

EFFECT OF SYNTHESIS REACTION TIME AND DOPING ON UV/VIS ABSORPTION OF POLYENE COATING

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Abstract

Absorbance spectra and energy band gap of synthesizing polyene from dechlorination of polyvinyl chloride (PVC) with varying synthesizing reaction times and doping percentages were examined in this paper. The reaction time of dechlorination was varied from 30 minutes, 1 hour, 2 hours and 4 hours. Polyene obtained from the dechlorination reaction was doped with potassium iodide (KI). Sample for UV/Vis test was prepared in the form of film. Modified EO film as control sample showed that there is only a single absorption peak around 327nm. The C-Cl adsorption band link from source link polymer was observed at 422nm and all polyene film samples and new bands absorption were observed from 422nm to 590nm, and 683nm upon the increase in reaction time. The lower energy band gap was observed at 683.18nm absorption with 1.08eV.

Keywords: Energy band gap, PVC, Polyene, UV/Vis

Abstrak

Spektrum penyerapan dan jurang jalur tenaga hasil sintesis poliena daripada pendeklorinan polivinil klorida (PVC) dengan tempoh tindak balas sintesis dan peratusan pengedopan yang berbeza dikaji dalam kertas kerja ini. Tempoh tindak balas pendeklorinan dipelbagaikan dari 30 minit, 1 jam, 2 jam, dan 4 jam. Poliena yang terhasil daripada tindak balas pendeklorinan didopkan dengan kalium iodida (KI). Sampel untuk ujian UV/Vis disediakan dalam bentuk filem. Filem EO yang diubah suai sebagai sampel kawalan menunjukkan hanya satu puncak serapan sekitar 327nm. Rantai jalur penyerapan C-Cl daripada rantai polimer sumber diperhatikan pada 422nm, dan penyerapan kesemua sampel filem poliena dan jalur baharu diperhatikan daripada 422nm sehingga 590nm, dan 683nm semasa peningkatan tempoh tidak balas. Jurang jalur tenaga yang lebih rendah diperhatikan pada penyerapan 683.18nm dengan 1.08eV.

Kata kunci: Jurang jalur tenaga, PVC, Poliena, UV/Vis

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1.0 INTRODUCTION

Polyvinyl chloride (PVC) is constructed of repeating vinyl groups (ethenyls), which have one hydrogen replaced by chloride (Cl). It consists of homopolymer about 56.7% of chlorine by weight and the structure is primarily linear-to-tail configuration but various regularities may occur in the chain structure [1]. In order to make PVC resin highly applicable, it must be compounded with stabilizers to improve the stability when it is exposed to heat and light. Stabilizers often used are heavy metals such as barium, tin, cadmium, and lead [2]. Due to its hard and tough properties, the flexibility of PVC can be improved with the

addition of plasticizers, such as high molecular weight organic esters, which can convert PVC resin into films, tubing and wiring insulation. An example of plasticizers used is Di-2-ethylexyl phthalate (di-octyl phthalate, DOP). Sometimes, Phosphate esters, such as tri-cresyl, are used as plasticizers to avoid the increase in flammability, which is caused by phthalate and other purely organic plasticizers.

It has valuable properties in chemical resistance and low flammability and for the drawback of PVC. However, it has low thermal stability at processing temperature [3]. A study by Balci *et al.* (2004) reported that PVC is a common insulating polymer but degrades easily via dechlorination through the

loss of hydrochloric acid (HCl) when exposed to heat, energetic particles or photons [4]. Under definite conditions, dehydrochlorination of PVC may cause alternating single and double bonds carbons or conjugated (polyene) structure.

Determination of energy band gap is important to determine the band gap of the electrons to jump from one band to another (valence band to conduction band) where it needs specific minimum amount of energy for the transition. The energy band gap of the conducting polymers was measured as the sun provides a vast amount of energy, but only a small portion of the incident sunlight is absorbed because of the large band gap of organic materials. The energy band model is the one of the most extensively used models to explain the electronic conduction instead of hopping and percolation mechanism and theories [5, 6]. Also provides the basis to understand whether a particular material is conductor, semiconductor or insulator.

Faris *et al.* (2015), Smith and Hashemi (2006) have discussed the mechanism of electron movement in the energy band theory. According to Faris *et al.* (2015), two new orbitals of high (antibonding) and low (bonding) energy are produced through overlapping of two compatible atomic orbitals when they are brought closer to each other. In real structural phenomenon there is usually a gap between the top of the occupied valence band and the unoccupied conduction band known as band gap. It is assumed to be essential to produce a net electron drift from valence band to conduction band to observe conduction in such material. Material of band gap ($\geq 1.5\text{eV}$) is declared as an insulator. If this gap is of moderate size ($\leq 1.5\text{eV}$) the movement of electrons will occur from valence to conduction band leaving a hole in their valence band and that material declared as semi conductors. Within this region, some materials with a very small band gap in which some electrons always get excited to conduction band by thermal means. They area called intrinsic semi conductors. The energy band diagram in polymeric materials is shown in Figure 1.

For the conductor, the band is partially filled without any energy gap at all. In these cases, conduction is very easy because electrons require only a small amount of energy to cause a net drift of electron. In cases of bi-phasis or heterogeneous system in which only one phase is conducting, the conductivity depends upon concentration of conducting phase. Conjugated polymers having alternate π -electrons display semi-conducting properties as a consequence of their low energy of optical transitions, low ionization energies and high electron affinities [6, 7].

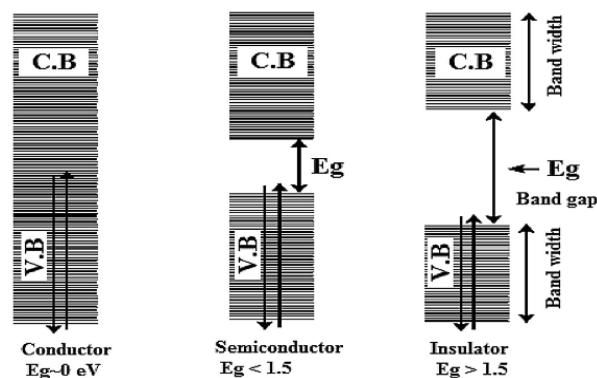


Figure 1 Energy band diagram for an intrinsic elemental of conducting polymers [8]

Another mechanisms explained by Smith and Hashemi (2006), where electron movement from the covalent band to conduction band. Above the valence band there is a forbidden energy band gap in which no energy states are allowed and which is 1.1eV for silicon at 20°C. Above the energy band gap there is an almost empty (at 20°C) for conduction band. When an electron is excited across the energy gap into the conduction band, two charge carriers are created, which is a negatively charged electron and a positively charged hole. Finally both electrons and holes carry electric current [9].

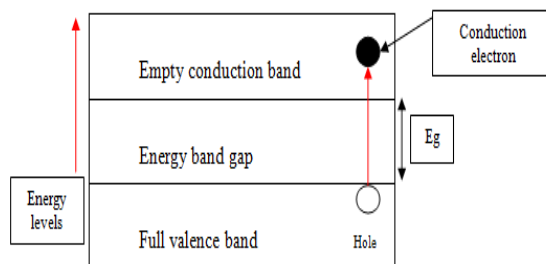


Figure 2 Energy band diagram when an electron is excited across the energy band gap [9]

The band gap of typical conjugated polymers ranges between 2.0eV and 3.5eV, which limits the possible absorption of solar energy. Chen and Hsu (2011) stated that a band gap of 1.1eV (1100nm), 77% of the incident solar energy on the Earth's surface can be absorbed. Therefore, it is necessary to design and synthesize new low band gap conjugated polymers that can absorb more of the solar energy. As a result, the absorption spectrum and energy levels of conjugated polymers can be tuned by functionalization and many synthetic efforts have been made to develop new low band gap polymers [10].

According to Rakovic (1996), another conducting polymers such as poly (p-phenylene), poly (p-phenylenesulfide), poly (p-phenylenevinylene)(PPV), polythiophene, polyaniline and the others, which are

based on aromatic ring systems unless doped, remain as insulators with a band gap $> 1.5\text{eV}$ [11]. Figure 3 shows several structures of conducting polymers.

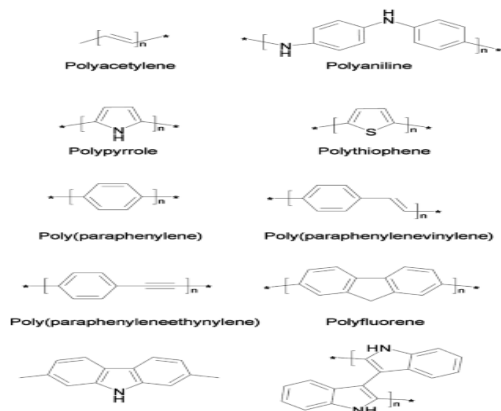


Figure 3 Structure of conducting polymers [12]

2.0 EXPERIMENTAL

2.1 Materials

Polyvinyl chloride (PVC) powder with grade HP65 is a product of Malaysian Electrochemicals industry (Malaysia). Other materials are tetrahydrofuran (THF) (MERCK Malaysia), polyethylene glycol (PEG) (R&M Chemicals Malaysia), potassium hydroxide (KOH) (R&M Chemicals Malaysia), and potassium iodide (KI) (SYSTEM Malaysia).

2.2 Dechlorination of Polyvinyl Chloride (PVC) and Doping

Dechlorination of PVC was done in three-neck flask reactor that consisted of condenser. PVC were dissolved in tetrahydrofuran (THF) and base solution (potassium hydroxide dissolved in PEG). Base solution of PEG was then dropped into the polymer solution to form a homogeneous system. The transparent color of the solution changed from yellow to dark red and finally to a dark color. The black powders were filtered using vacuum pump. Finally, the filtered particles were then washed with distilled water and methanol. Then, PEG, base and salt was successively extracted in a soxhlet extractor with methanol. The clean products were dried under vacuum conditions at 60°C for 24 hours before being characterized. Reaction time of dechlorination was varied from 30 minutes, 1 hour, 2 hours and 4 hours reaction time. Doping of polyene powders was carried out by immersing in 5%. From the previous study, 2 hours doping with 5% KI showed the highest value of conductivity; hence it was then proceeded with doping with 10%, 20% and 30% of potassium iodide (KI) [13].

2.3 Preparation of Polyene with Modified EO Coating

The polyene powder had been dispersed in modified EO and was cured through ultraviolet (UV) light conveyer machine (F300S MODULAR 6 ULTRAVIOLET), with an intensity of 0.107 W/cm^2 (measured by an IL 390B Light Bug radiometer from International Light Inc.), lamp with 80 W, energy at 0.4400 J with 2 times at 37 seconds per pass [14].

2.4 Characterizations

UV/Visible spectra measurements were carried out on LAMBDA 35/UV/VIS DOUBLE BEAM SPECTROMETER. The purpose of this test is to determine the absorbance bands of lights that for the synthesized polyene sequences. From the absorbance reading, the energy band gap can be calculated and be plotted with $((ah\nu)^2 \text{ vs. } h\nu/\lambda)$. Where a is absorbance from $-y$ axis, h is a plank constant $= 4.135 \times 10^{-15}$, and ν is a velocity $= 3.0 \times 10^8$, λ is a wavenumber from $-x$ axis.

3.0 RESULTS AND DISCUSSION

3.1 UV/Vis Absorption

Characterization on UV absorbance properties of polyene was investigated in the range of $300\text{nm} - 900\text{nm}$ to determine their wavenumber absorption maximum. It would indicate a series of conjugated polyene segments as series of absorption peaks. The band gap energy of polyene coated with modified EO film was determined using band gap energy equation from band gap energy $(E) = h \nu/\lambda$. The polyene powder was then prepared as thin film. The reaction times for synthesizing polyene were varied from 30 minutes, 1 hour, 2 hours and 4 hours. Figure 4 illustrates the UV/vis spectra of polyene film at different reaction times of synthesizing polyene.

Modified EO film (control sample) showed that there is only a single absorption peak around 325.88nm due to conjugated segments in the modified EO [15]. This material is held by C-H (carbon-hydrogen) linkages and ether and ester links having very strong absorption at UV region [15]. The absorption bands at 387.42nm were observed for vinyl chloride and all polyene film samples as material has C-Cl link from the vinyl chloride chain. As the reaction time increases, the overall absorption increased remarkably with a broad absorption band. Then the bands shifts from peaks at 424.10nm to 560nm , 683.18nm and at the maximum absorption wavenumber at 800nm around the IR region.

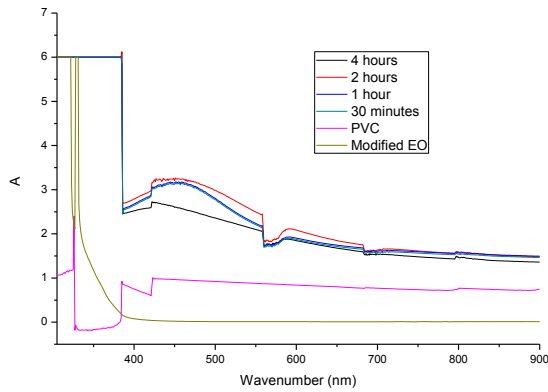


Figure 4 UV/Visible absorption spectra of modified EO film, vinyl chloride resin and 30 minutes, 1 hour, 2 hours and 4 hours treated polyene film

Four obvious peaks were observed since these phenomena indicate that long conjugated polyene segments were formed through the elongation of the short conjugated C=C bonds as the dechlorination reaction time increased [16]. It is obvious that the absorption of the polyene sample is almost located at the same positions; 424.10nm, 560.10nm and 683.18nm as their corresponding monomers, which absorption depends on the type of conjugated length of polyene chains or chromophore, where the polymers with the same chromophore have similar absorption peaks [17].

On the other hand, for the 2 hours reaction time of polyene sample, absorption peaks are more significant and distinct. 2 hours is the optimum reaction time for the formation of conjugated bonds (C=C). For 4 hours reaction time, the absorption showed narrower peaks, which were inferred that the long polyene sequences were shortened by side reaction if the reaction time was too long [18, 19], and some of the conjugated was diminished due to long reaction time [20]. Figure 5 and 6 shows there is only single peak were observed on UV/Visible absorption spectra for all samples with 5% of KI doping and 2 hours sample with 5%, 10%, 20% and 30% of KI doping, respectively.

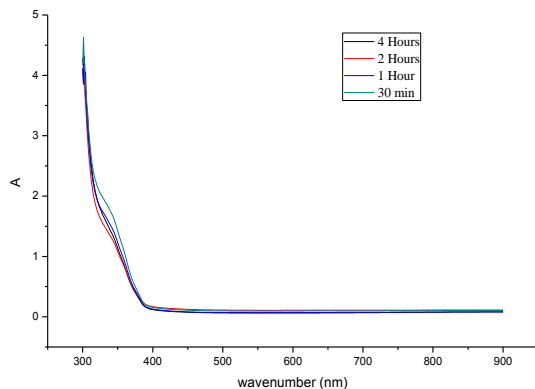


Figure 5 UV/Visible absorption spectra of all samples with 5% KI doping

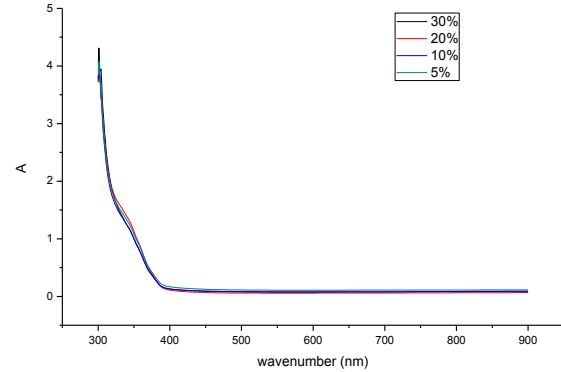


Figure 6 UV/Visible absorption spectra of 2 hours sample with 5%, 10%, 20% and 30% KI doping

The intensity of absorption in UV/vis implies the number of photons absorbed in the samples. This meant that 2 hours polyene sample, which absorbed the highest intensity, has the maximum number of photons absorbed, which causes electronic transition of pi-orbital to its excited states in greater frequency, proving that it possesses the highest number of conjugated bond.

3.2 Determination of Energy Band Gap and Number of Conjugation

From the UV/visible spectra, the energy band gap graph was plotted with the following graph ($(ah\nu)^2$ vs. $h\nu/\lambda$), in order to determine energy band gap of polyene samples. The intercept on the energy axis ($h\nu/\lambda$), on extrapolating the linear portion of the curves to zero absorption value would give the value of energy band gap [21]. Determination of energy band gap is important so as to determine how much energy is needed from the sun for conduction, as well as how much energy is generated. It means the band gap of the electrons jump from one band to another (valence band to conduction band) where it needs a specific minimum amount of energy for the transition.

All results show the same energy band gap at the same wavenumber (nm) whereby the reduction in energy band gap corresponds to high wavelength absorption maximum, which indicates high absorption of light. Besides that, the energy band gap was also found at four different wavenumbers for all samples (Figure 7), which infers that the long polyene sequences formed different chain lengths of polyene due to different degrees of dechlorination side reactions.

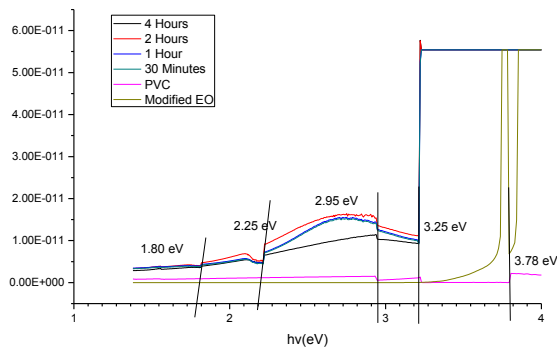


Figure 7 Energy band gap spectra for undoped film samples

The lowest energy band gap (1.80eV) was located at 638.2nm for all polyene samples and for the modified EO, it shows only single peak absorption at 325.8nm with 3.78eV. The number of conjugation of polyene has been calculated based the UV/Vis spectra and according to Woodward-Fieser rules. The parent diene of a whole series is 214nm (from one to four conjugated double bonds), and each additional conjugated bond should be added with 30nm [22]. The lowest energy band gap is at high wavenumber at 683.18nm, which indicates the most feasible electron transfer and the lowest energy difference in electron volts between the top of the valence band and the bottom of the conduction band of the polyene system. Series of energy band gap with 3-peak absorption wavelength were observed for the polyene system while vinyl chloride resin has 2 absorption peaks with a peak for EO resin. All coating with polyene samples exhibited the existence of EO peak, 325.88nm UV/vis spectrum which indicates the C-Cl absorption band.

3.3 Energy band gap for Polyene Film with 5% KI Doping

The energy band gap for doping samples with 5% KI showed that the lowest energy band gap is at 2 hours of reaction time (3.05eV). A lower energy band gap leads to better electron transfer between the valence bands (EV) to conduction band (EC). Referred to Table 1, the lowest energy band gap of 3.05eV was found for the polyene sample for 2 hours of doping with 5% KI samples. This means that the 2 hours samples have the lowest energy difference in eV values between the top of the valence band and the bottom of the conduction band in insulators and semiconductors. Polyene formed from 30 minutes of reaction time has the highest value of energy band gap ~3.22eV. The value did not vary much as the conjugated form with KI had been saturated by the electron transfer mechanism due to doping reaction [23]. Figure 8 shows the energy band gap for all samples with 5% of KI doping.

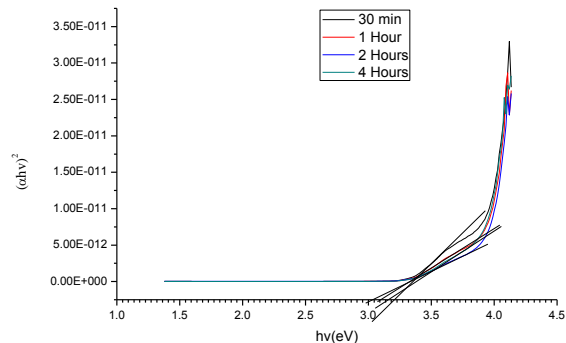


Figure 8 Energy band gap spectra for all samples with 5% KI doping

Upon doping, only one absorption of the energy band gap was recorded. This may be due to long polyene sequences shortened by side reaction and the diminishing of some conjugated bonds. The decrease of energy band gap eV between doping samples may be due to the increased I⁻ doping content. KI content is responsible for the production of localized states in the optical band gap [21].

Table 1 Energy value (eV) for all samples and number of conjugation with 5% KI doping

Sample	Wavenumber (nm) λ	Number of conjugation	Energy band gap (eV)
30 Min	393.5	6	3.22
1 Hour	403.5	6	3.15
2 Hours	408.4	6	3.05
4 Hours	404.4	6	3.13

These overlaps are responsible for decreasing energy band gap when KI content is increased in the polymer matrix [24] where the decrease in the optical band gap results in an increase in the degree of disorder in the polyene films.

3.4 Energy Band Gap for 2 hours Sample Doping with 5%, 10%, 20% and 30% KI

Energy band gap of 2 hours polyene samples with 5%, 10%, 20% and 30% doping showed little difference as conjugated bond started to reduce the delocalization of electronic transition due to the doping introducing ion mobility interference to the unsaturated pi double bond centre; hence this tends to average out the electronic cloud at this centre during the doping process. From Table 1 and Table 2, it can be seen that the energy band gap for doped samples does not show much difference on energy

band gap value (eV), where it was found that eV is around 3.15eV to 3.05eV with wavenumber around 393.5nm to 408.4nm.

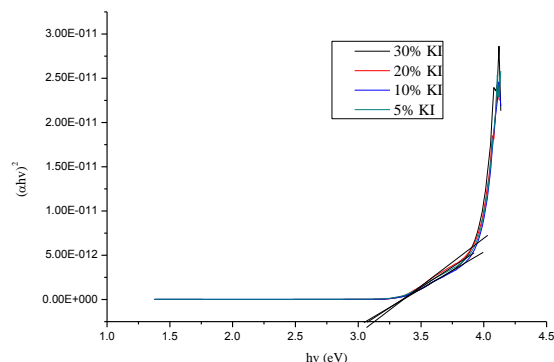


Figure 9 Energy band gap spectra for 2 hours sample with 5%, 10%, 20% and 30% KI doping

Adhikari, S. and Banerji, P. (2010) report that Iodide (I⁻) as an anion dopant may increase the conductivity, which has the ability to increase the conductivity of PANI [25]. In the result obtained from Table 2, it was found that without iodide doping, the energy band gap of ~2.95eV for undoped polyene has about similar electron transfer between the conjugated polyenes. No absorption peaks at 683.18nm, 60.10nm and 424.10nm were observed; only one peak was exhibited (Figure 9). This is because iodide has stronger electronegativity, which meant it has strong electron withdrawing ability to transfer the electron of pi (π) orbital of all the other polyene sequences, and forms into one conjugated polyene forming a centralize structure hence stabilizing the eV to about 3.0eV.

Table 2 Value of energy band gap for 2 hours sample and number of conjugation with 5%, 10%, 20% and 30% KI doping

Sample	Wavenumber (nm) λ	Number of conjugation	Energy band gap (eV)
Undoped	424.1	7	2.95
5%	408.4	6	3.05
10%	408.4	6	3.05
20%	403.4	6	3.08
30%	401.0	6	3.12

4.0 CONCLUSION

From this study, four absorption peaks was observed from all polyene which indicates that long conjugated polyene segments were formed through

the elongation of short conjugated C=C bonds as the dechlorination reaction time increased. Upon relating of peak absorption spectra to energy band gap, the lowest energy band gap (1.80eV) was located at 638.18nm for undoping sample was observed. For modified EO, only single peaks absorption was found at 325.88nm which is 3.78eV. Doping of polyene with KI had slightly reduced its energy band gap. Greater electron mobility of pi (π) orbital of unsaturated conjugated polyene and had reduced is energy band gap.

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