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(Research Article)

Synthesis, characterization and photostability study of triazole derivatives

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Abstract

heterocyclic derivative contain triazole ring was synthesized and characterized the product and their structures by infrared spectroscopy, ^{1}H-NMR, ^{13}C-NMR and instrumental techniques. Compound (4) was synthesized by reacting of Schiff base (3) with an three moles of alkyl halide (*p*-phenyl phenacyl bromide). Final product played an important role in photostabilizer of polymer (PS), and showed the activity as a photostabilizer when exposed to UV light (300 hours).

Keywords: Triazole; Photostability; Heterocyclic compounds; Schiff base; Synthesis

1. Introduction

Heterocyclic compounds, which have at least 2 different atom types in the ring, are the most prevalent form of organic substrate [1-3]. Triazole, likewise famed as pyrrodiazole, it is an example of a class of organic heterocyclic compounds that contain nitrogen. It's one of five diunsaturated ring types , containing three carbon atoms in the neighboring site and two in the non-adjacent site[4- 6]. Sulphur-containing triazole heterocycles are also important because of their useful applications, which provide all of the essential components for a strong photostabilizer [7, 8], because PS has been shown to oxidize when exposed to natural sunshine [9]. The photostabilization process includes using additives (preferably with a heterocyclic ring) and applying them to Polystyrene (PS) to achieve specific features in modified films, as an example improved thermal stability, multiphase physical reactions, compatibility, impact response, flexibility, and rigidity [10].

2. Instruments

Melting points were measured by using hot stage (FALC melting point device). Fourier-transform infrared spectroscopy were measured by using instrument BRUKER (FT.IR), KBr disc ^{1}H NMR, ^{13}C NMR, Microscope, SEM, and AFM.

3. Chemicals

Chemicals were analar or pure grade, and solvents were dried and purified.

3.1. Synthesis

3.1.1. Preparation of 4-hydroxybenzohydrazide (1).

4-hydroxy methyl benzoate (0.03 mol, 5 g) in 25ml ethanol is refluxed for 4 hours in a round bottom flask with hydrazine hydrate (0.10 mol, 5 ml). The precipitate formed was filtered, dried and then washed by ethanol [11].

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3.1.2. Preparation of 4-(4-amino-5-mercapto-4H-1,2,4-triazol-3 yl)phenol (2)

4-hydroxybenzohydrazide (0.03 mol, 4.8g) was added to mixture of dissolved potassium hydroxide (0.05 mol , 3 g) in (25 mL) of absolute ethanol. Mixture was cooled in ice bath with stirring. Add to this mixture (0.19 mole, 11.5 mL) carbon disulfide in small amounts with constant stirring. Overnight, at room temp., the reaction mixture was left to stir constantly. The potassium-2-(4-hydroxybenzoyl) hydrazinecarbodithioate formed and reacted with hydrazine hydrate (0.14 mol, 7 ml) with reflux for 8 hours. The mixture color changed to green beside to liberation of H_2S (tasted by lead acetate paper) and a homogenous solution resulted. By addition cold water (20 ml) and acidification with concentrated hydrochloric acid, a white solid was precipitated, filtered, ethanol-washed and left to dry [11].

3.1.3. Preparation of 4,4'-(((1,4- phenylenebis(methaneylylidene)) bis (azaneylylidene)) bis(5-mercapto-4H-1,2,4triazole-4,3-diyl))diphenol (3)

Compound (2) (2 mol, 0.5 g) with terphthaldehyde (1 mol ,0.3 g) were mixed in (10 ml) DMF and glacial acetic acid (three drops), then mixture was refluxed for about 10 hours. The Precipitate was filtered, dried and washed by ethanol [12].

3.1.4. Preparation of 2,2'-(((((1,4-phenylene bis(methaneylylidene)) bis (azaneylylidene))bis(5-((2-([1,1'-biphenyl]-4-yl)-2-oxoethyl)thio)-4H-1,2,4-triazole-4,3-diyl))bis(4,1-phenylene))bis(oxy))bis(1-([1,1'-biphenyl]-4-yl)ethan-1-one) (4)

In round bottom flask dissolving (1mol. 0.1g) from compound (3) in 10 ml of acetone with 2drops of DMF and (2mol. 0.04g) of K2CO3. When dissolved, added (4mol. 0.2g) from *p*-phenyl phenacyl bromide (4). The mixed solution was stirred overnight until being poured into ice water, filtered and left the precipitate to dry for next step [13]. As shown in scheme 1

3.1.5. Preparation of modified films

In round bottom flask dissolve (0.005g) of product (4) in 10 ml chloroform with 2drops of DMSO, after dissolving added (0.5g) of polystyrene for the round with stirring more than 3hours. After that poured into the glasses sink and left for 24 hours to remove all chloroform by evaporation technique [14].

4. Results and discussion

The synthesized compounds (1, 2, 3 and 4) are shown at figure 1. The following tables list the physical properties as well as FT-IR spectrum data.

Table 1 Physical feature of [2, 3, 4] Compounds

No.	Chemical Formula	Molecular Weight (g/Mol)	Color	Melting Point (C^0)	Yield (%)
2	$C_8H_8N_4OS$	208.24	White	240-242	94
3	$C_{24}H_{18}N_8O_2S_2$	514.58	Light yellow	Over300	79
4	$C_{80}H_{58}N_8O_6S_2$	1291.51	Yellow	248-250	80

The general synthesis method of (2, 3, and 4) compounds shown in Scheme 1. And evidenced by FT-IR device values that appearance of (1522-1591) cm^{-1} owing to (C=N) group of triazole ring, as well as of two other characteristic stretching vibration bands at (3309 and 3269) cm^{-1} belong to (NH_2) group, these bands disappeared when Schiff base formed due to formation of C=N at (1652). The FT-IR characteristic spectrum for chemicals [2], [3], [4] is shown in Table 2.

Table 2 FT-IR Spectrum data of [2, 3, 4] compounds

No.	Frequency numbers (cm^{-1})
2	(1522 cm^{-1}) for C=N of triazole ring. stretching (3309 cm^{-1} and 3269 cm^{-1}) for symmetric and asymmetric NH_2 and weak bands at (653 cm^{-1} , 2799 cm^{-1}) for C-S and S-H group respectively Figure 2
3	(3215 cm^{-1}) for O-H group , (3073 cm^{-1}) for C-H aromatic , (2926 cm^{-1} and 2801 cm^{-1}) for C-H aliphatic , (1652 cm^{-1} , 1591 cm^{-1}) for C=N exocyclic and endocyclic respectively. Beside to disappearance of (3309 cm^{-1} and 3269 cm^{-1}) for symmetric and asymmetric NH_2. Figure 3
4	(3005 cm^{-1}) for C-H aromatic, (1681 cm^{-1}) for C=O, (1629 cm^{-1}, 1555 cm^{-1}) for exocyclic and endocyclic C=N, and (1130, 833) cm^{-1} for C-O and *p*-position. Figure 4

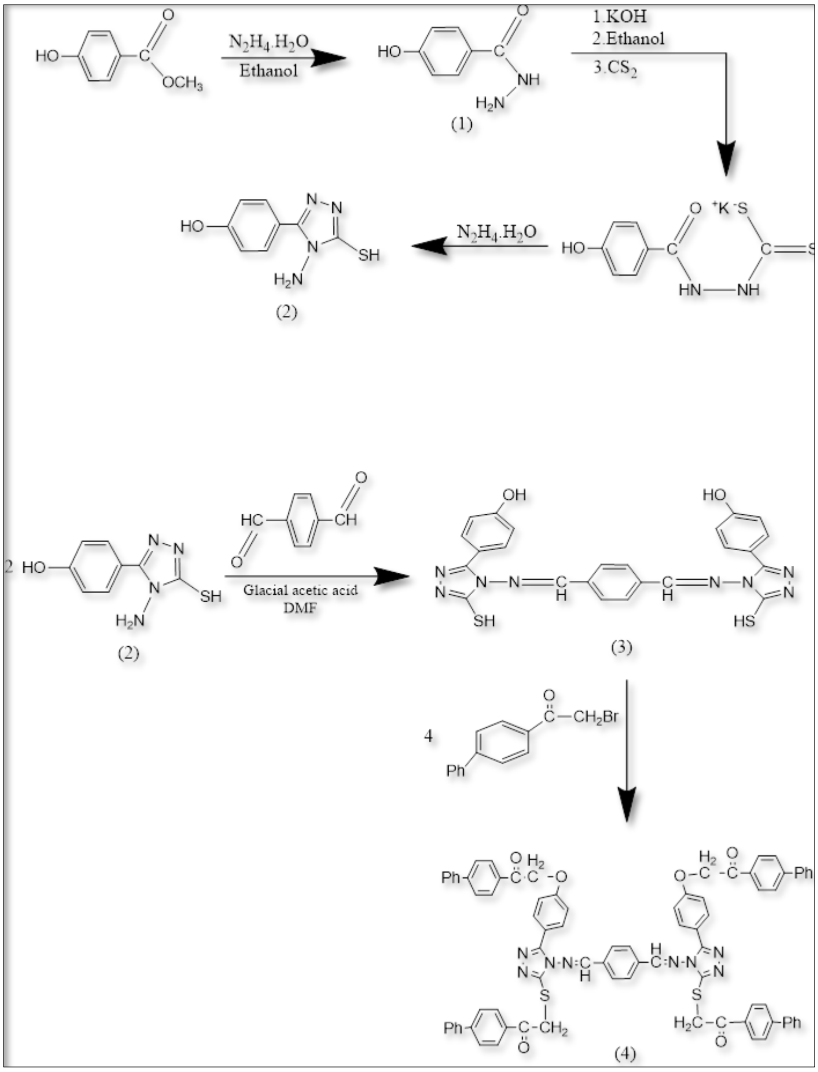

Figure 1 General synthetic pathway of (1, 2, 3, and 4) compounds

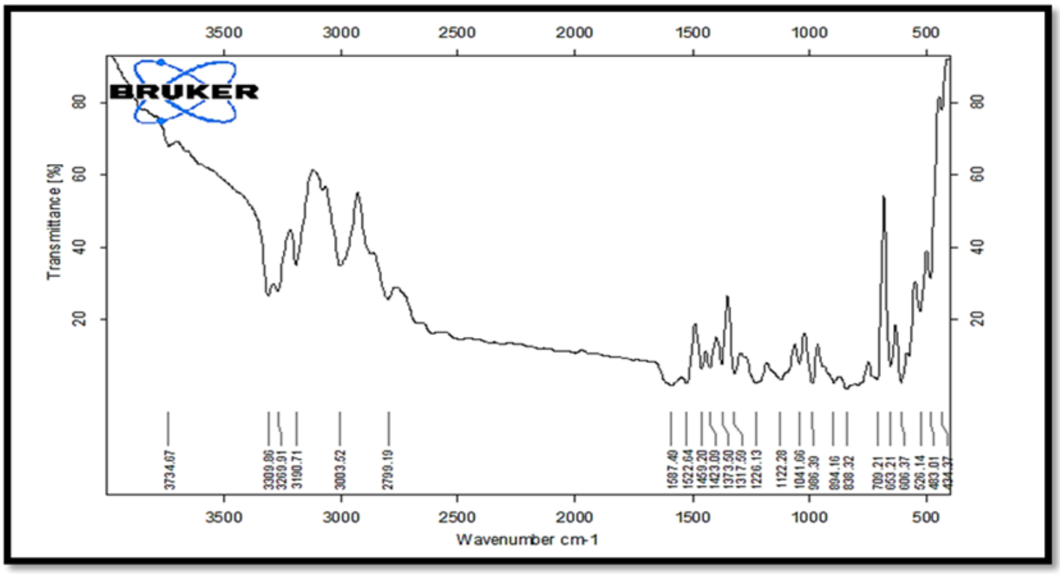

Figure 2 FT-IR of compound 2

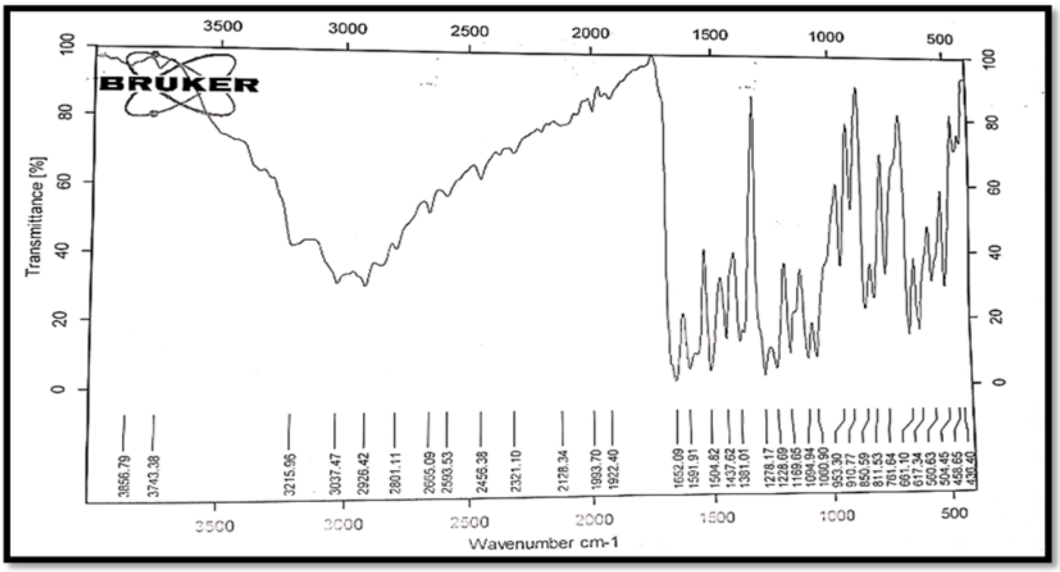

Figure 3 FT-IR of compound 3

5. Characterization on polystyrene

5.1. Using weight loss method

The formation of free radical moieties during photooxidation of polystyrene causes cross-linking of the polymeric chains. To find out how effective complex (4) is as stabilizer, the weight loss was estimated using equation [1] after the polystyrene was irradiated for 300 hours.

Weight loss % = [(W1 -W2) / W1] 100 % …… [1]

The graph between weight loss and irradiation time show that polystyrene with additive more effective than polystyrene alone.

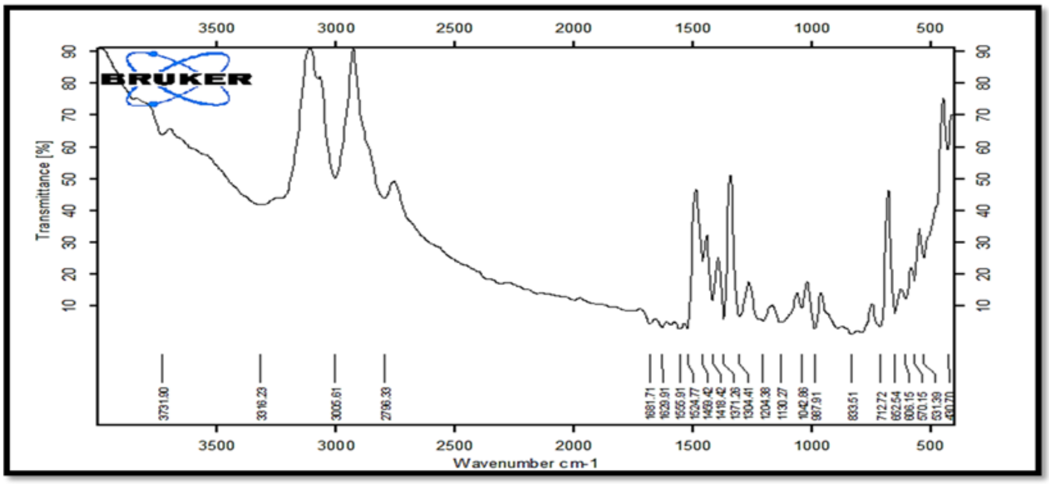

Figure 4 FT-IR of compound 4

Table 4 Measurement of weight loss for polystyrene with additives

Additives	**Irradiation time (hours)**						
	0	50	100	150	200	250	300
polystyrene	0	0.831	0.869	1.025	1.284	1.37	1.549
PS + 4	0	0.72	0.737	0.852	0.89	0.891	1.09

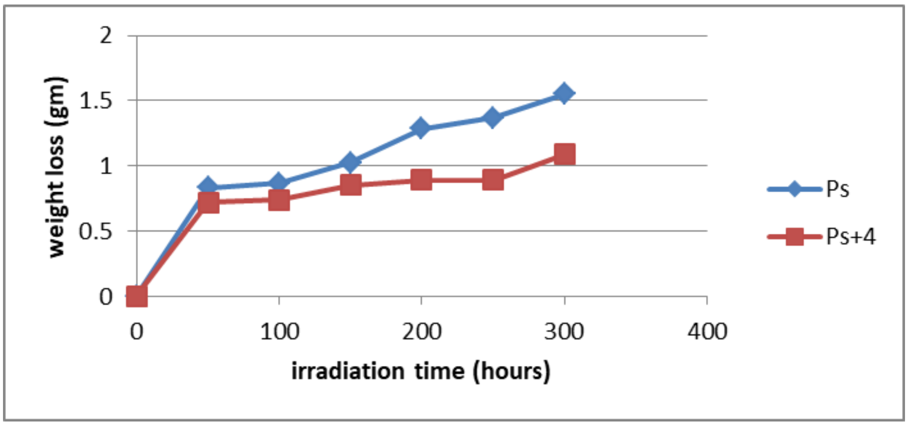

Figure 5 Weight loss vs. irradiation time of polystyrene with (4) compound

5.2. By Light Microscope, AFM, SEM, and EDX

Light Microscopes, Atomic Force Microscopes, and Scanning Electron Microscopes (SEM) are all utilized to get a clear image of the surface of polystyrene films. EDX is also give additional information about the chemical component found in polystyrene film. From the photos below, it can be seen that when polystyrene was irradiated, it developed cracks, roughness, and a change in color owing to photo-degredation, which caused polystyrene bonds to break. However, by adding (4) compound to the polymer, the surface became softer and had fewer cracks. Figure 6, 7, 8, 9

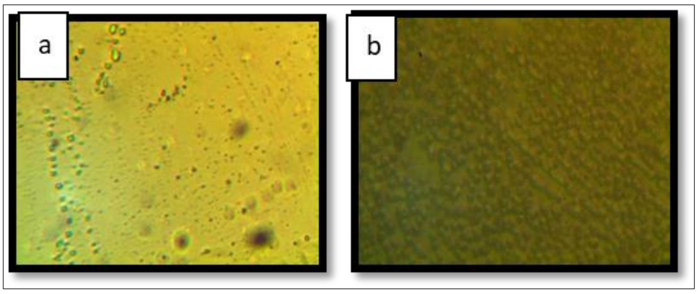

Figure 6 Light Microscope image of (a) polystyrene (b) PS + 4

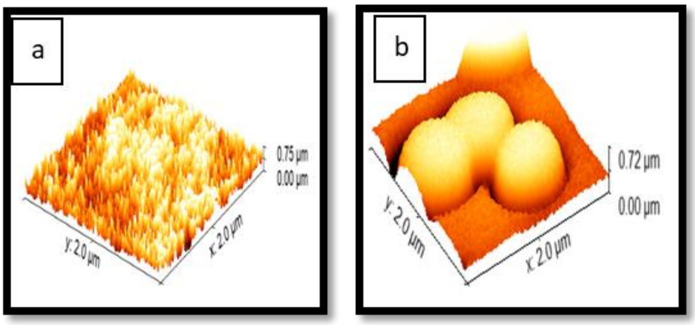

Figure 7 AFM image of (a) polystyrene (b) PS+ 4

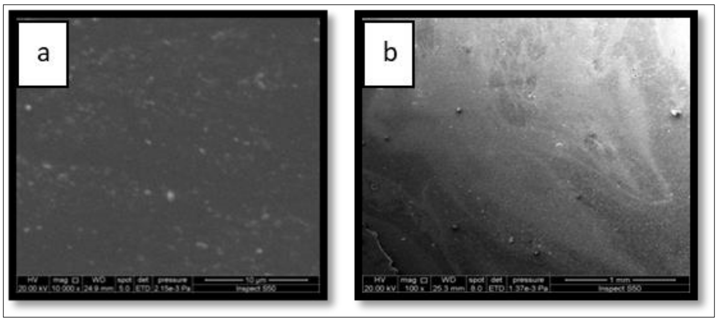

Figure 8 SEM image of (a) polystyrene (b) PS+ 4

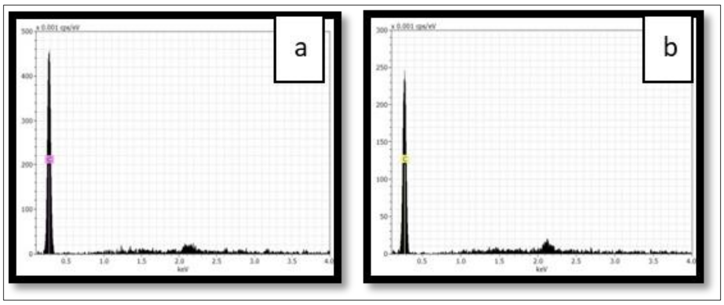

Figure 9 EDX of (a) polystyrene (b) PS +4

6. Conclusion

From synthesized (4) compound and there characterization by (Infra-red , hydrogen and carbon nuclear magnetic resonance) spectrum data and applied on polystyrene film and from Microscope, AFM, SEM, and EDX conclude the polymer with this compound efficient more than polymer without additives that ensure the (4) compound can be used as photostabilizer of polystyrene.

Compliance with ethical standards

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Disclosure of conflict of interest

The authors declare that there is no conflict of interests regarding the publication of this paper.

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