ppm were assigned to H8' and H9' of 5CE-3. This was in agreement with the shifting of signals for Ha (7.34 ppm) to Ha' (6.69 ppm) when PAI2 cross-linked to form CPAI2 as well as presence of Hb' at 6.45 ppm. The signals of Ha' and Hb' were weakly seen in CPAI2-0.2. However these signals had greater intensities in CPAI2-0.5 and CPAI2-3. The rest of the proton signals could not be identified because of the broadening nature. For 5CE, two weak proton signals at 3.85 (H6') and 4.03 ppm (H7') (Figure 7) were attributed to the proton signals of cyclobutane ring formed from the photocrosslinked reaction. These weak protons signals were not seen in CPAI2 which may be because of the broadening effect of proton signals after the photocrosslinking and they may have also been overlapped with the alkyloxy proton in CPAI2.

Fluorescence Properties of PAI1-PAI5

The emission spectra of PAI1-PAI5 (Figure 8) were recorded by exciting the polyesters in their corresponding absorption band

by using chloroform as the solvent ($1 \times 10^{-5} M$). Since the absorption spectra of PAI1-PAI5 were similar, all the polyesters were recorded under the excitation of 340 nm. The existence of photoresponsive behavior of the polyesters can be evidenced by the emission spectra. The emission spectrum of PAI2 before UV irradiation showed maximum emission wavelength at 483 nm. However, after irradiation with UV light at various time intervals, a red shift toward higher wavelength of 495 nm was observed. Fascinatingly, the fluorescence intensity was found to be increased intensively upon subsequent UV irradiation. Photocrosslinking of the polyesters induced rigidity of the whole polymeric network and restricted the molecular rotation to an extent that may cause π to π stacking that led to the increase of fluorescence intensity. From Figure 9, PAI2 initially exhibited weak fluorescence in solution, and after photocrosslinking CPAI2 showed highly intense emission which further supported the increasing intensity trend in the emission spectrum. As shown in Figure 10, the fluorescence intensities before (0 s) and after UV irradiation for 1300 s were found to be not much affected by the difference of the alkyloxy chain lengths.

Morphological Studies

Scanning electron microscopy (SEM) was used to obtain high resolution images which enable the visualization of morphological information without losing any accuracy during analysis. From Figure 11(a,b), a representative polyester, PAI2 was characterized to understand the morphology of both the virgin (PAI2) as well as the photocrosslinked polyester (CPAI2). As observed in the SEM images, the virgin PAI2 had smoother surface while CPAI2 showed rougher surface with more voids indicating the presence of cyclobutane ring formed from the $2\pi + 2\pi$ cycloaddition. The morphology of CPAI2 was found to be in thread-like or fiber-like texture connecting from one to another.

CONCLUSIONS

A series of soluble photoluminescent polyesters namely poly {bis(4-hydroxybenzylidene)acetone-5-(*m*-alkoxyisophthalate)} ($m = 10, 12, 14, 16, \text{ and } 18$), PAI1-PAI5 were successfully synthesized

and characterized. The thermal stabilities of **PAI1-PAI5** were greatly related to the length of the alkyloxy chains as the chain length increases thermal stability decreases. During successive irradiation of UV light, the decrease in the intensity of the $\pi \rightarrow \pi^*$ transition of the benzylidene chromophore with the absorption at $\lambda = 333.5$ nm was attributed to the dimerization of olefinic double bond in the polymeric chain leading to the formation of a cyclobutane ring via $2\pi + 2\pi$ cycloaddition. The photocrosslinking was further confirmed via FT-IR and ^1H NMR techniques. It was found that the time taken for maximum photocrosslinking of **PAI1-PAI5** was independent of the length of the flexible alkyloxy chains and they were found to achieve maximum percentage of photocrosslinking (70–72%) at 1300 s. Interestingly, as the UV irradiation time was subsequently increased, the fluorescence intensity increased. The morphology of the photocrosslinked polyesters, **CPAI1-CPAI5** illustrated compact network of the polymeric system with thread-like or fiber-like texture as well as rough surface and more voids. The newly synthesized photoresponsive polyesters, **PAI1-PAI5** could be potential luminescent materials in the field of biomedical as well as in opto-electronic applications which are worth to be explored.

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