

## Photostabilization of Poly (Vinyl Chloride) by Some Schiff Base Derived from 2-amino pyridine

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### Abstract

In this paper, Schiff bases (1-6) were prepared and synthesized by condensing aromatic aldehydes (benzaldehyde, salicylaldehyde, *p*-methoxybenzaldehyde, *p*-hydroxybenzaldehyde, furfural and isatin) with 2-amino pyridine. Spectrophotometric (FTIR) and (UV.VIS) and elemental analysis (CHN) techniques have been employed for the characterization of the additives. All the additives were used as photostabilizer for poly (vinylchloride). The rate of photodegradation and photostabilization of PVC films (40 $\mu$ m) were monitored with irradiation time by measuring the carbonyl (Ico) and polyene (Ipo) indices values (the concentration of additives kept constant at 0.3% wt/wt). The light of wave length 313nm is used for irradiation of polymer films at temperature around 40°C.

The effect of additives concentration range from (0.1-0.5) wt% on the rate of photodegradation and photostabilization was also monitored for PVC films. The rates increase when additives concentration is increased. The effect of film thickness is also studied and results show that increasing film thickness will decrease the rate of photo degradation in the following sequence:

[2] > [5] > [4] > [6] > [3] > [1] > PVC

According to the experimental results several mechanisms were suggested depending on the structure of the additive.

Keywords: 2-amino pyridine , Poly vinyl chloride (PVC), Photostabilization, Schiff Base.

### Introduction

Thermoplastic form poly(vinyl chloride) (PVC) have a considerable commercial importance in industry and in everyday life. They are successfully replacing some natural raw materials due to their particular properties which offer a combination of easy modification in thermoplastic processing, rigidity, compatibility with a large number of other products, resistance to corrosion and low cost technology [1].

Plastics are very susceptible towards solar ultraviolet radiation, so when it is exposed to long term outdoor environment it would undergo more or less rapid degradation resulting in discoloration, embitterment, cracking of surface, stiffing and ultimately lose or decrease in their useful mechanical engineering properties [2]. The extent of outdoor service life of synthetic polymer (plastics) or naturally occurring polymers are limited by their structures., The type and amount of additives and the magnitude of UVB 313[UVB is made up of wavelength from approximately [280-320nm] rays absorbed, while temperature and humidity may further reduce service life [3]. Plastics can

be protected against thermal and photodegradation by using efficient stabilizers designed to prohibit or slow down the degradation process in order to prolong the use life of the plastic [4-9].

2-Aminopyridine compounds are well known class of compounds for a long time, and still are interestingly considerable due to their application in various fields.

The primary use of 2-aminopyridine is an intermediate in the manufacture of pharmaceuticals: anti-histamines and piroxican. Lornoxican and Tenoxican are considered as new non-steroidal, anti-inflammatory drugs of oxycam class inhibiting cyclooxygenase the key enzyme of prostaglandin biosynthesis at site of inflammation [10-13]. The photostabilizing effect of mono Schiff base in films of PVC had been studied [14-16]. To the best of our knowledge there is no previous attempt to in the literature investigate the photostabilization of PVC films by Schiff bases compounds containing pyridine and azomethine groups.

## Experimental

### Chemicals:

All chemicals used in this work were reagent grade (BDH/Aldrich) namely salicylaldehyde, benzaldehyde, *p*-methoxybenzaldehyde, *p*-hydroxybenzaldehyde, 2-furan carboxaldehyde and 2-amino pyridine which used without further purification.

### I) Synthesis of Schiff Bases (1-6) :

General procedures for the synthesis of Schiff bases of amino pyridines had been reported earlier [17-19]. The Schiff bases were prepared by adding 30ml aromatic aldehyde ethanolic solution 0.01mole, to ethanolic solution 30% of aromatic amine(0.01mole).The mixture was stirred for ~6hrs.at 100°C. The resulted solution was evaporated under vacuum to remove the solvent. The product then collected by filtration, washed several times with methanol and recrystallized from DMF and then dried under vacuum. The physical properties of Schiff bases (1-6) are listed in Table (1).

### Experimental Techniques :

#### 1. Films Preparation :

A poly (vinyl chloride) solution (5gm/100ml) in tetrahydrofuran (THF) was used to prepare different thickness of polymer films (measured by digital micrometer type 2610A Gerny) with (0.1-0.5)wt/wt% of the prepared compounds. The films were prepared by evaporation technique at room temperature for 24hrs to remove the possible residual (THF) solvent. Film samples were further dried at room temperature for 3hrs under reduced pressure. The films were fixed on stand specially used for irradiation which is aluminum plate (0.6mm) in thickness supplied (Q-panel) company.

#### 2.Irradiation Technique :

Accelerated weather meter Q.U.V. tester (Philips Germany), was used for irradiation of polymers films. The accelerated weathering tester contains stainless steel plate which has two holes in the front side and another one behind each side contains lamps type (Fluorescent Ultraviolet Lights) 40 watt each, the lamps are of the type (UV-B313) giving spectrum range between (290-360nm) and the maximum wavelength is (313 nm). The polymer film samples vertically fixed and

parallel to the lamps assure the vertical incidence of UV radiation on the samples. The places of the irradiated samples are changed places from time to time to be sure that intensity of light incident on all sample is equal.

### 3- Photo Degradation Measurements :

The photodegradation of polymer film samples were followed using Shimadzu spectrophotomet model 84005. The absorption spectra of the film samples were recorded in the range of (4000-400)  $\text{cm}^{-1}$ . The carbonyl and polyene groups absorption are allocated at 1720 and 1633 $\text{cm}^{-1}$  respectively [20]. The photodegradation during different irradiation times were followed by through the variation in carbonyl and polyene absorption band. Then carbonyl (Ico) and polyene (Ipo) indices were subsequently calculated by comparison of the FTIR absorption peaks at 1720 $\text{cm}^{-1}$  and 1633 $\text{cm}^{-1}$  with reference peak at 1428 $\text{cm}^{-1}$ , respectively. This method is called band index method [21], which includes:

$$I_{\text{C=O,C=C}} = A_{\text{S}}/A_{\text{R}} \dots \dots \dots (1)$$

$A_{\text{S}}$  : Absorbance of peak under study.

$A_{\text{R}}$  : Absorbance of reference peak.

$I_{\text{C=O,C=C}}$ : Index of the group under study.

The absorbance (A) at the specified wave number for carbonyl and polyene for PVC were calculated using the following relation shown in equation (2).

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

Actual absorbance, the difference between the absorbance base line and top of the absorption peak is calculated using the base line method [22].

## Results and Discussion

The characterization of Schiff bases additives was confirmed on the basis of their chemical structure using FTIR, UV-Visible and (CHN) analysis. The code name, chemical structure are reported in Table (1).The spectra show an absorption band at (1620-1650) $\text{cm}^{-1}$ , which were assigned to (C=N) stretching of the Schiff base additive formed as well, the other informative bands are listed in Table (2).

**Table (1)**  
**Physical Properties of Schiff Bases Additives.**

Comp. No.	Structure	Names	M. Formula M.Wt	yield %	m.p. °C	Color	Recrystallization Solvent	C.H.N		
								C%	H%	N%
								*Cal (Fou)	*Cal (Fou)	*Cal (Fou)
1.		N-benzylidin-2-amine	C <sub>12</sub> H <sub>10</sub> N <sub>2</sub> 182	68	110-112	Yellow	DMF	79.1 (78.9)	5.49 (5.01)	15.38 (15.12)
2.		2-[(pyridine-2-ylimino)methyl]phenol	C <sub>12</sub> H <sub>10</sub> N <sub>2</sub> O 198	60	162-163	Pale yellow	DMF	72.72 (71.51)	5.05 (4.99)	14.14 (13.73)
3.		4-[(pyridine-2-ylimino)methyl]phenol	C <sub>12</sub> H <sub>10</sub> N <sub>2</sub> O 198	50	120-122	Brown	DMF	-	-	-
4.		N-[4-dimethylamino]benzylidene pyridine-2-amine	C <sub>14</sub> H <sub>15</sub> N <sub>3</sub> 225	55	189-190	Yellow	DMF	74.66 (73.85)	6.66 (6.38)	18.66 (18.03)
5.		3-(pyridine-2-ylimino)-1,3-dihydro-2H-indol-2-one	C <sub>13</sub> H <sub>9</sub> N <sub>3</sub> O 223.23	50	220-222	Brown	DMF	69.95 (68.89)	4.03 (3.96)	18.83 (17.69)
6.		N-Furan-2-ylmethylidene pyridine-2-amine	C <sub>10</sub> H <sub>9</sub> N <sub>2</sub> O 172	50	190-192	Yellow	DMF	69.77 (68.02)	4.65 (4.53)	16.28 (15.63)

\*Cal = Calculated value

Fou = Found value element analysis (C. H. N.)

**Table (2)**  
**Characterization of FT-IR and UV Absorption bands of Schiff Bases Additives.**

Comp. No.	$\nu_{C=N}$	$\nu_{C-Har.}$	$\nu$ Others	$\lambda_{max}$ (nm)
1.	1618	3132	Out of plane $\nu$ (=C-H) 750,720	227,285
2.	1622	3120	Out of plane $\nu$ (=C-H) 750	245,293,310
3.	1630	3050	Out of plane $\nu$ (=C-H) 820	236,275, 327
4.	1593	3080	$\nu$ (C-H) al.2887-2992 Out of plane $\nu$ (C-H) 815	210,245,337,427
5.	1640	3070	$\nu$ NH 3291 $\nu$ C=O 1695	256,387,399,416
6.	1635	3150	$\nu$ C-O 1150	255,285,352

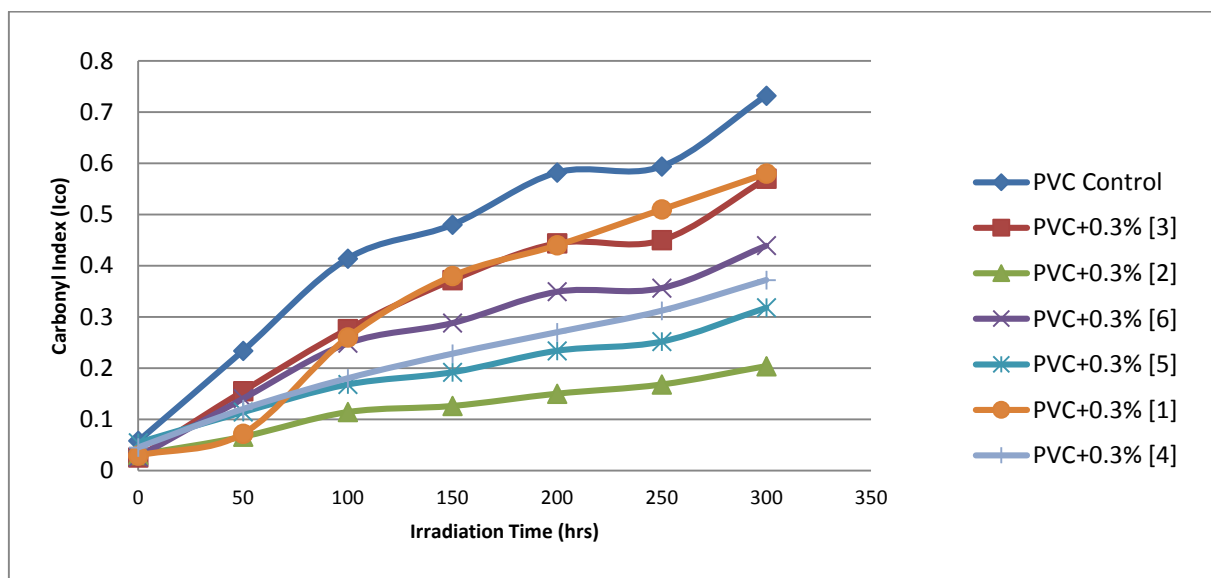
In order to study the photo chemical activity of these additives for the photostabilization of PVC films, the carbonyl and polyene indices were monitored

with irradiation time using FT-IR spectrophotometry. The irradiation of PVC films using light of wave length  $\lambda=313\text{nm}$  led to new changes in their FTIR

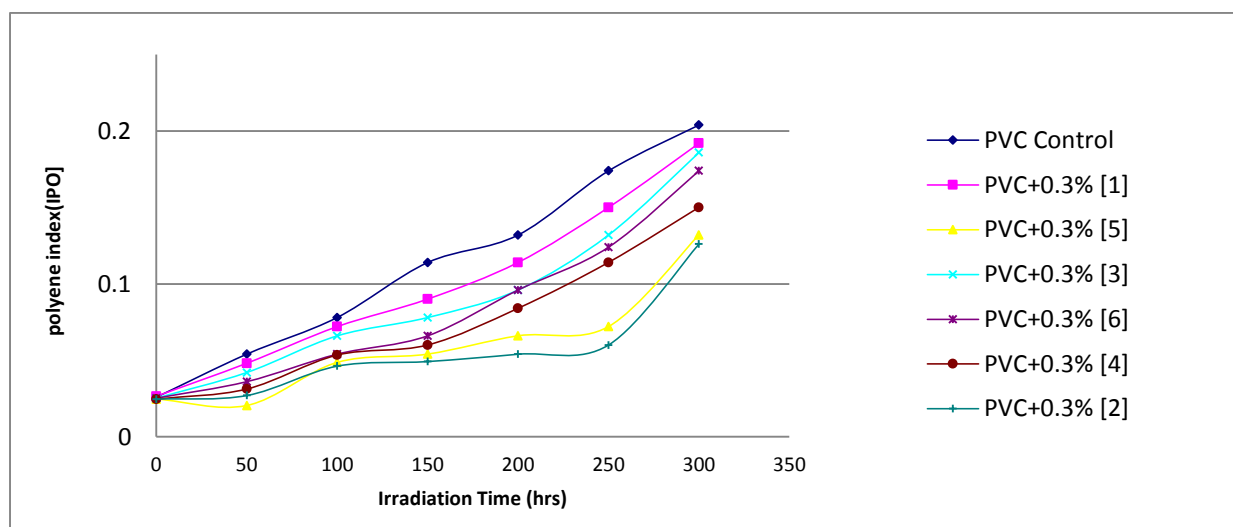
spectrum. Appearance of bands at  $1770\text{cm}^{-1}$  and  $1720\text{cm}^{-1}$ , respectively is attributed to the formation of carbonyl groups. The first is related to chloro ketone while the second one is assigned to aliphatic ketone, together with formation of a band at  $1633\text{cm}^{-1}$  related to polyene group[23].

The absorption of the carbonyl and polyene group are used to follow the extend of polymer degradation during irradiation, this

absorption was calculated as carbonyl index (Ico) and Polyene index (Ipo). Accordingly, one could expect that the growth of carbonyl index is a measure for the extent of degradation. As seen from Figs. (1 and 2).the presence of compounds (1-6) show lower growth rate for carbonyl and polyene indices with irradiation time with for PVC film without additives (control).



**Fig.(1) The variation in the carbonyl index (Ico) with irradiation time for PVC film (40µm) in thickness containing (0.3%wt) of additives (1-6).**



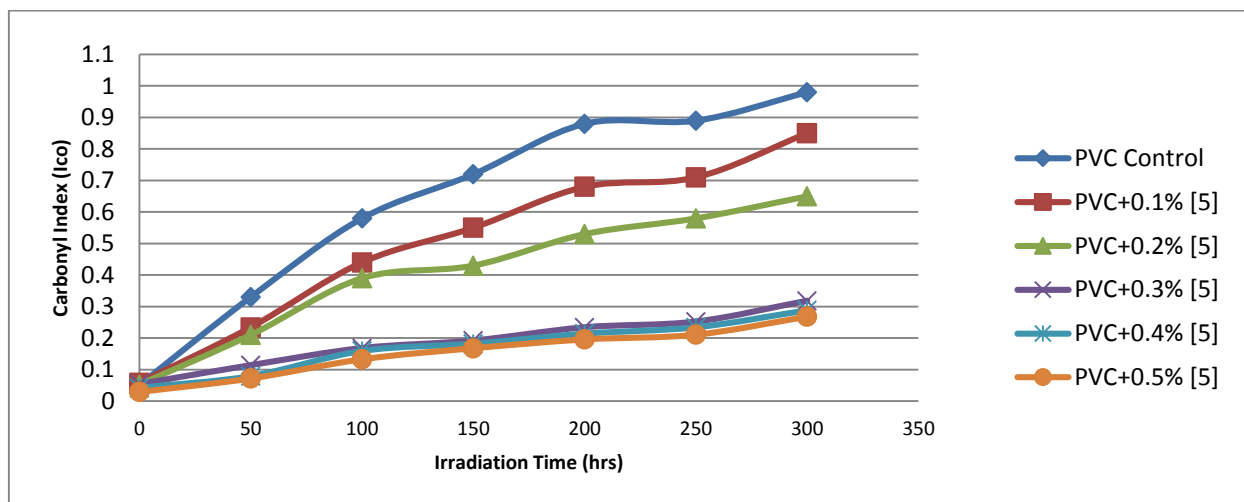
**Fig.(2) The variation in the Polyene index (Ipo) with irradiation time for PVC film (40µm) in thickness containing (0.3%wt) of additives (1-6).**

It can be observed, from the results obtained in Figs. (1 and 2) that the growth rate of carbonyl and polyene indices with irradiation time is lower in the presence of

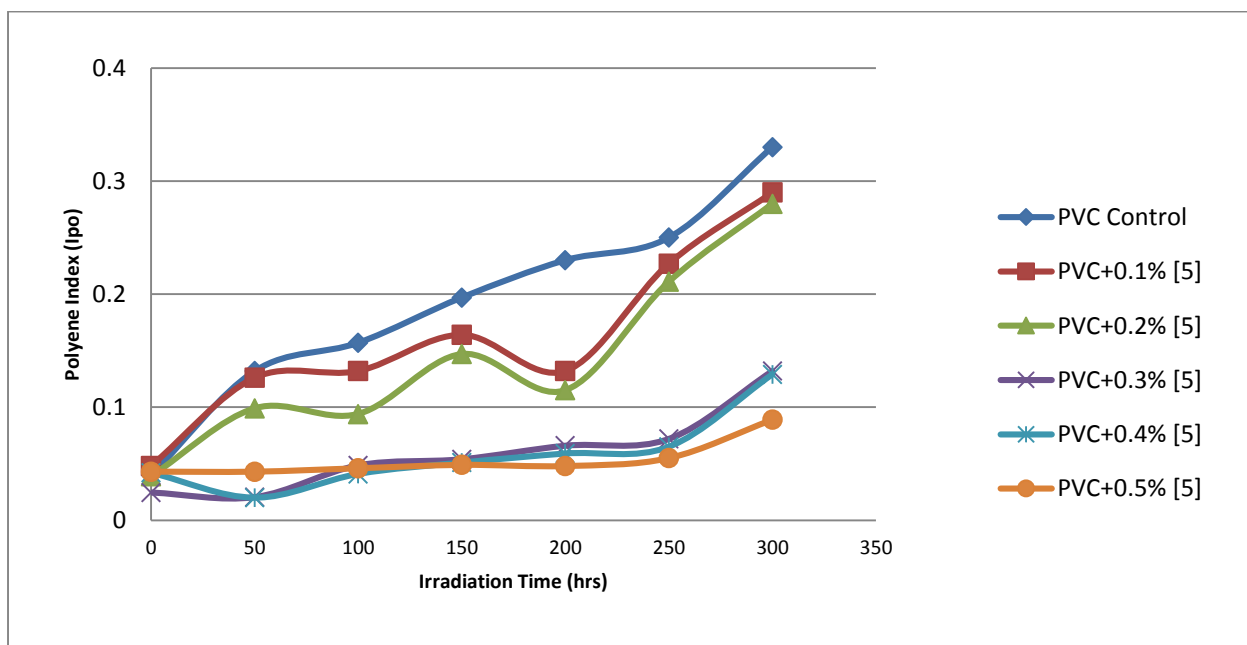
additives (1-6) with respect to the poly (vinyl chloride) without additives (control). So these additives in their FTIR spectral behavior might all be considered as photostabilizers. Also,

Fig.(1 and 2) shows that the relative photostability of additives incorporated with PVC is increased in the order :  
 $[2] > [5] > [4] > [6] > [3] > [1] > \text{PVC}$ .

The calculated values of carbonyl and polyene indices for different concentration (0.1-0.5)%wt/wt for different concentration of additive (5) are shown at Figs.(3, 4).

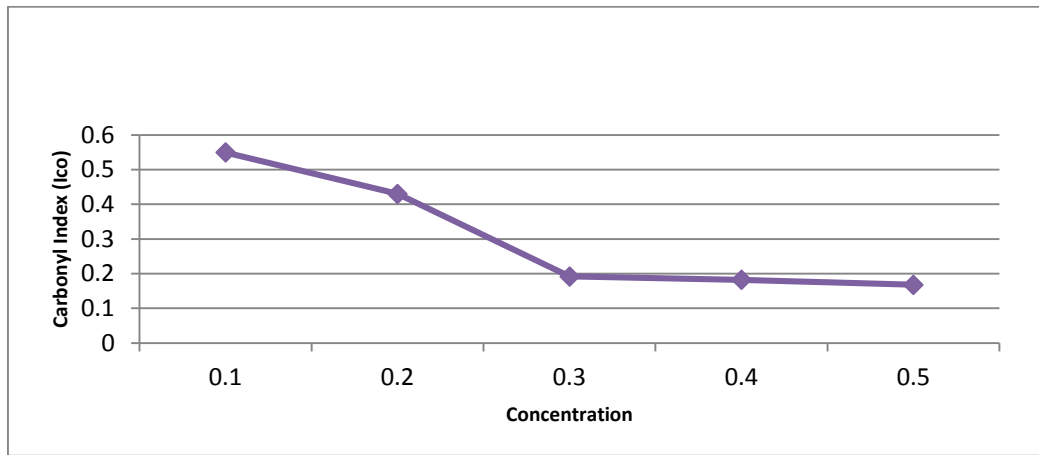


**Fig.(3) Change in Carbonyl index with irradiation time for PVC film (40 $\mu\text{m}$  in thickness containing different concentrations of compound (5).**

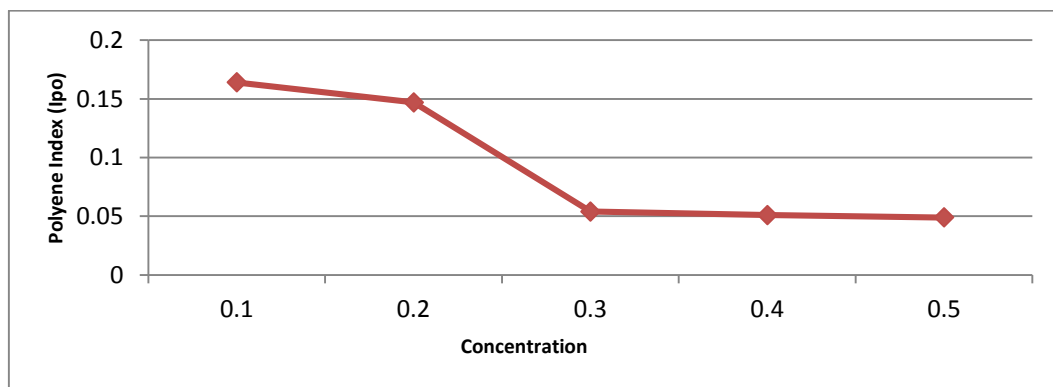


**Fig.(4) Change in Polyene index with irradiation time for PVC film (40 $\mu\text{m}$ ) in thickness containing different concentrations of compound (5).**

Figs. (5 and 6) show the relationship between the additives concentration and carbonyl (Ico) or polyene (Ipo) indices for PVC film at fixed irradiation time (150 hrs) and fixed thickness (50  $\mu\text{m}$ ).



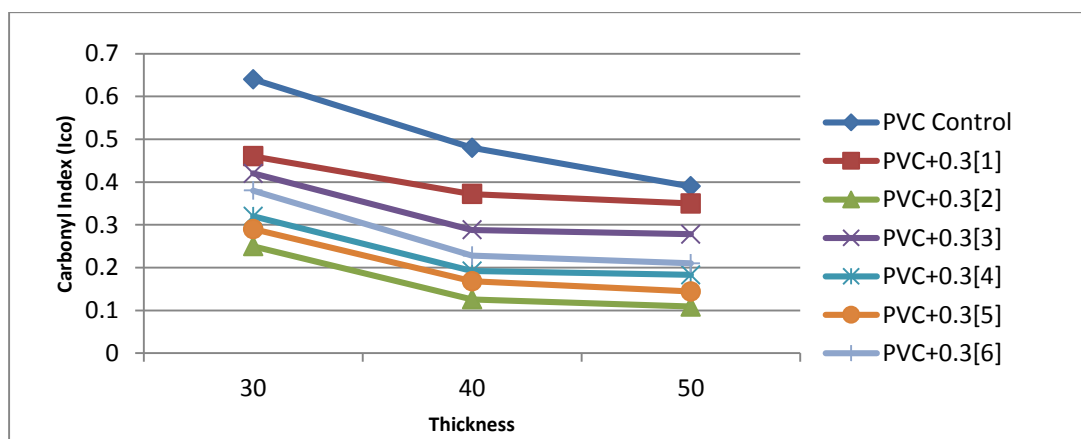
**Fig.(5) Changes in carbonyl index (Ico) with irradiation time (150hrs) for PVC films (40 $\mu$ m) thickness containing different concentrations of additive (5).**



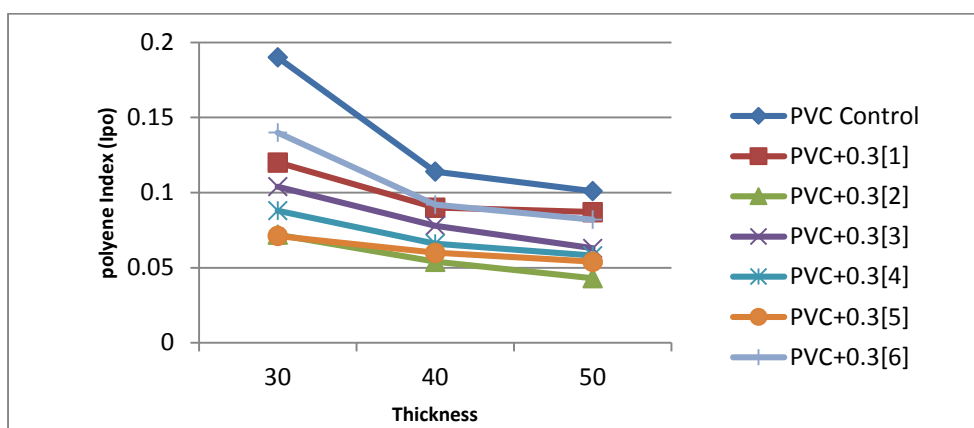
**Fig.(6) Changes in polyene index (Ipo) with irradiation time (150hrs) for PVC films (40 $\mu$ m thickness containing different concentrations of additive (5).**

Results of Figs. (3-6) show that the carbonyl and polyene indices decrease with increasing concentration of additives in films. The photooxidation of (1000 $\mu$ m) and ( $\sim$ 80 $\mu$ m) thicknesses of has already been reported by Anton et al [24] together with other studies [15,25] for the influence of films thickness in the presence of additives.

In order to investigate the effect of the films thickness on the PVC films with and without additives, FTIR spectroscopy was used to verify the occurrence of surface oxidation of different thicknesses using (30, 40 and 50  $\mu$ m) of PVC film stabilized with additives as their profile of carbonyl and polyene concentration as displayed in Figs. (7 and 8).



**Fig.(7) The relationship between the carbonyl indices with different thicknesses at (150hrs) irradiation time PVC films containing (0.3%) of additives.**



**Fig.(8) The relationship between the polyene indices with different thicknesses at (150hrs) irradiation time PVC films containing (0.3%) of additives.**

Figs. (7 and 8) show the relationship between the carbonyl index and polyene index with film thickness, which indicated the decrease of photodegradation with increasing of thickness of the film.

#### **Suggested mechanisms of photostabilization of PVC by additives:**

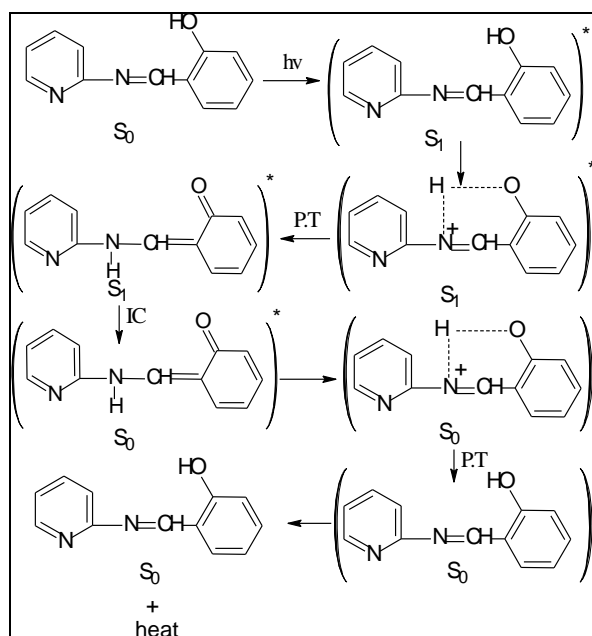
Through the overall results obtained the efficiency of Schiff base additive as photostabilizers for PVC films can be arranged according to the change in the carbonyl and polyene concentration as a reference for the comparison as shown in Fig.(1-8).

[2] > [5] > [4] > [6] > [3] > [1] > PVC

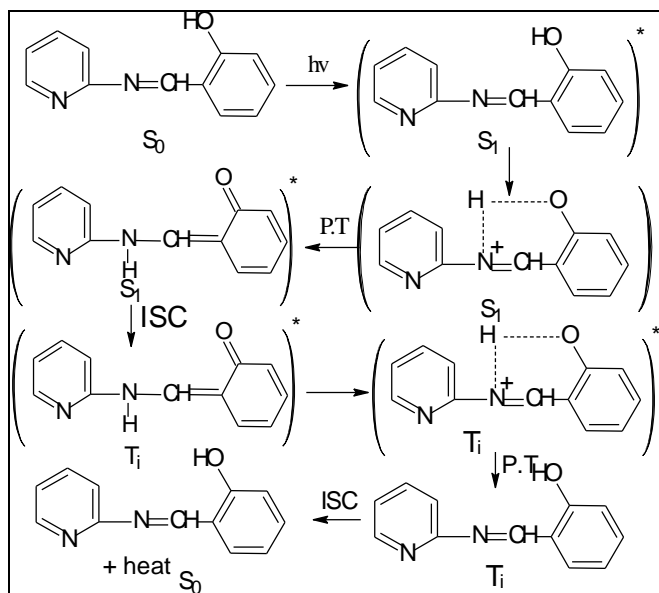
Schiff base stabilizes PVC via various mechanism such as UV absorber, screener by radical scavenger. Therefore the action of Schiff bases as photostabilizer in PVC might

be attributed to UV absorption of these additive and dissipate the UV energy to harmless heat energy.

This is also suggested for benzophenone [26-27], benzotriazole [28-29] and aromatic Schiff base photostabilizer [15]. These stabilizer stability and provide very good long time stability and are usually offered to these mechanism. The most probable mechanism involved in a photostabilization is the change in energy of absorbed photon to the intermolecular proton transfer; this action may occur by two proposed cycles Scheme (1) and (2). The first passes by intersystem crossing (ISC) process to the triplet state, while the second is referred to internal conversion (IC) process to the ground state.



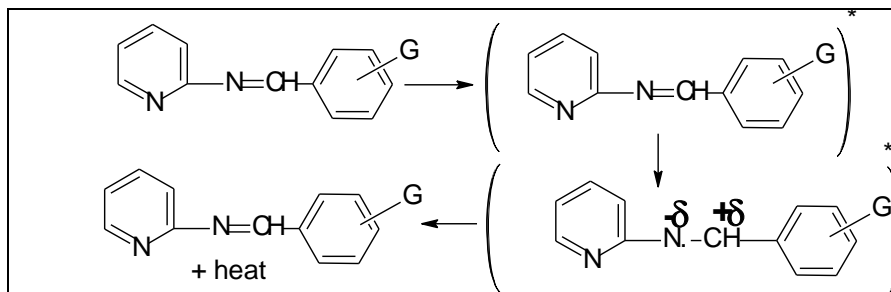
**Scheme (1) The suggested mechanism of photostabilization of PVC by 2-(2-pyridylimino methyl) phenol compound through absorption of UV light and intermolecular proton transfer (internal conversion IC).**



**Scheme (2) The suggested mechanism of photostabilization of PVC by 2-(2-pyriylimino methyl) phenol compound through absorption of Uv light and intermolecular proton transfer (intersystem crossing ISC).**

Another mechanism [30] explains the use of these compounds as photostabilizer is by change separated species, which could be inform the excited state such a structure would

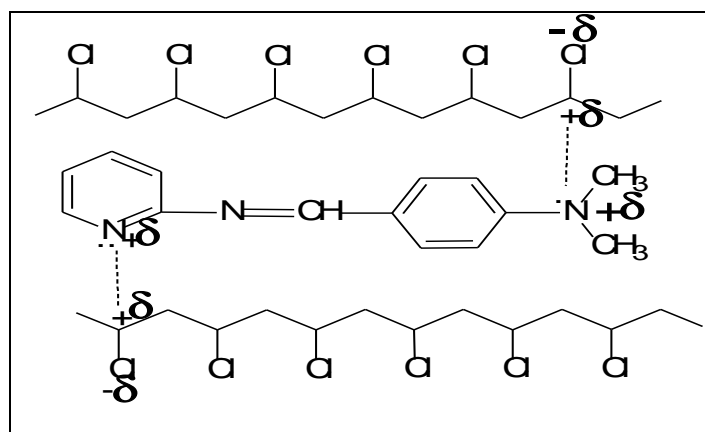
allow dissipation of energy through rotation on increased vibration about the control bond as shown in Scheme (3).



**Scheme (3) The suggested mechanism of photostabilization of PVC by 2-(2-pyriylimino methyl) phenol compound as Uv absorber [30].**

The compounds are selected with two different atoms of different electronegativity such as nitrogen and sulfur [30].

The polarity of the compound explains the attraction between the stabilizer and PVC (Scheme (4)).

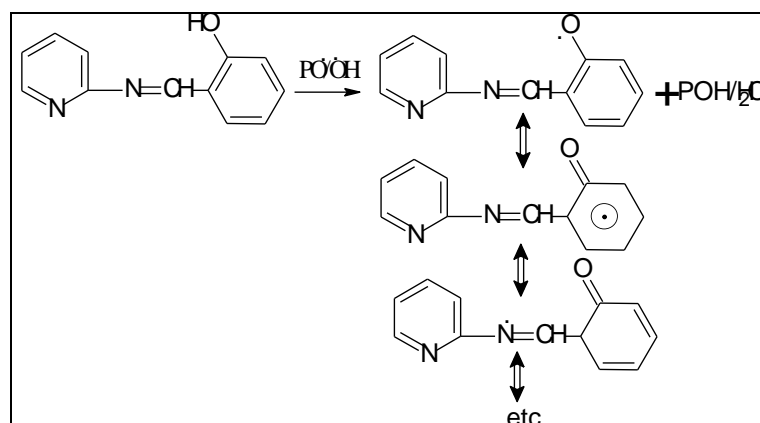


**Scheme (4) The suggested mechanism of photostabilisation of PVC by N-[4-dimethylamino] benzylidene-pyridine-2-amino) through the interaction between PVC and Schiff base [15].**



The hydroxyl group of the additive might act as radical scavenger for photostabilization process, therefore, these Schiff bases besides

acting as UV absorber they may also act as radical scavenger additives, Scheme (5).



**Scheme(5) The Suggested mechanism of photostabilization of PVC by 2-(pyridin-2-ylimino)methyl phenol as radical scavenger.**

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## الخلاصة

تم من خلال هذا البحث تحضير قواعد شف (1-6) وذلك بتكاثف الألديهيدات الأروماتية:

(benzaldehyd, salicylaldehyde,  
 ρ-methoxybenzaldehyde,  
 ρ-ydroxybenzalsehyde, 2-amino pyridin

مع 2- أمينو بيريدين (2-furan carboxyladehyde)

شخصت قواعد شف المحضرة من خلال مطيافية الأشعة تحت الحمراء (FTIR) والأشعة البنفسجية، المرئية (Uv.Vis)، التحليل الدقيق للعناصر (C. H. N.) وبعض الخواص الفيزيائية الأخرى كقياس درجة الانصهار. استخدمت قواعد شف كمضافات إلى رقائق PVC لدراسة تأثيرها في الاستقرار الضوئي لبوليمر PVC التجاري، وتم متابعة سرعة التجزئة والاستقرارية الضوئية لرقائق بسلك (40µm) والحاوية على المضافات المحضرة بنسبة وزنية (0.3%) مع زمن التشعيع بقياس قيم معاملي الكربونيل (Ico) والبولين (Ipo). استخدم في تشعيع الرقائق البوليمرية ضوء بطول موجي (313nm) وبدرجة حرارة 40°C. كذلك تم دراسة تركيز المضافات في المدى بين (0.1-0.5) % على التجزئة والتثبيت الضوئي لرقائق PVC، وأظهرت النتائج أن سرعة التثبيت الضوئي تزداد بزيادة تركيز المضاف. كما تم دراسة تأثير سمك الرقائق البوليمرية على التجزئة والتثبيت الضوئي، ووجد أن زيادة سمك الرقائق البوليمرية تقلل سرعة التجزئة الضوئية.

وأظهرت النتائج أن جميع المضافات تعمل كمثبتات

ضوئية لبولي فينايل كلورايد وحسب الترتيب التالي :

PVC > [1] > [3] > [6] > [4] > [5] > [2]

وفقا للنتائج العملية المستحصلة تم اقتراح الميكانيكة

والتي تعتمد علي تركيب المضافات كمتصات للاشعة فوق

البنفسجية وصائدات الجذور الحرة.