

**FULL PAPER**

# New photostabilizers for poly (vinyl chloride) derived from heterocyclic compounds

Mohammed Alwan Farhan<sup>a,\*</sup>  | Olfat Abed Nief<sup>a</sup> | Wassan Baqir Ali<sup>b</sup><sup>a</sup>Department of Chemistry, College of Science, Mustansiriyah University, Baghdad, Iraq<sup>b</sup>Department of Chemistry, College of Science, Diyala University, Diyala, Iraq

In this work, the photo-stabilization of poly (vinyl chloride) was studied using new compounds such as Schiff base stabilizers derived from heterocyclic compounds, with symbols [I1-I4] which were used as additives to prevent the PVC photodegradation. Many samples have been prepared as films for this purpose by adding the additives to poly (vinyl chloride) with varying weight percentages of additives and thicknesses. The photostabilization activities for these new compounds were measured by detecting the carbonyl group with irradiation time (the absorption band of carbonyl was determined at 1724-1772  $\text{cm}^{-1}$ ). FT-IR spectra PERKIN ELMER SPEACTUM-65, JASCO used to determine the absorption spectra of PVC films, also the viscosity average molecular weight ( $\bar{M}_v$ ), the average number of chain scission (S), and the degree of deterioration ( $\alpha$ ) were studied. Scanning Electron Microscope (SEM) and light microscope are also used to test the polymeric films surface. We discovered that in the existence of additives, the rate of photostabilization follows the trend:-

I4 &gt; I3 &gt; I1 &gt; I2 &gt; PVC

According to the experimental results, a UV absorption peroxide decomposer has been proposed for mechanisms.

**\*Corresponding Author:**

Mohammed Alwan Farhan

Email: [Alshamary\\_198840@yahoo.com](mailto:Alshamary_198840@yahoo.com)

Tel.: +07713498091

**KEYWORDS**

Photostabilizers; UV irradiation; Schiff base; hetero cyclic; chain scission; PVC.

**Introduction**

Poly vinyl chloride (PVC) has special mechanical and physical properties, with chemical formula  $(\text{CH}_2\text{-CHCl})_n$ , it is colorless rigid material. The PVC relatively has high density and low softening point and likewise, it is mostly used as a thermoplastic material [1]. It is commercially produced by a number of techniques but generally emulsion, suspension, solution and bulk polymerization of vinyl chloride. PVC has the advantage of being flame resistant, unlike many other polymers. It is used as rigid pipe in about 50% of cases [2].

Vinyl chloride polymers have a considerable contribution in the plastic

market due to their low production cost. Poly (vinyl chloride) products' high performance has boosted their use in building, especially in exterior application of buildings side such as windows and covering structures. PVC mostly makes the use of pipes and sidings in North America, while it is primarily applied as pipes and window frames in Asia and Europe. However, PVC subjected to photodegrades when exposed to long periods of sunlight or high heat [3].

The physical and mechanical properties of PVC may change as a result of this photodegradation problem [4]. PVC chains may form conjugated double bonds (polyene structure) as a result of dehydrochlorination, resulting in discolouration of PVC [5]. Cross-

linking and a lowering in molecular weight likewise happen for PVC chains because of photodegradation operations [6]. The poor stability of PVC hinders its use in areas exposed to direct sunlight, so it should be photo-stabilized in order to be suitable for usage in harsh conditions.

Photostabilization is the process of slowing or stopping a polymer's photodegradation. Because many commonly used plastics deteriorate when exposed to sunlight, photostabilization of polymers is significant [7]. PVC stabilizers are divided into two types: primary stabilizers that deactivate allylic chlorides produced during photo-degradation of PVC chains, and secondary ones which act as scavengers of chloride radicals and hydrogen chloride [3].

As PVC additives, a variety of organic compounds have been used. Plasticizers and the other commercial stabilizers can be applied to improve the PVC photostability. A UV stabilizer should have a high solubility and compatibility with the polymer in addition to the ability to successfully protect polymeric materials [8]. Photostabilizers included Schiff base compound [9-11] aromatics material [12-14] and heterocyclic [15-17]. Other materials used as additives

stabilizer include complexes metals and inorganic salts [18-25].

In this work, the PVC photostabilizer was done by using heterocyclic and Schiff base compounds. Schiff bases stabilizes PVC by various mechanisms ways, for example screener, UV absorption, and radical scavenger [26,27]. In this research, four compounds [I1-I4] have been synthesized by the method previously described by Mohammed *et al.* [28] and their uses as a photostabilizers for poly(vinyl chloride).

## Material and methods

### Materials

3-((5-mercapto-1,3,4-thiadiazol-2-yl)imino)indolin-2-one (I1)

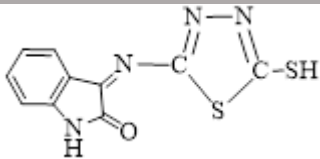
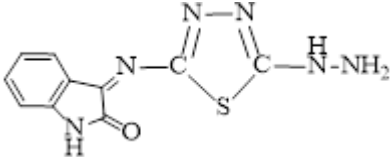
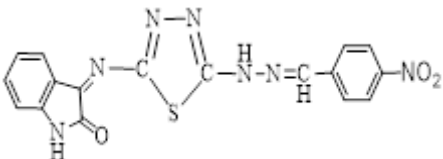
3-((5-hydrazinyl-1,3,4-thiadiazol-2-yl)imino)indolin-2-one (I2)

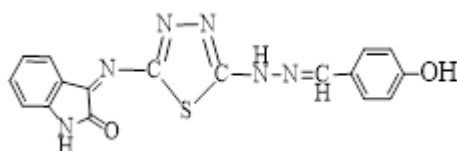
3-((5-(2-(4-nitrobenzylidene)hydrazinyl)-1,3,4-thiadiazol-2-yl)imino)indolin-2-one (I3)

3-((5-(2-(4-hydroxybenzylidene)hydrazinyl)-1,3,4-thiadiazol-2-yl)imino)indolin-2-one (I4)

These compounds were synthesized by the method previously described by Mohammed *et al.* [28], (Table 1).

**TABLE 1** Symbols, name, and structures of the synthesized compounds

Symbol Comp.	Structural Formula of Compound	Name of Compound
I 1		3-((5-mercapto-1,3,4-thiadiazol-2-yl)imino)indolin-2-one
I 2		3-((5-hydrazinyl-1,3,4-thiadiazol-2-yl)imino)indolin-2-one
I 3		3-((5-(2-(4-nitrobenzylidene)hydrazinyl)-1,3,4-thiadiazol-2-yl)imino)indolin-2-one
I 4		3-((5-(2-(4-



hydroxybenzylidene)hydrazinyl)-  
1,3,4-thiadiazol-2-  
yl)imino)indolin-2-one

### Irradiation experiments

#### Method of preparation films

(3 gm) of poly (vinyl chloride) dissolved in (100 ml) tetrahydrofurane (THF) to prepare 3% PVC solution for usage to make the significant thickness of polymer films (40,70, and 100  $\mu\text{m}$ ), as calculated by digital micrometer, type (2610, Germany) and as

displayed in Figure 1. These films were designed by casting and vaporization techniques in glass trays at 25  $^{\circ}\text{C}$  for 24 hr. to remove the possible solvent residue. The concentration of additives was (0.1%, 0.3% and 0.5%). After the evaporation of solvent that's led to the formation of polymer films, the polymeric films are taken from the glass trays and pasted to cardboard sheets that have a dimensional 2 $\times$ 2 cm.



**FIGURE 1** Measurement of films preparation thickness

#### Accelerated testing technique

Q-plate Company produced a quicker weather-meter (QUV, Q-Panel Company, Florida, USA) to irradiation of PVC films. A plate of stainless steel with one hole on the back side and two more on the front is used in the accelerated weathering test. Each side has a 55 Watt lamp (type Fluorescent Ultraviolet Lights) from Q-Panel Company, with wavelengths ranging from (250-380 nm) with the intensity of the light wavelength at ( $1.87 \times 10^{-5}$  Ein  $\text{dm}^{-3} \cdot \text{s}^{-1}$ ).

The poly vinyl chloride films were fixed parallel to the lamps to ensure that UV radiation is incident vertically on the films samples. The distance between the polymeric film and the light source was (10 cm) [26,27].

#### Measuring of photodegradation methods

FT-IR Spectrophotometer was used to track the photodegradation of pure PVC film and PVC films with synthesis compound as additives, in the range of (4000-400)  $\text{cm}^{-1}$ , the absorption spectra of poly vinyl chloride films were recorded. The position of carbonyl absorption is given at (1724)  $\text{cm}^{-1}$ . Changes in carbonyl peaks were used to track the photodegradation steps through varying irradiation durations. The carbonyl indexes ( $I_{\text{co}}$ ) were measured by comparing the FTIR absorption peaks at (1724)  $\text{cm}^{-1}$  with reference peaks at (1427)  $\text{cm}^{-1}$ . This process is known as the band index method, and consists of the following equations (1).

$$I_s = \frac{A_s}{A_r} \quad (1)$$

where:

$A_s$  = Absorbance for band at study.

$A_r$  = Absorbance for reference band.

$I_s$  = Group index at study.

The absorbance (A) at wavenumber specific for PVC carbonyl is measured by the relation explained in equation (2).

$$A = 2 - \log \%T \quad (2)$$

$\%T$  = the transmittance percentage.

The baseline technique is used to calculate the actual or real absorbance that differences between the highest peak's absorbance and the baseline (A Top beak-base line) [29].

*Viscosity- average molecular weight determination ( $\bar{M}_v$ )*

The intrinsic viscosity  $\eta$  as role of average molecular weight, it is calculated through equation (3) [30].

$$[\eta] = K (\bar{M}_v)^\alpha \quad (3)$$

Where K and  $\alpha$  are constant quantities for PVC at temperature and solvent system.

Intrinsic viscosity was examined by using the U-tube Ostwald viscometer which allows reading the times of flowing of polymer solution (t) and the THF solvent ( $t_0$ ). The viscosities below were calculated by the following equations.

$$\text{Relative viscosity } \eta_{rel} = t/t_0 \quad (4)$$

$$\text{Specific viscosity } \eta_{sp} = (t/t_0) - 1 \quad (5)$$

$$\text{Intrinsic viscosity } \eta = [(\sqrt{2}/c)(\eta_{sp} - \eta_{re})]^{1/2} \quad (6)$$

Using the following relation, the quantum yields of main chain scission ( $\phi_{cs}$ ) were derived from viscosity measurements [30].

$$(\phi_{cs}) = (CA/\bar{M}_{vo}) [([\eta_0]/[\eta]^{1/\alpha-1})/I_0 t] \quad (7)$$

In which, C=concentrations (3gm/100 mL), A = Avogadro's numbers, ( $\bar{M}_{vo}$ ) = the initial viscosity averages molecular weights,  $[\eta_0]$  = Intrinsic viscosity of PVC polymer before irradiation,  $[\eta]$  = Intrinsic viscosity of polymer after irradiation,  $I_0$ =Incident intensity ( $1.87 \cdot 10^{-5}$  Ein.  $\text{dm}^{-3} \cdot \text{s}^{-1}$ ) and t = Irradiation time in second.

The number of average chain scission (S) was determined by using the equation (8) [5].

$\bar{M}_{vo}$  And  $\bar{M}_{vt}$  are the viscosity average molecular weights before and after irradiation time, respectively.

$$S = \frac{\bar{M}_{vo}}{\bar{M}_{vt}} - 1 \quad (8)$$

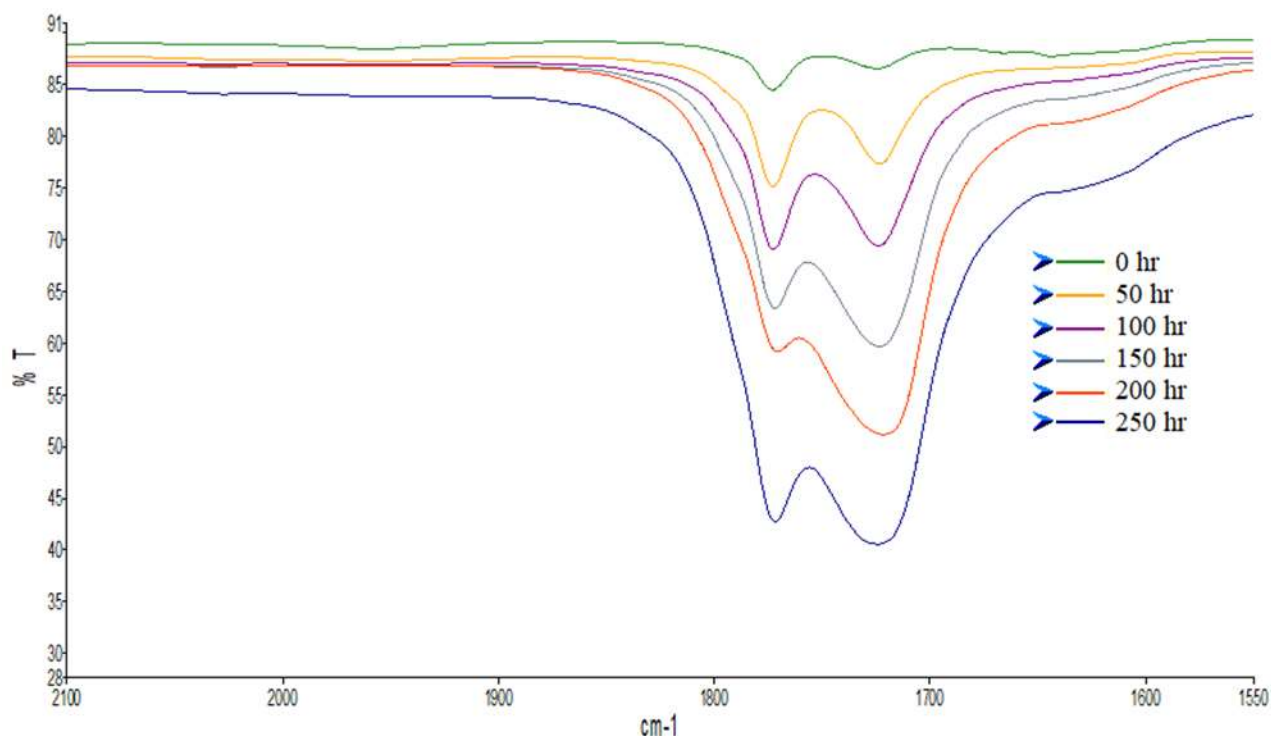
According to the following equation (9), the degree of deterioration ( $\alpha$ ) changes over time.

$$\alpha = \frac{m}{\bar{M}_{vo}} S \quad (9)$$

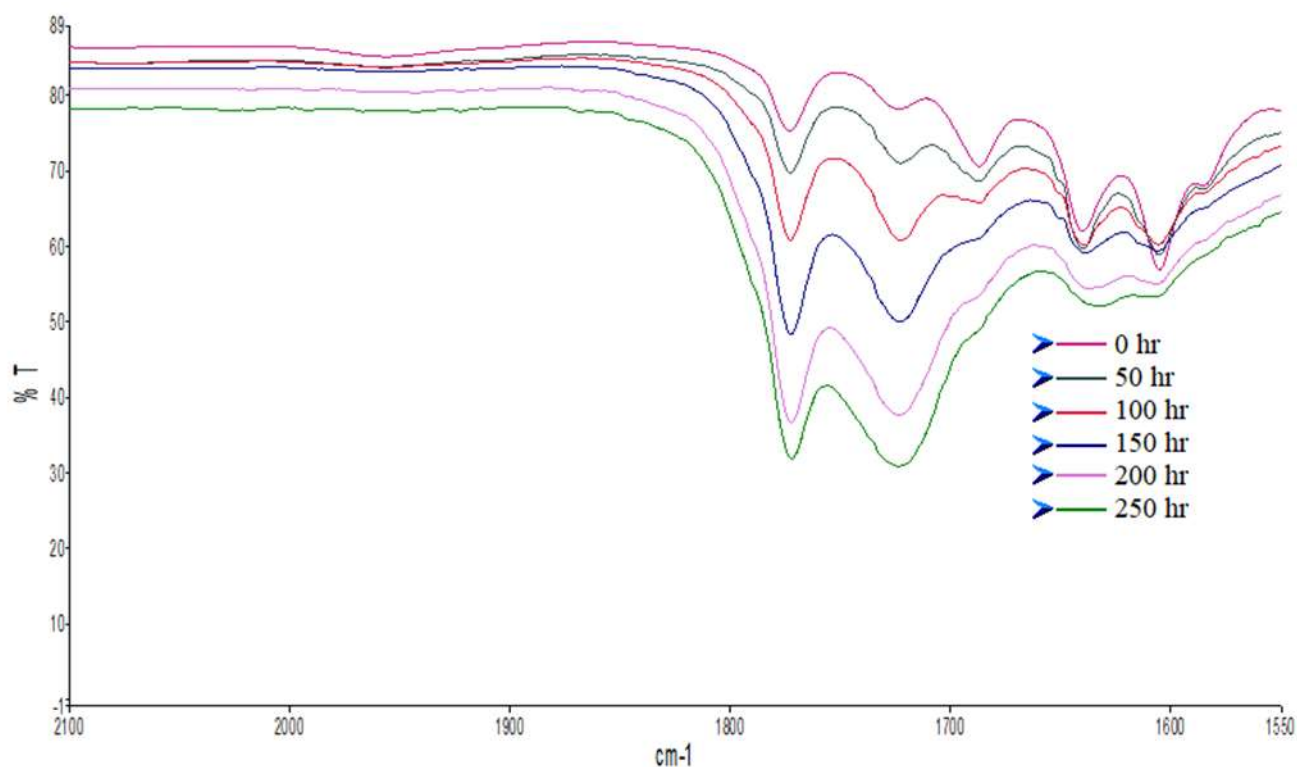
Where m and  $\bar{M}_{vo}$  are the molecular weights of the monomers and polymer before irradiation time, respectively [17].

## Results and discussion

Compounds [I1-I4] were used as the additives for photostabilization of PVC films. The irradiation of PVC films with UV light at wavelength 313 nm, led to appearance of bands at  $1772 \text{ cm}^{-1}$  and  $1724 \text{ cm}^{-1}$  was attributed to the formation of carbonyl groups related to carbonyl compounds such as (Chloroketone and aliphatic ketone) [31]. Using an FTIR spectrophotometer, the carbonyl index was monitored as a function of irradiation times. When the PVC films were irradiated, the FTIR spectrum changed dramatically. Figures 2 and 3 display the difference in FT-IR spectra of PVC film without additives and whit 0.5% additives, respectively.



**FIGURE 2** FT-IR spectrum of PVC (control) under UV ray degraded for several exposure time



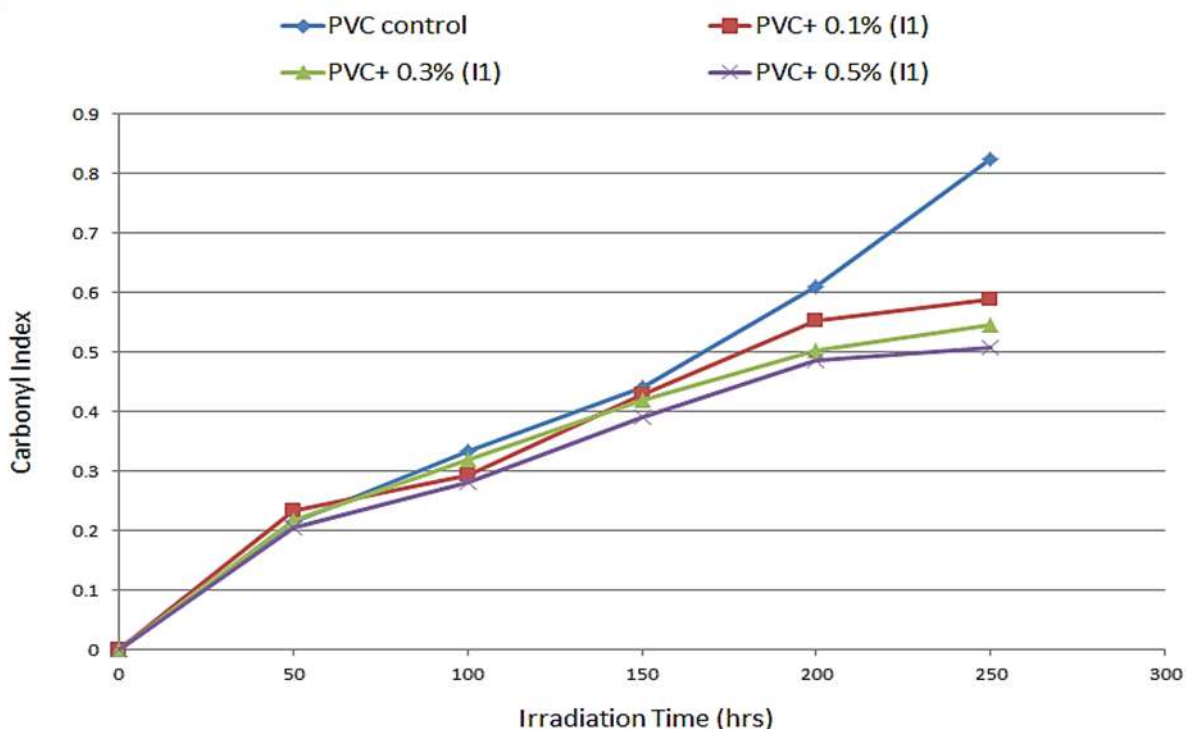
**FIGURE 3** FT-IR Spectra of PVC with sample stabilizer 0.5% (I4) under UV degraded for several exposure time

Because the intensity of this band is proportional directly to the degradation bands, changes in the intensity of the

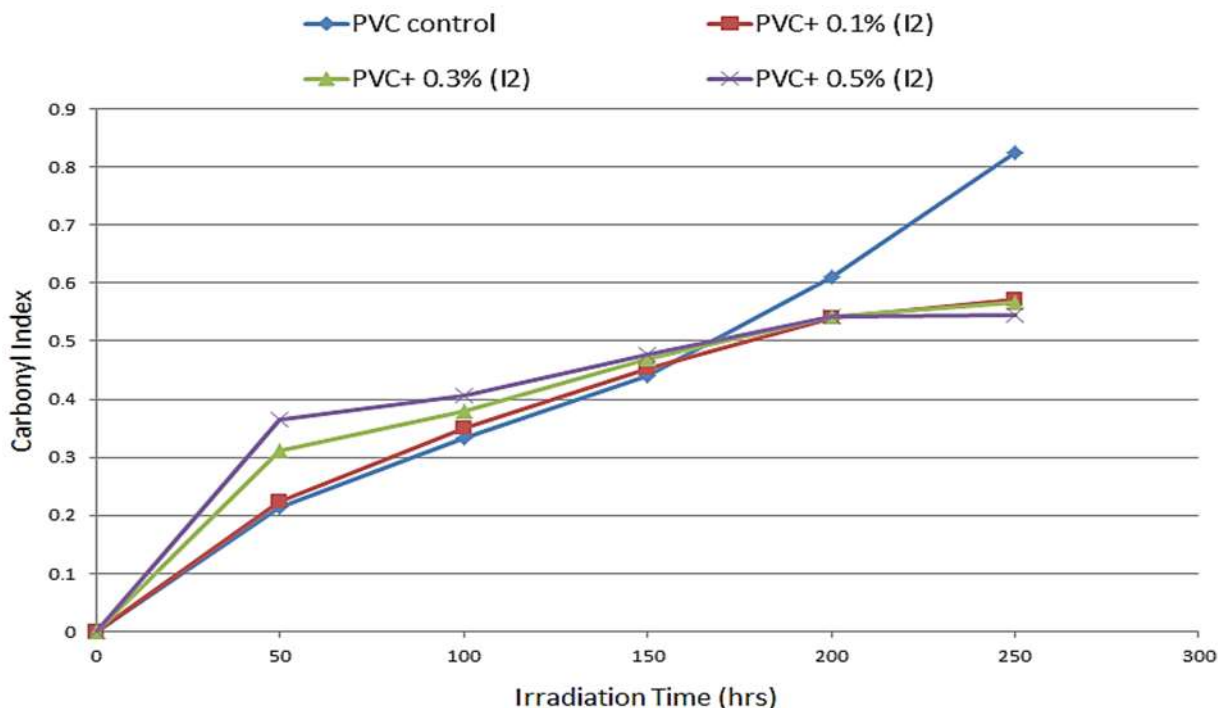
carbonyl group extended absorption bands were used to track the range of polymer breakdown through irradiation [32].

The results from Figures 4-7 illustrate that when the weight ratio of additives increases, the carbonyl index ( $I_{CO}$ ) decreases and demonstrates that additive concentrations of

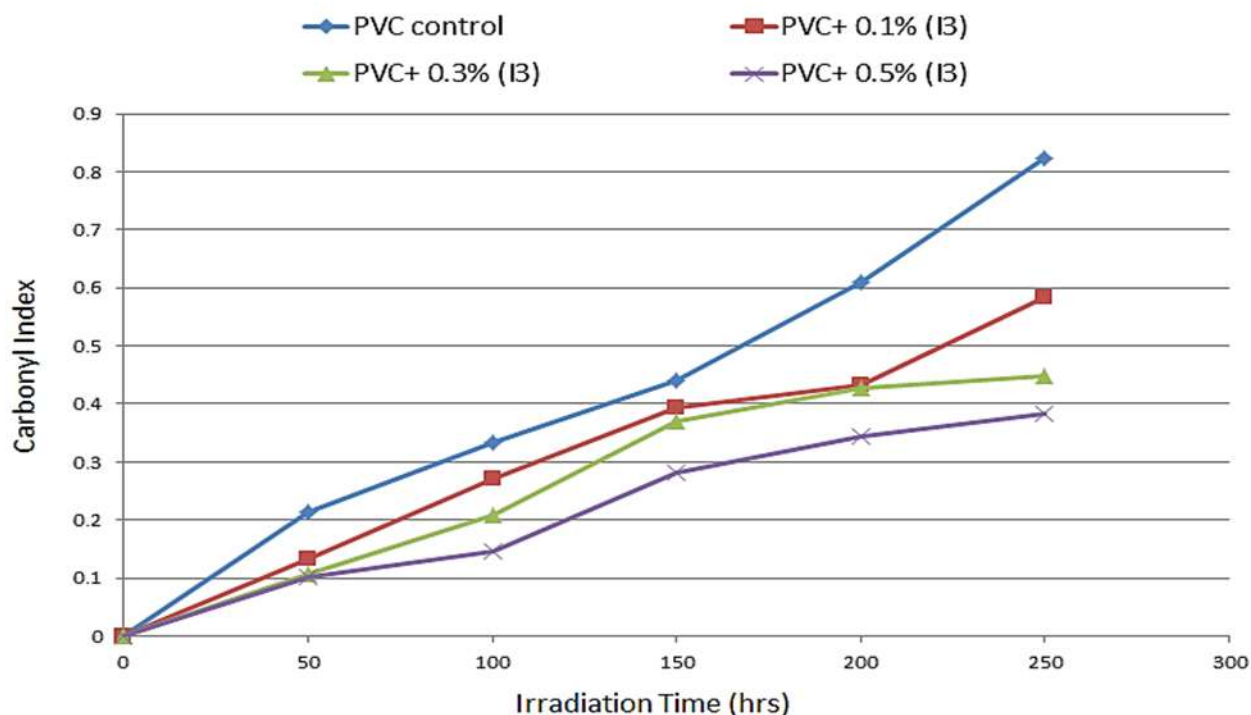
0.5% by weight gave the best results. The following figures are displayed with the 40 $\mu$ m films thickness.



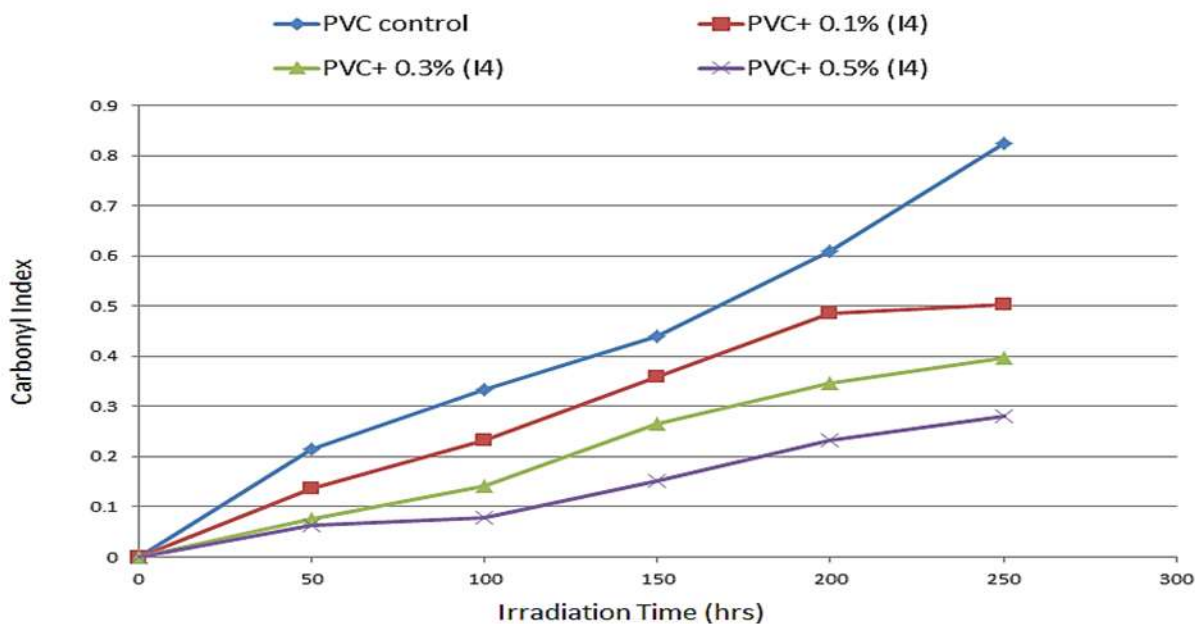
**FIGURE 4** The relationship between the carbonyl index and irradiation time of PVC films with several concentrations (0.1-0.5% for I1 additive)



**FIGURE 5** The relationship between the carbonyl index and irradiation time of PVC films with several concentrations (0.1-0.5% for I2 additive)



**FIGURE 6** The relationship between the carbonyl index and irradiation time of PVC films with several concentrations (0.1-0.5% for I3 additive)

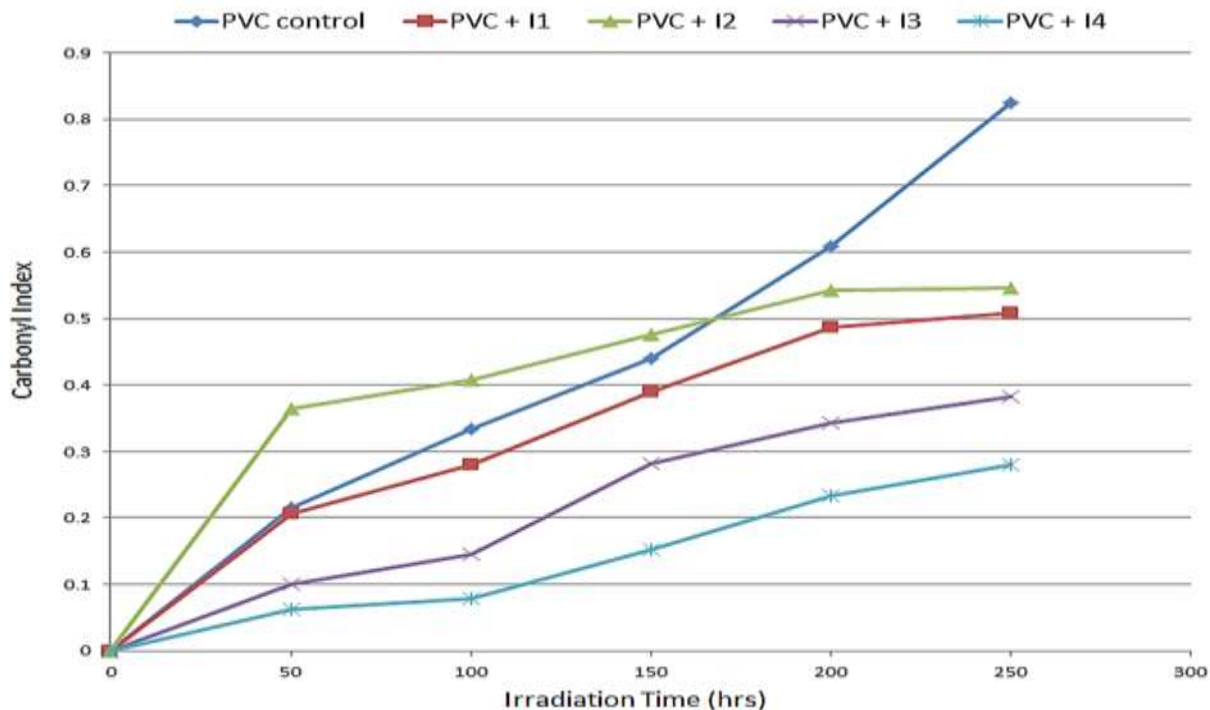


**FIGURE 7** The relationship between the carbonyl index and irradiation time of PVC films with several concentrations (0.1-0.5% for I4 additive)

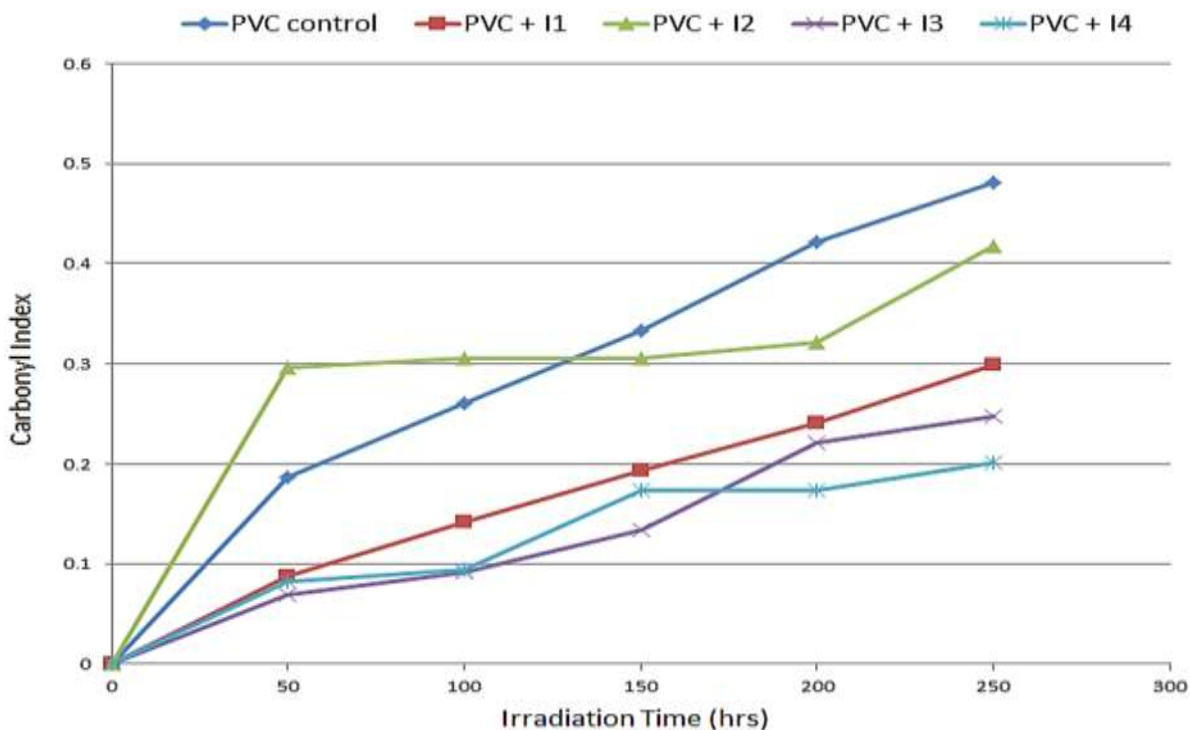
The photodegradation rate of PVC films was studied in different thickness of (40, 70, and 100  $\mu\text{m}$ ) without and with fixed concentration of (0.5%) of additive. Figures 8-10 displays the relationships between the carbonyl index and irradiation time, it

explains that the rate of photo-oxidation ( $I_{co}$ ) at fixed irradiation time decreases when the film thickness of the additive utilized increases, as does the rate of photo-oxidation (carbonyl index) for the PVC control. Figure 11 depicts the correlations between the

carbonyl index ( $I_{CO}$ ) and thickness film, photodegradation decreases, indicating that as the film thickness increases,

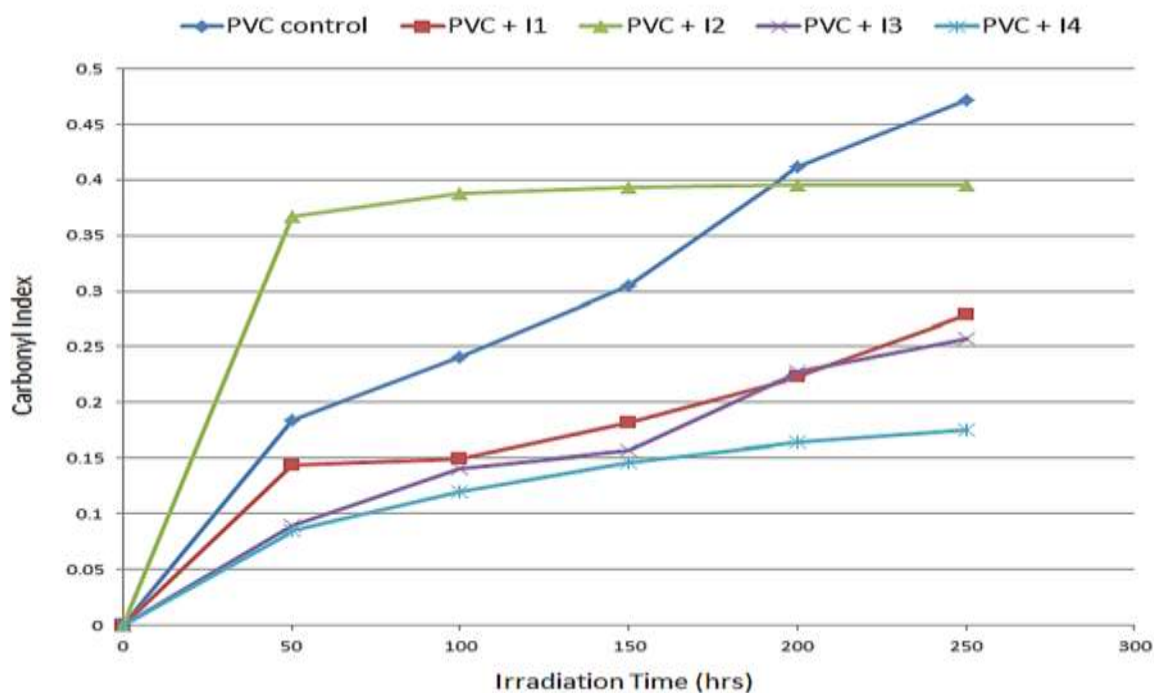


**FIGURE 8** The relationship between carbonyl index and irradiation time of PVC films with (40 μm) and that containing 0.5% wt. of additives

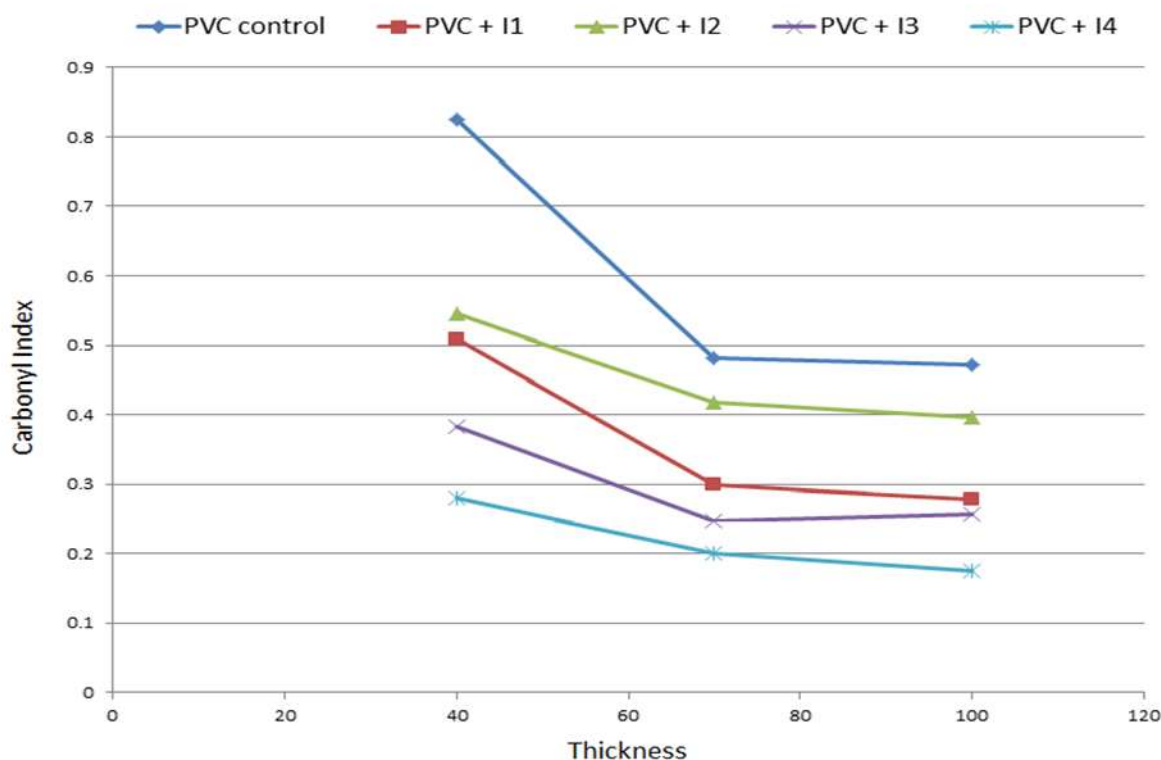


**FIGURE 9** The relationship between carbonyl index and irradiation time of PVC films with (70 μm) and that containing 0.5% wt. of additives





**FIGURE 10** The relationship between carbonyl index and irradiation time of PVC films with (100 μm) and that containing 0.5% wt. of additives



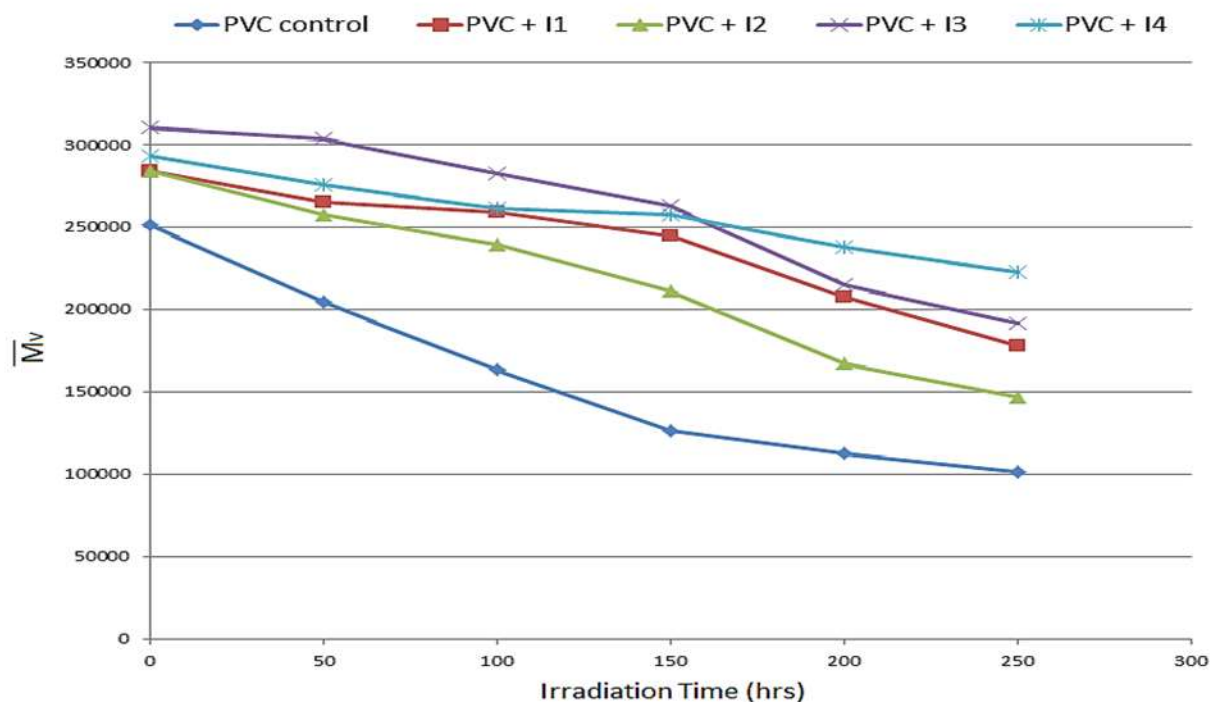
**FIGURE 11** The relationship between carbonyl index and films thickness (μm) for PVC film at 250 (hrs.) irradiation time containing 0.5% wt. of additives

The average molecular weight ( $\bar{M}_v$ ) of PVC degradation was calculated by viscosity

measurement using equation (3) [ $K=1.5 \times 10^{-4}$  g/ml,  $\alpha'=0.77$ ] and THF solvent at 30 °C.

When PVC films (with or without additive) are irradiated, the intrinsic viscosity and viscosity averages molecular weight decrease as the irradiation period increases. The

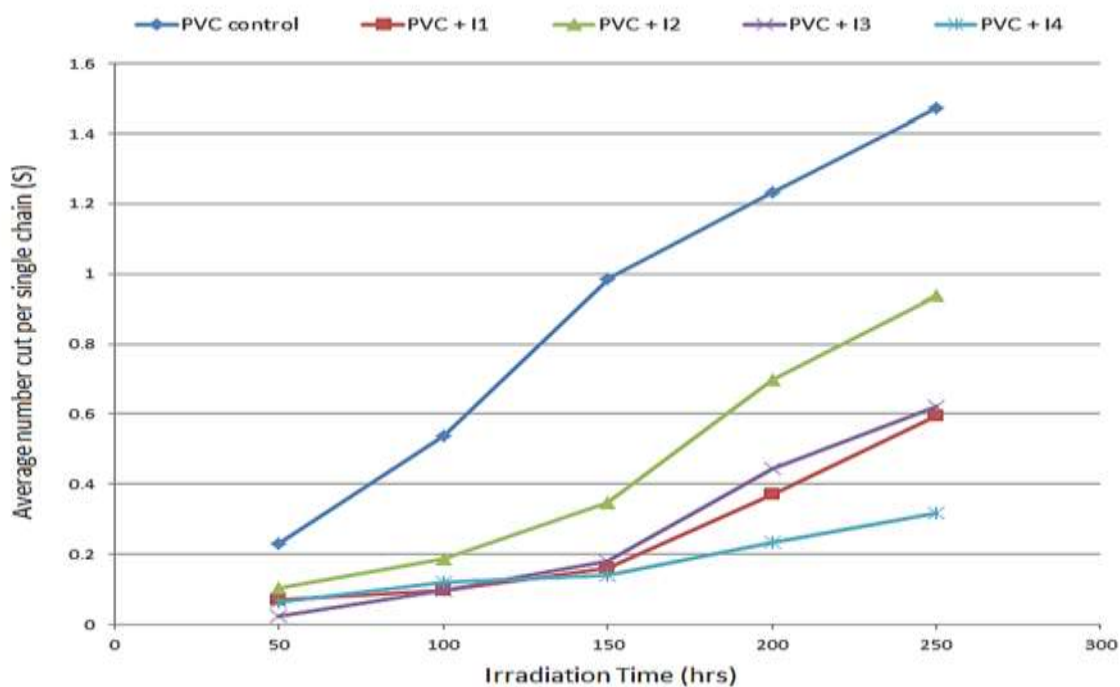
change in the ( $\bar{M}_v$ ) of PVC films with and without additions after irradiation time, with (0.5 wt%) additives is shown in Figure 12.



**FIGURE 12** Change in the viscosity average molecular weight ( $\bar{M}_v$ ) through irradiation of PVC films (40 $\mu$ m in thickness) (control) and with 0.5% of additives

The diagram in Figure 12 indicate a reduction in ( $\bar{M}_v$ ) suggesting that the reduction in ( $\bar{M}_v$ ) is due to the split of the main chain at numerous sites which are spread over the polymer chain.

By using the equation 8, the average number of chain scissions (S) was computed. Figure 13 displays the plot of (S) against time. The relationship suggests that crosslinking may result in a rise in the degree of branching.

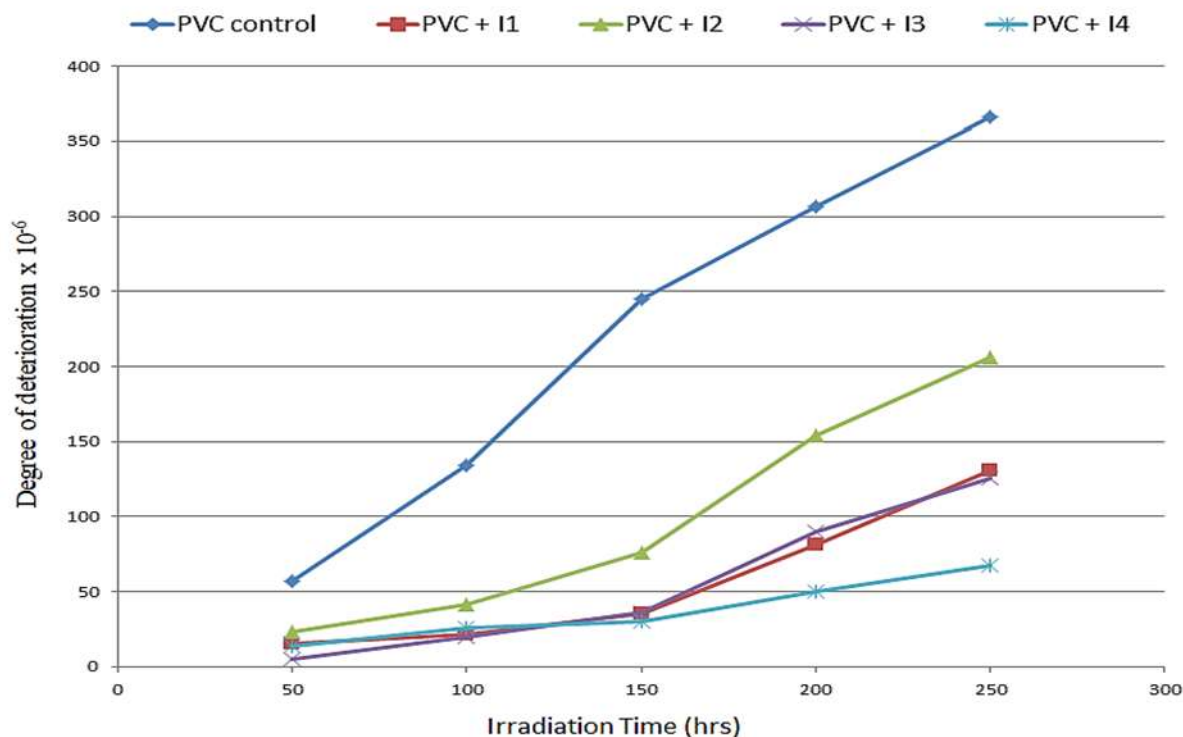


**FIGURE 13** Change in the number average chain scission (S) through irradiation of PVC films (40 $\mu$ m in thickness) (control) and with 0.5% additives

The plot illustrates that a rise in the branching degree and this results from occurrence crosslinking. It has been observed that some films after irradiation are not completely soluble in THF solvent, and this indicates on the idea of the crosslinking occurrence in these films during irradiation.

According to the equation 9, the degree of deterioration ( $\alpha$ ) changes with time. In Figure

14, the diagram of ( $\alpha$ ) as a function of irradiation periods illustrates that  $\alpha$  values of the irradiated films are high for free PVC (PVC control) and lower when the additives are present. The value of  $\alpha$  rises quickly with time in the early steps of photodegradation of PVC these signs mention a breaking of bonds randomly occurrence in PVC chain.



**FIGURE 14** Change in degree deterioration ( $\alpha$ ) through irradiation of PVC films (40 $\mu$ m in thickness) (control) and with 0.5% additives

The quantum yields of chain scission ( $\Phi_{cs}$ ) were obtained using equation (7) and summarized as indicated in Table 2 for PVC

films without additive and PVC films with (0.5% wt/wt) of additives.

**TABLE 2** Quantum yield ( $\Phi_{cs}$ ) for the chain scission for PVC films (40  $\mu$ m) thickness with and without 0.5 (wt/wt) additive after 250 hrs. irradiation time

Polymer with w% additives	Quantum yield of main chain scission ( $\Phi_{cs}$ )
PVC (Control)	$6.28 * 10^{15}$
PVC + I1	$2.71 * 10^{15}$
PVC + I2	$3.85 * 10^{15}$
PVC + I3	$2.16 * 10^{15}$
PVC + I4	$1.22 * 10^{15}$

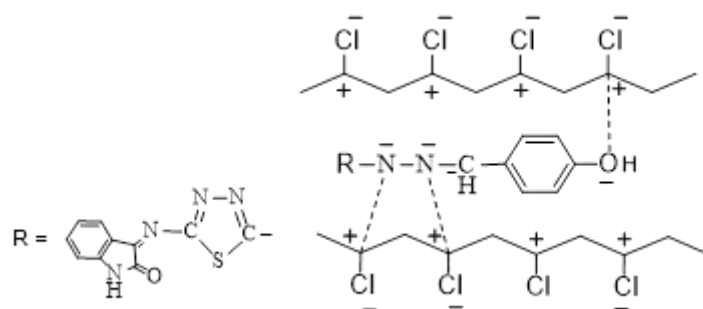
PVC films without additives have higher ( $\Phi_{cs}$ ) values than those with additives, which grow in the following trend:

$$PVC > I2 > I1 > I3 > I4$$

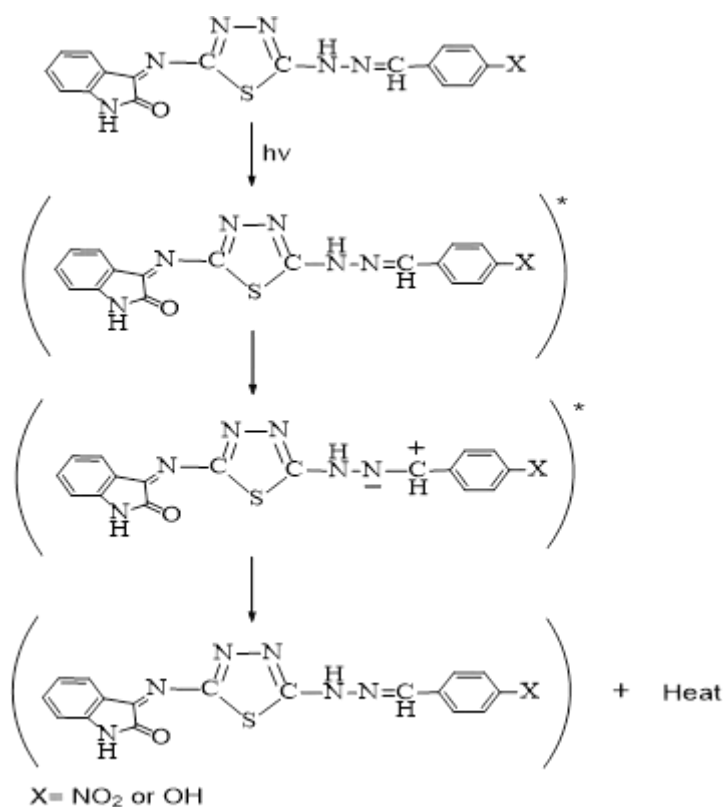
The reason for the low ( $\Phi_{cs}$ ) values in the presence of additives is that during the irradiation the energy absorbed by the PVC is from one site and the electronic excitation are

distributed over all the existing bonds, and this reduces the probability of bonds' breaking, or that the absorbed energy is disposed of or distributed through non-reactive processes.

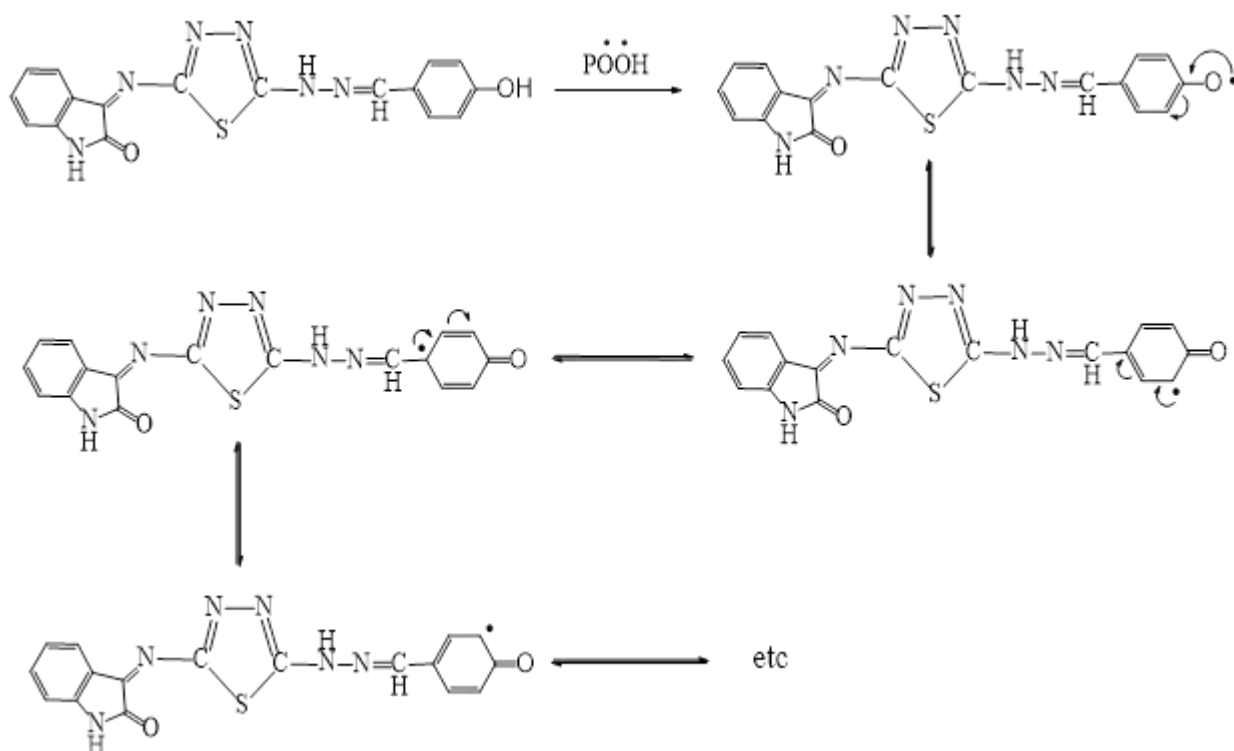
Some mechanics were proposed based on the findings of the experiment [31,32] Schemes 1-3.



**SCHEME 1** The suggested mechanism of photostabilization of PVC by Schiff base compound (14) as primary stabilizers



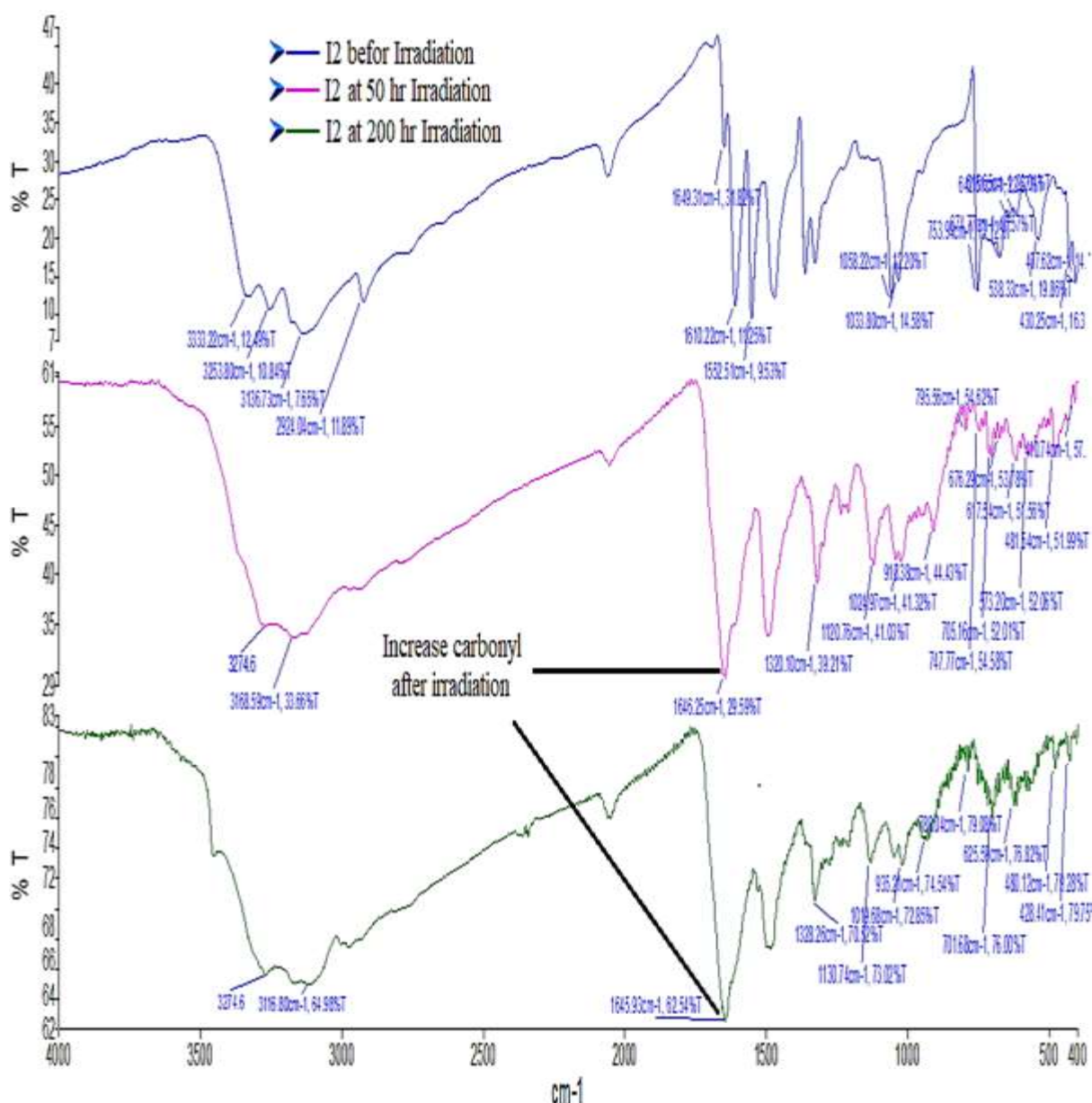
**SCHEME 2** The suggested mechanism of photostabilization of PVC Schiff bases compounds as UV absorber



**SCHEME 3** The suggested mechanism of photostabilization of PVC by Schiff bases compound (I4) as radical scavenger

From the Figures 8-10 that explain the relationship between the carbonyl index and irradiation time for PVC films (40, 70, and 100  $\mu\text{m}$  thickness) with different concentration of I2 compound additive, we demonstrate that the compound I2 initially act as photo degradation for PVC polymer. And from FT-IR tests for just I2 compound before and after irradiation Figure 15, we

notice that with the increase the irradiation time, the growth of the carbonyl group increase and the intensity of the amine group decreases, this indicate that the compound I2 initially act as a photo degradation for PVC polymer, and after irradiation the polymeric films by UV light for a long time, it is dissociated into the other compounds which acted as photo-stabilizers for PVC polymer.

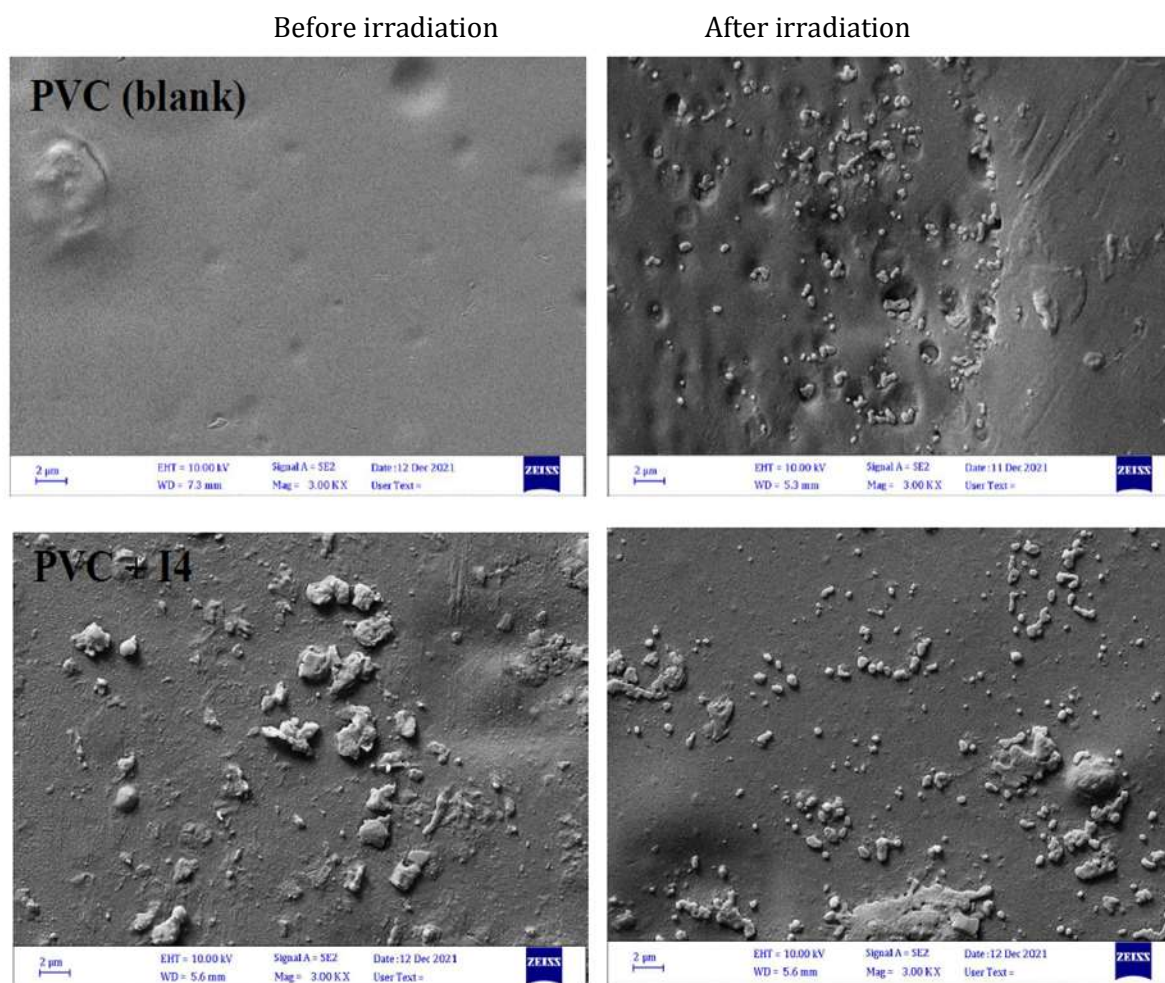


**FIGURE 15** FT-IR spectrum of pure I2 compound before and after irradiation

*Surface morphological study of poly (vinyl Chloride) films by scanning electron microscope and light microscope as a criterion for the photostabilizing efficiency*

The morphology of surface films of blank PVC (nonirradiated PVC), PVC irradiated for 250 hr., and PVC in the presence of I4 as organic additives (irradiated for 250 hr.) was studied with scanning electron microscope (SEM) (Figure 16). The surface of PVC film (blank) was smooth and free of any white spots

indicating degradation, while the surface of PVC film irradiated for 250 hour was filled with white spots indicating to HCl had evolved resulting the holes or grooves. While in the case of PVC in the presence of additive (I4) irradiated for 250 hour, the surface was almost smooth, and less white spots appeared and there is no holes, this indicated the high stabilizing efficiency of the examined organic stabilizer and the extent to which the polymer surface is protected from degradation by dehydrochlorination [33].

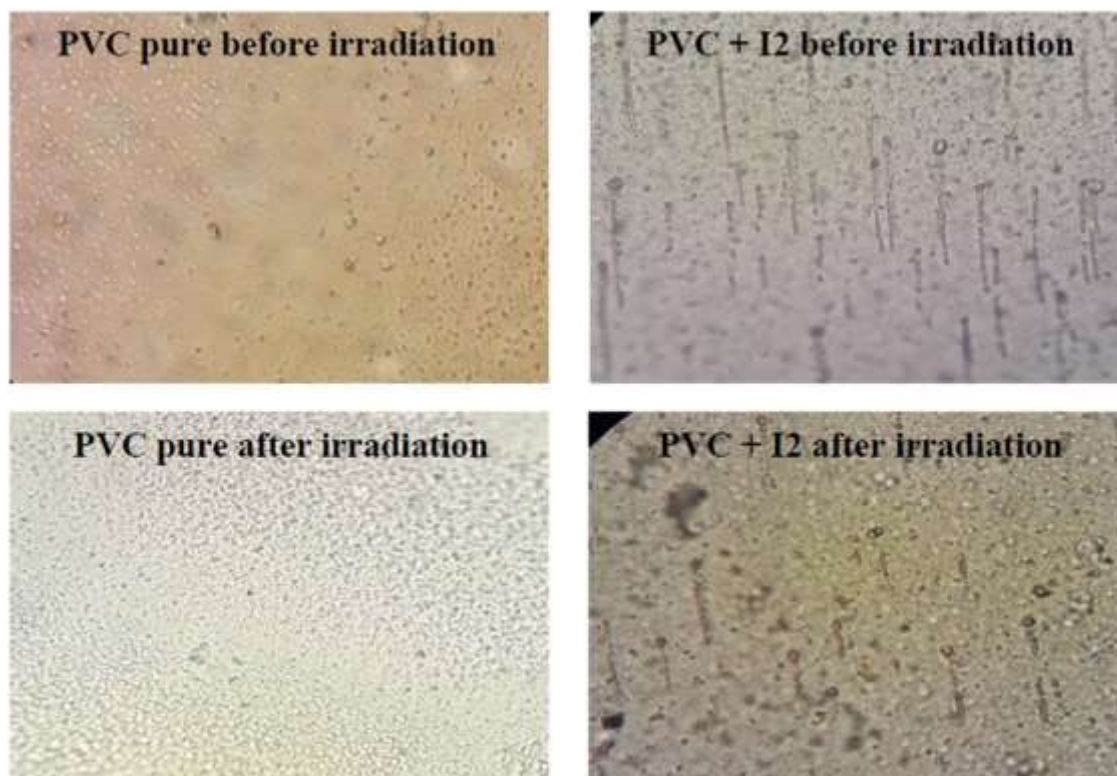


**FIGURE 16** SEM images before and after 250 hour of irradiation of PVC blank film, and PVC containing 0.5% compound (I4)

Figure 17 displays a light microscope images tests of polymer films surfaces using a high-power field (400X magnifications) before and after irradiation, before irradiation we note that the film (PVC + 0.5% I2) comparing it with pure PVC film (PVC control), it is the beginning degradation and the change in the crystalline regularity of the surface, becomes rough, and the formation of spots, holes and bubbles due to the breakage of the polymeric chains, and by comparing it

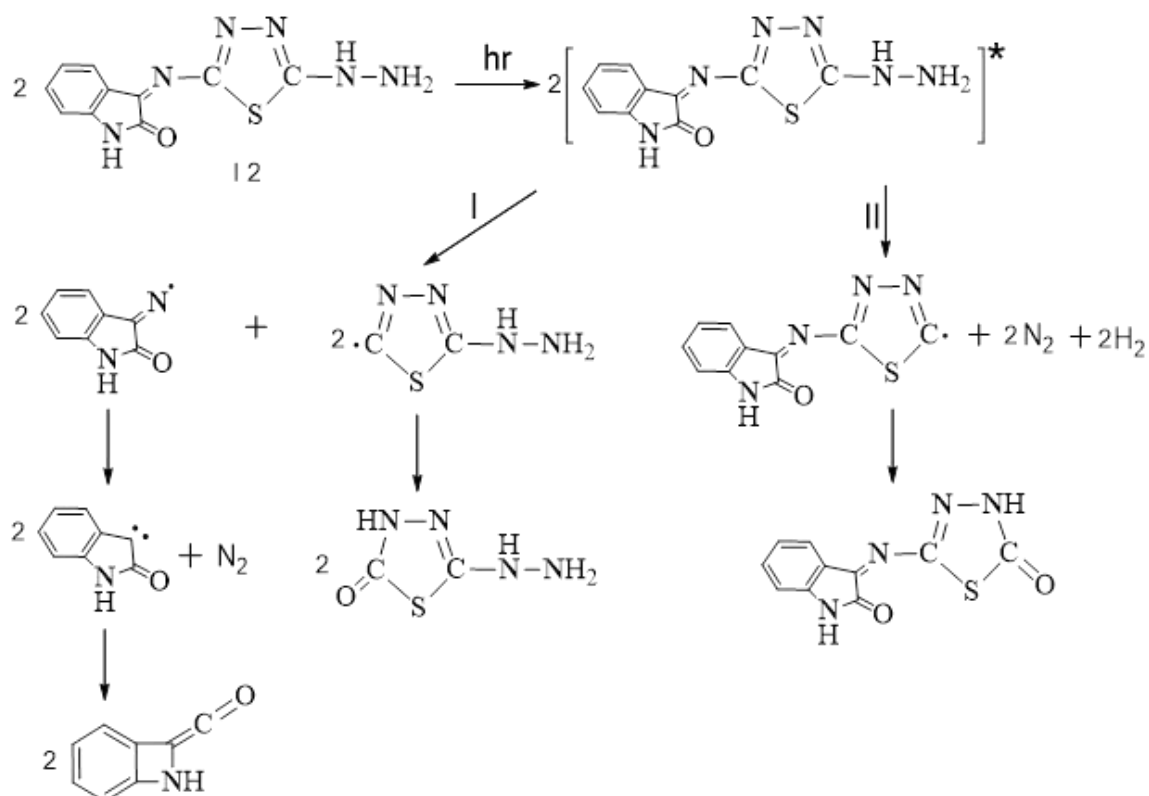
again with its image after irradiation, we notice that there is no significant change and the presence the same spots, holes, and bubbles. This is another indication for I2 compound work as a photodegradation for PVC before irradiation, and after subjected it to UV light irradiation for a period time, it is dissociated into other compounds (fragments) which acted as photostabilizers for PVC polymer [34].





**FIGURE 17** Light microscope images for PVC pure film surface and (PVC + 0.5%I<sub>2</sub>) (40 μm thickness) before and after 250 hr. irradiation. 400X magnifications

The suggested mechanism of dissociated of I<sub>2</sub> compound by UV light explained in Scheme 4.



**SCHEME 4** The suggested mechanism of dissociated of pure I<sub>2</sub> compound by UV light

## Conclusion

The photostabilization of (PVC) films containing the additives (0.5 percent by weight) was following the trend  $I_4 > I_3 > I_1 > I_2 > \text{PVC}$  as the additives' efficiency increased. In comparison to PVC control, the carbonyl index growth for the additive (I4) was lowered. These additives stabilize poly vinyl chloride films through UV absorption, peroxide decomposer, and radical scavenger mechanisms. According to the photostability and mechanisms discussed above, the additives were to be the most effective in photostabilization processes. The compound I2 initially act as the photo degradation for PVC polymer, and after irradiation polymeric films by UV light for a long time, it is dissociated into the other compounds (fragments) that acted as photostabilizers for PVC polymer.

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## Orcid:

Mohammed Alwan Farhan:

<https://www.orcid.org/0000-0003-0795-2058>

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