

Photostabilization of polystyrene films by anthraquinones derivatives and their complexes with copper (II), oxovanadium (IV) and nickel (II) ions.

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Abstract:

In the present work the photostabilization of polystyrene film (150 ± 5 μm thickness) has been studied using two anthraquinones, A and B derivative and six complexes of this compounds with three ions, copper (II), oxovanadium (IV) and nickel (II), these are:

1. [Bis (1,4-dihydroxyanthraquinone)] Copper (II).
2. [Bis (1,4-dihydroxyanthraquinone)] Oxovanadium (IV).
3. [Bis (1,4-dihydroxyanthraquinone)] Nickel (II).
4. [Bis (1-amino-4-hydroxyanthraquinone)] (Copper (II)).
5. [Bis (1-amino-4-hydroxyanthraquinone)] Oxovanadium (IV).
6. [Bis (1-amino-4-hydroxyanthraquinone)] Nickel (II).

All this compounds and complexes were identified by FT-IR and UV-Vis. Spectroscopy. The melting point of these complexes were determined. The degree of photodegradation was monitored by FT-IR and UV-Visible spectroscopy techniques and also by the loss in weight and viscosity. The two anthraquinone derivative and their prepared complexes give a good stabilization with compare with unstabilized polystyrene especially Ni complexes and copper complexes which give a good resistance for degradation than anthraquinones only.

Keywords: Photodegradation, Anthraquinones, Stabilizers, polystyrene

1. Introduction

Polystyrene (PS) is one of the most ubiquitous of commercial polymers. Due to the huge variety of its applications, photo initiated degradation and oxidation in air are important issues. These decomposition processes have been reported to typically occur over the range of 250– 400 nm [1], and can be initiated even by ordinary sunlight [2]. According to the accepted terminology, photodegradation refers to light-initiated chain scission, crosslinking and other processes under vacuum or inert atmosphere. On the other hand, oxidative photodegradation (or degradative photooxidation) occurs in air and includes, in addition to the processes described above, the formation of various oxygen-containing functional groups such as hydroperoxides or carbonyls. In this work, the term photodegradation will be used in general, and the environment in which it occurs will be obvious from the context.

Numerous mechanisms for PS photodegradation have been proposed over the years [3], but a totally

consistent theory is yet to be agreed upon, due to the complexity of the kinetics and the formation of various photodegradation products. An accepted classical mechanism [1,4] for PS degradation under ultraviolet (UV) light is given in Scheme 1. When irradiated at $\lambda < 300$ nm, PS forms the PS radical (Eq. (1)), which in air leads to a peroxy radical, and eventually a PS hydroperoxide (Eq. (2)). The latter photolysis into an alkoxy radical (Eq. (3)), leading to chain scission with the formation of shorter chain PS radicals and carbonyl species (Eq. (4)). In addition to chain scission, conjugated alkenes also form in the aliphatic portion of PS through stable polyene radical intermediates (Eq. (5)). Polyene formation (which causes, in addition to other factors, yellowing in degrading PS) is the major decomposition process occurring when PS is irradiated under vacuum. The mechanism above shows that the reactive PS hydroperoxide is formed upon exposure to atmospheric oxygen. However, the photodegradation process can be initiated by in-

chain structural defects [5,6], such as hydroperoxides formed during the polymerization of styrene and subsequent processing.

The objective of this work was to investigate the influence of the addition of two types of anthraquinones derivative, 1,4-dihydroxy anthraquinone (A) and 1-amino-4-hydroxy

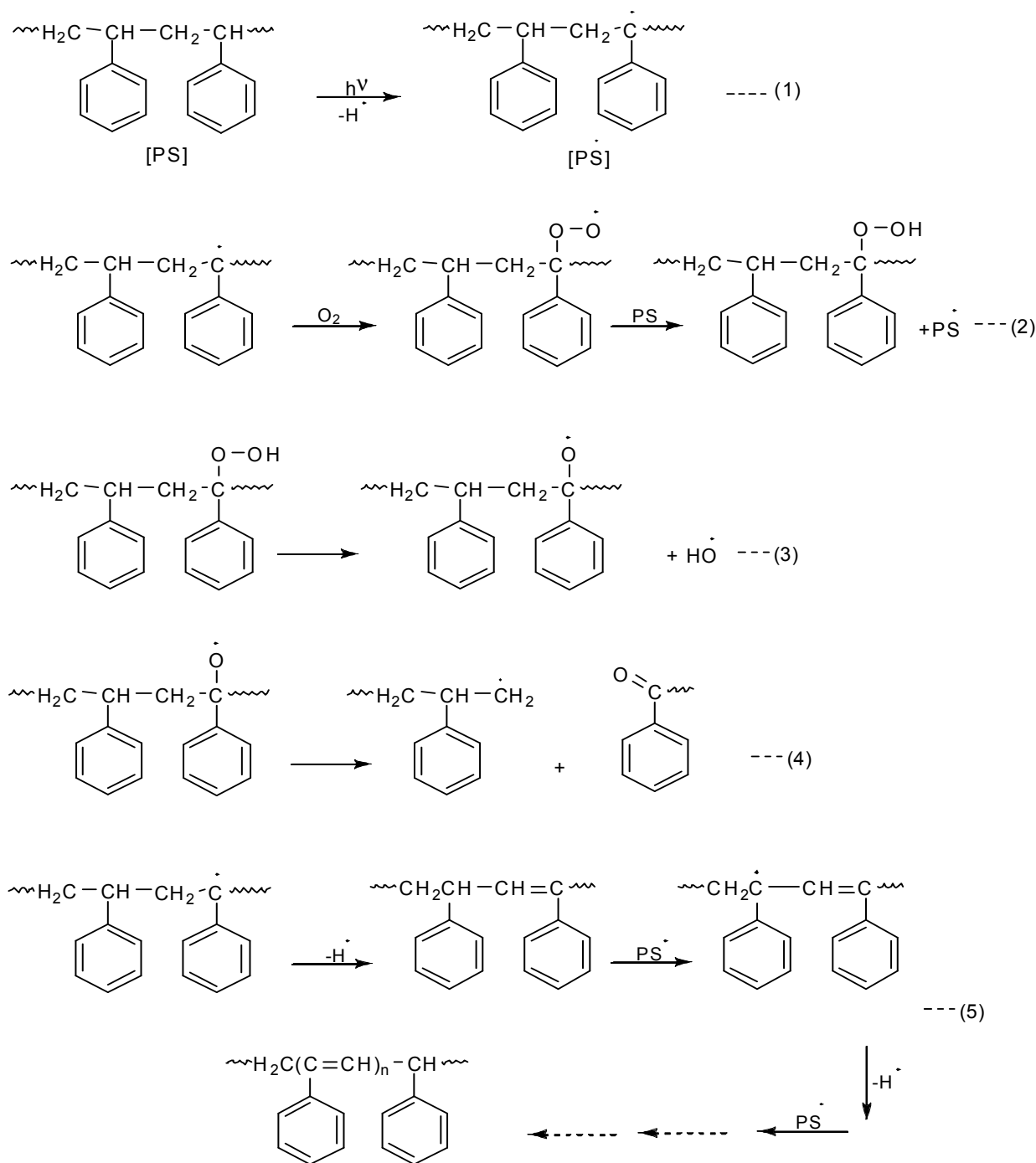
anthraquinone (B) and their complexes with copper, nickel and vanadium on the photostabilization of polystyrene, aiming at the improvement of the stability of this polymer when exposed to natural environment. The oxidative photo-degradation was carried out using an artificial of UV.

2-Experimental

2-1-Materials

Ethanol, methanol, chloroform and THF used were purchased from BDH, polystyrene (average

Mw 230000) were purchased from state company for



Scheme 1. Mechanism for photodegradation of polystyrene

petrochemical industries, Copper (II) chloride dihydrate, Vanadyl sulfate pentahydrate from

Fluka, Nickel (II) acetate tetrahydrate from Merck.

2-2- procedures

2-2-1- Preparation of 1-amino-4-hydroxyanthraquinone

A 10.4 g (0.05 mole) of sulfanilic acid dehydrate is diazotized by dissolving in 20 ml of conc.HCl and diluting it with 30 ml of distilled water. The solution was cooled in an ice-bath to below 5 °C. Sodium nitrate (7 g) was dissolved in 30 ml of distilled water and this solution was cooled in ice bath. Then the sulfanilic acid solution was diazotize by adding sodium nitrate solution with stirring, and the diazonium salt is kept in ice-bath. 11.2g (0.05 mole) of 1-hydroxyanthraquinone is dissolved in the warm solution of 11g of sodium hydroxide in 60 ml of water, the solution is cooled to 25 °C, 40 g of ice is added, and the suspension of the diazonium salt is then introduced, the mixture is

stirred well and allowed to stand for one hour. The solution is warmed to 45-50 °C, about 5 of 25 g of technical sodium hydrosulfite is added cautiously, and the mixture is stirred until the froth subsides, the remainder of the hydrosulfite is then added. The tan suspension of the 1-amino-4-hydroxyanthraquinone is heated to about 70 °C to affect sufficient coagulation to permit filtering. It is the cooled to 25 °C by stirring in ice-bath, then the precipitate is filtered and washed with 1% sodium hydrosulfite solution. Recrystallization from aqueous ethanol.

2-2-2- Complex I [Bis (1,4-dihydroxyanthraquinone)] Copper (II) (ACu)

A 1,4-dihydroxy anthraquinone (4 mmole) in absolute ethanol (25 ml) was added with stirring to aqueous solution of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (2mmole) at room temperature, a brown precipitate formed was

filtered and washed several times with methanol and diethyl ether, dried in vaccum , decomposition point 295C°, yield 82%

2-2-3- Complex II [Bis (1,4-dihydroxyanthraquinone)] Oxovanadium (IV) (AVO)

The preparation method and recrystallization method is similar to the above, using $\text{VO}_2 \cdot 5\text{H}_2\text{O}$

instead of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$. Orange precipitate was formed. Decomposition point 299 C°, yield 74%

2-2-4- Complex III [Bis (1,4-dihydroxyanthraquinone)] Nickel (II) (ANi)

The preparation method and recrystallization method is similar to the preparation method of complex I, using $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ instead of

$\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$. A yellow precipitate was formed. Decomposition point 290 °C , yield 86%

2-2-5- Complex IV [Bis (1-amino-4-hydroxyanthraquinone)] Copper (II) (BCu)

1-amino-4-hydroxy anthraquinone (4 mmole) in methanol (30 ml) was added to aqueous solution of $\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$ (2 mmole). The mixture was heated at 40 °C with continues stirring for 20 minutes. A

violet precipitate formed was filtered and washed several times with methanol and diethyl ether, dried in vaccum. Decomposition point 301 °C, yield 72%

2-2-6- ComplexV[Bis(1-amino-4-hydroxyanthraquinone)]Oxovanadium(IV) (BVO)

The preparation method and recrystallization method is similar to the preparation method of complex IV, using $\text{VO}_2 \cdot 5\text{H}_2\text{O}$ instead of

$\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$. A purple precipitate was formed. Decomposition point 305 °C, yield 81%

2-2-7- [Bis (1-amino-4-hydroxyanthraquinone)] Nickel (II) (BNi)

The preparation method and recrystallization method is similar to the preparation method of complex IV, using $\text{Ni}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$ instead of

$\text{CuCl}_2 \cdot 2\text{H}_2\text{O}$. A brown precipitate was formed. Decomposition point 293 °C, yield 75%

2-3-Films preparation

A 5% polystyrene in chloroform was used to prepare ($150 \pm 5 \mu\text{m}$) PS films [7] (measured by a micrometer type Mitutoyo (Japan)) containing 0.3% concentration of anthraquenones derivatives and metal chelate complexes. The films were

prepared by the evaporation the solvent at room temperature for 24 hours. To remove the possible residual chloroform solvent, films samples were further dried at 50 °C over night under reduced pressure.

2-4-Irradiation and degradation experiments

The degradation experiments were performed in a (Bausch & Lomb) mercury lamp with an intensity of 80 Wm^{-2} . The specimens used in degradation experiments, (dimensions $80 \times 20 \text{ mm}$), were cut

2-5- Characterization

The characterization of the degradation products was carried out by FT-IR spectrophotometer (FTIR-8400 Shimadzu-Japan) (University of Basrah, college of science) the absorption spectrum of the film samples were recorded in the range of ($4000-400 \text{ cm}^{-1}$). As a measure of photo degradation of PS film, the band at 1725 cm^{-1} was used.

The UV-Visible spectrophotometer type (spectro SC) (University of Basrah, college of

from thin films (about $150 \mu\text{m}$ thick). Samples were taken after various exposure times for irradiation (100,200,300,400,500 hrs.) and characterized by UV and FTIR spectroscopy

science) was used to measure the changes in the absorbance at $\lambda=400\text{nm}$ using 0.013 gm/ml solution in THF.

Also the photo degradation of polymer film were followed by the change in viscosity (intrinsic viscosity η_{int}) from 0.012 gm/ml solution in toluene using capillary ubbelohde, at $25 \pm 1 \text{ }^\circ\text{C}$.

The loss in the weight of degradable polymer was determined by using balance type (Sartorius-BL 1500S).

3. Result and discussion

3-1- Analysis of IR spectra of copper (II) and oxovanadium (IV) and nickel (II) complexes

The binding sites in these complexes are deduced from their infrared spectra. According to IR spectra of (A) and their complexes, we obtained that changing of the position of C=O stretching in ketone group and changing the shape and the position of O-H stretching and O-H out of plane deformation and C-O stretching and C-O deformation in phenol group as compared to the spectra of free ligand that indicate that oxygen atom in ketone group and oxygen atom in phenol group are involved in the coordination between (A) with Cu (II), VO (IV) and Ni (II) ions in their prepared complexes [8]. In addition, in complex ACu we obtained a new weak band centered at 472 cm^{-1} assigned to Cu-O stretching vibration. But in complex AVO, we obtained a new strong and sharp band centered at 985 cm^{-1} assigned to V=O stretching vibration and a new weak band centered at 431 cm^{-1} assigned to V-O stretching vibration. In complexes ANi we obtained a new weak band centered at 414 cm^{-1} assigned to Ni-O stretching vibration [9]. See figure (1).

According to IR spectra of (B) and their complexes, we obtained that C-O stretching (1299

cm^{-1} m) and C-O deformation (1167 cm^{-1} m, O-H out of plane deformation (724 cm^{-1} s) in phenol group that remains practically unchanged, indicating that O-H group is not taking part in coordination. However, changing the position of C=O stretching in ketone group and changing the shape and the position of NH_2 antisym. the sym. stretching and NH_2 deformation, NH_2 wagging, C-N stretching in amine group as compared to spectra of the free ligand indicate that oxygen atom in ketone group and nitrogen atom in amine group are involved in coordination to preparing (B) complexes [8],

In addition, in complex BCu we obtained new bands, the two weak bands centered at 470 cm^{-1} and 486 cm^{-1} are assigned to Cu-O and Cu-N stretching vibration. But in complex BVO, a strong and sharp band centered at 1132 cm^{-1} is assigned to V=O stretching vibration and a new weak band centered at 450 cm^{-1} is assigned to V-N stretching vibration. In complex BNi, a new weak band centered at 425 cm^{-1} is

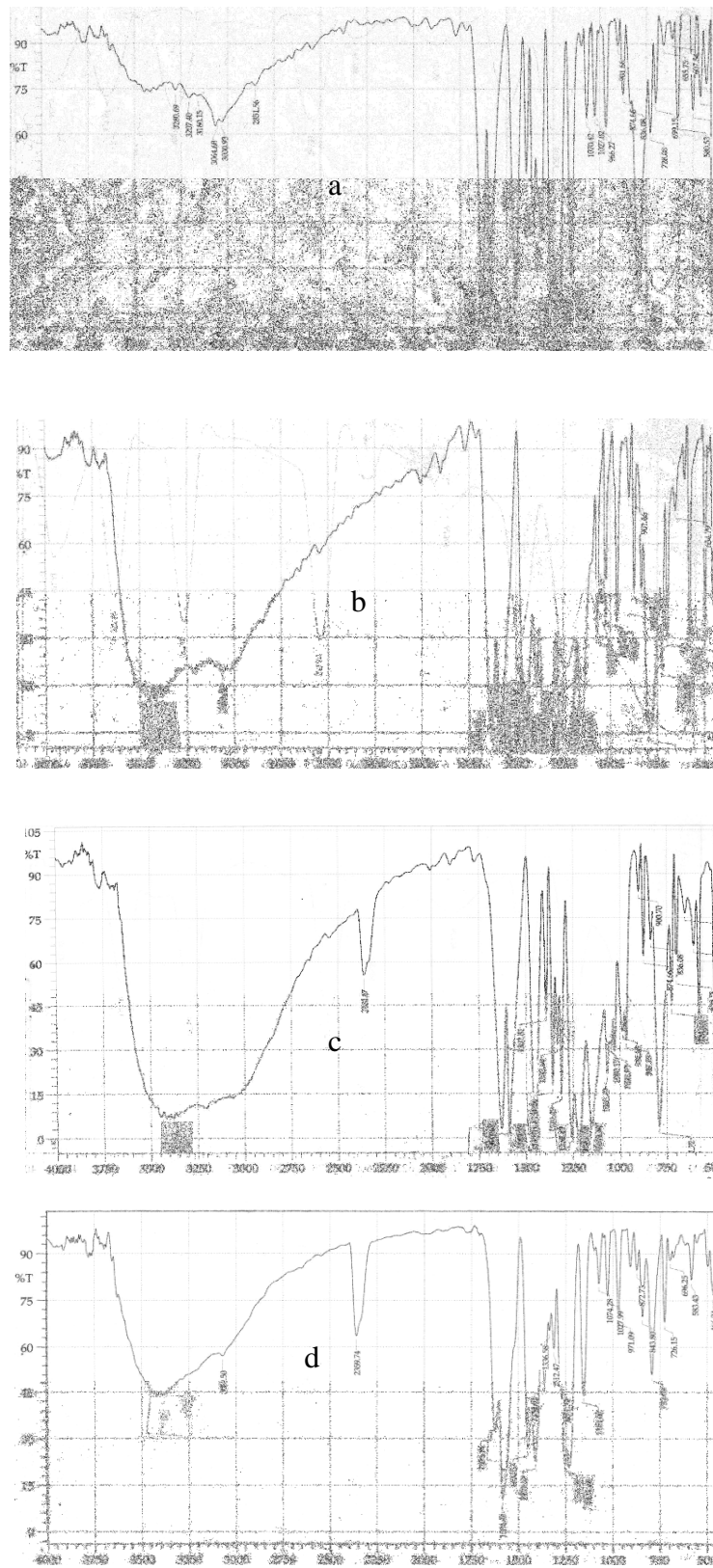


Figure (1): Infrared spectrum for A and complexes, (a): A, (b): ACu, (c): AVO, (d): ANi

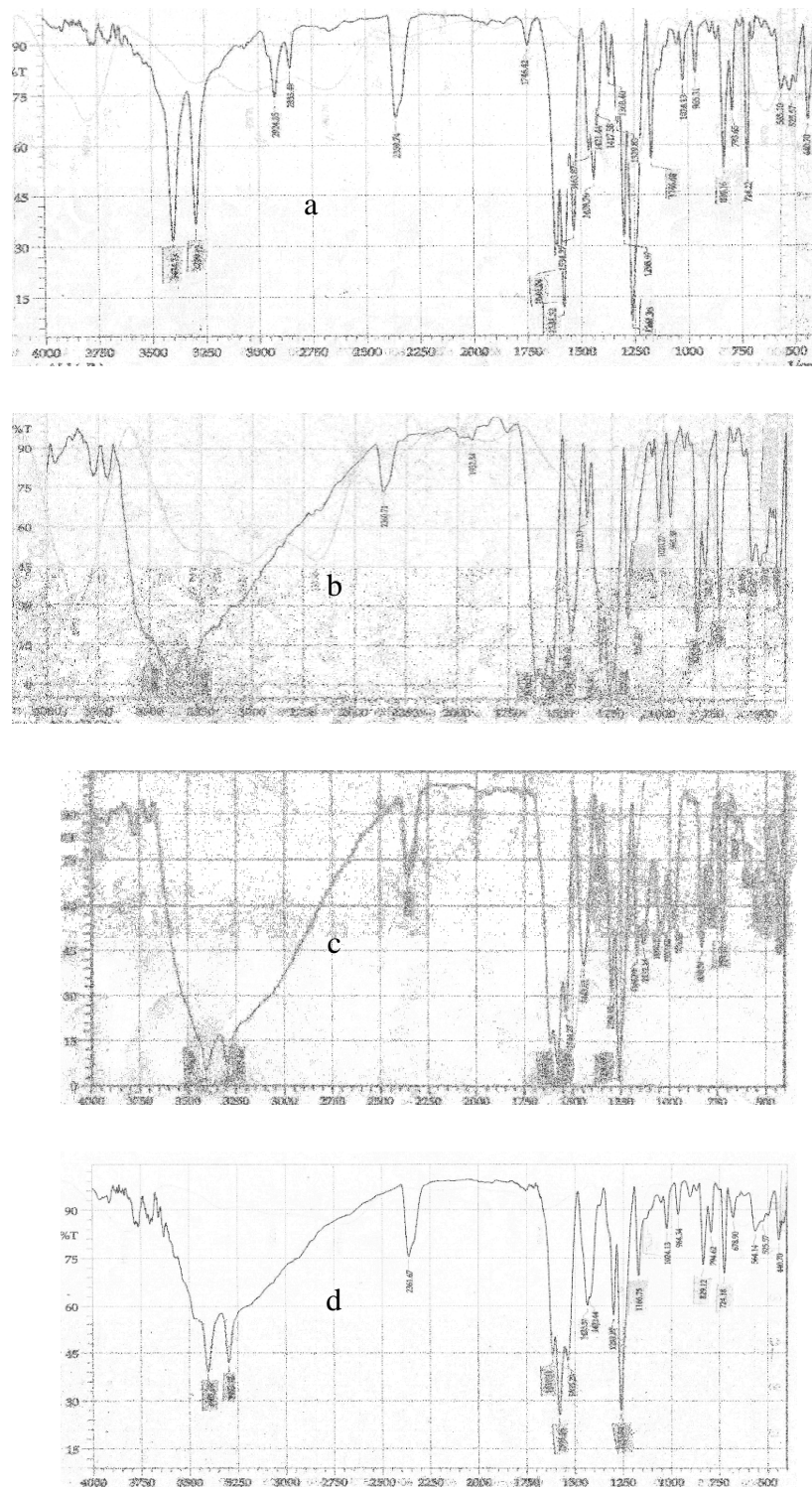


Figure (2): Infrared spectrum for B and complexes, (a): B, (b): BCu, (c): BVO, (d): BNI

assigned to Ni-N stretching vibration [9], See figure (2). Table (1) shows selected IR bands for A and B

and their complexes.

3-2- Analysis of UV spectra of copper (II) and oxovanadium (IV) and nickel (II) complexes

The electronic spectra of the complexes between (1,4-dihydroxy anthraquinone and 1-amino-4-hydroxy anthraquinone) with Cu (II), VO (IV) and Ni (II) ions were obtained in absolute ethanol by using UV-Visible spectrophotometer in preparing Cu (II) complexes, two intense absorption bands were observed due to transition ($b_{2g} \rightarrow b_{1g}$) and ($eg \rightarrow b_{1g}$) respectively and this state is similar to that of some Cu (II) complexes [10]. The values of molar absorbance ϵ are 485 and 356 for complex ACu and BCu. ϵ values were attributed to d-d transitions. According to D_{4h} symmetry [11], these absorptions are assigned as shown in table (2), see figure (3a), (4a).

obtained and this is similar to that in some VO (IV) complexes [12]. ϵ values are 510, 456 for complex AVO and complex BVO respectively, which one attributed to d-d transition too. According to C_{2v} symmetry [11], these absorptions are assigned as shown in table (3), see figure (3b), (4b).

In preparing Ni (II) complexes, three intense absorption bands were observed, the values of molar absorbance ϵ are 530 and 570 for complex ANi and BNi. ϵ values led us to attributed the bands to that kind d-d transitions. These absorption are assigned as shown in table (4), see figure (3c), (4c).

In prepared VO (IV) complexes, we noticed that complex AVO has three intense absorption bands. But in complex BVO, two intense bands were

Table (2): The assignment of the electronic transitions (cm^{-1}) for Cu (II) complexes.

Complexes	$\Delta E (b_{2g} \rightarrow b_{1g})$	$\Delta E (b_{2g} \rightarrow eg)$
ACu	17794	21053
BCu	17544	18657

Table (3): The assignment of the electronic transitions (cm^{-1}) for VO (IV) complexes.

Complexes	$\Delta E (b_2 \rightarrow e^* \pi)$	$\Delta E (b_2 \rightarrow b_1^*)$	$\Delta E (b_2 \rightarrow a_1^*)$
AVO	17986	19531	20833
BVO	17794	18939	-

Table (4): The assignment of the electronic transitions (cm^{-1}) for Ni (II) complexes.

Complexes	$\Delta E (A_{2g} \rightarrow B_{1g})$	$\Delta E (A_{2g} \rightarrow E_g)$	$\Delta E (A_{2g} \rightarrow A_{21g})$
ANi	13793	15152	25316
BNi	17513	19455	20833

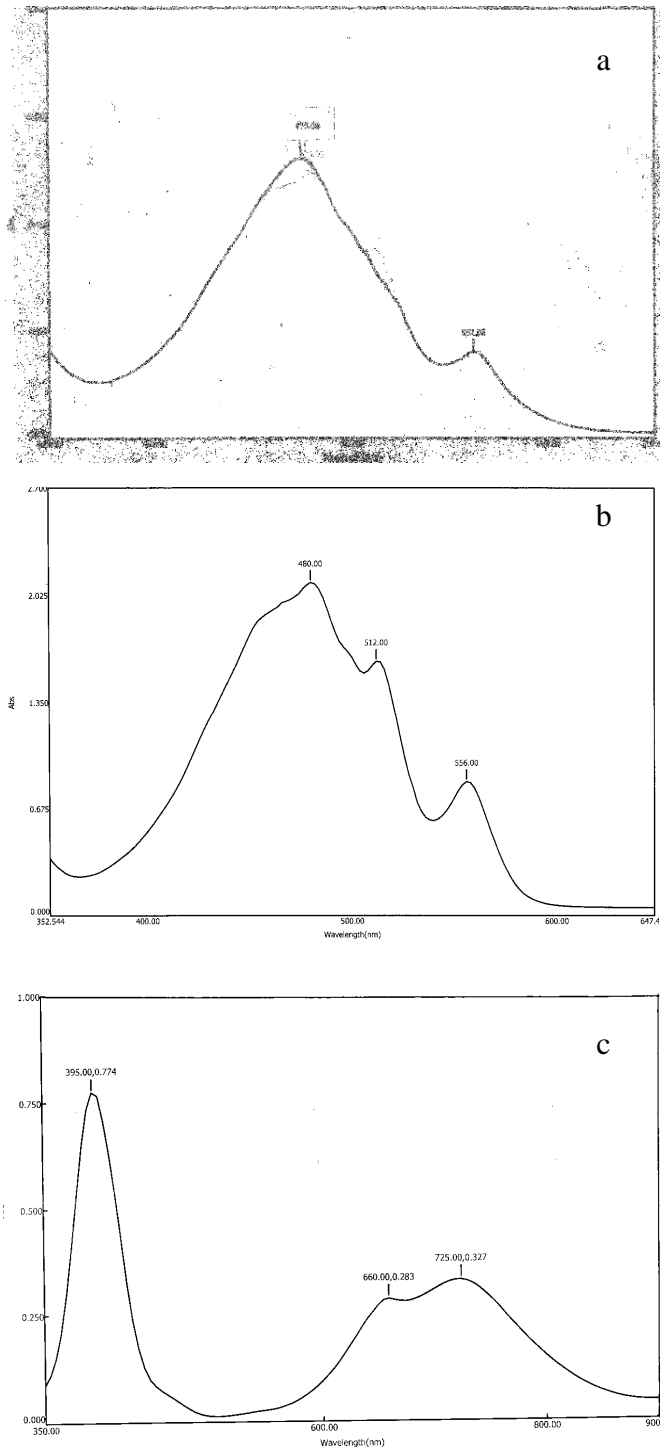


Figure (3): Electronic spectra for (a): ACu, (b): AVo, (C): ANi

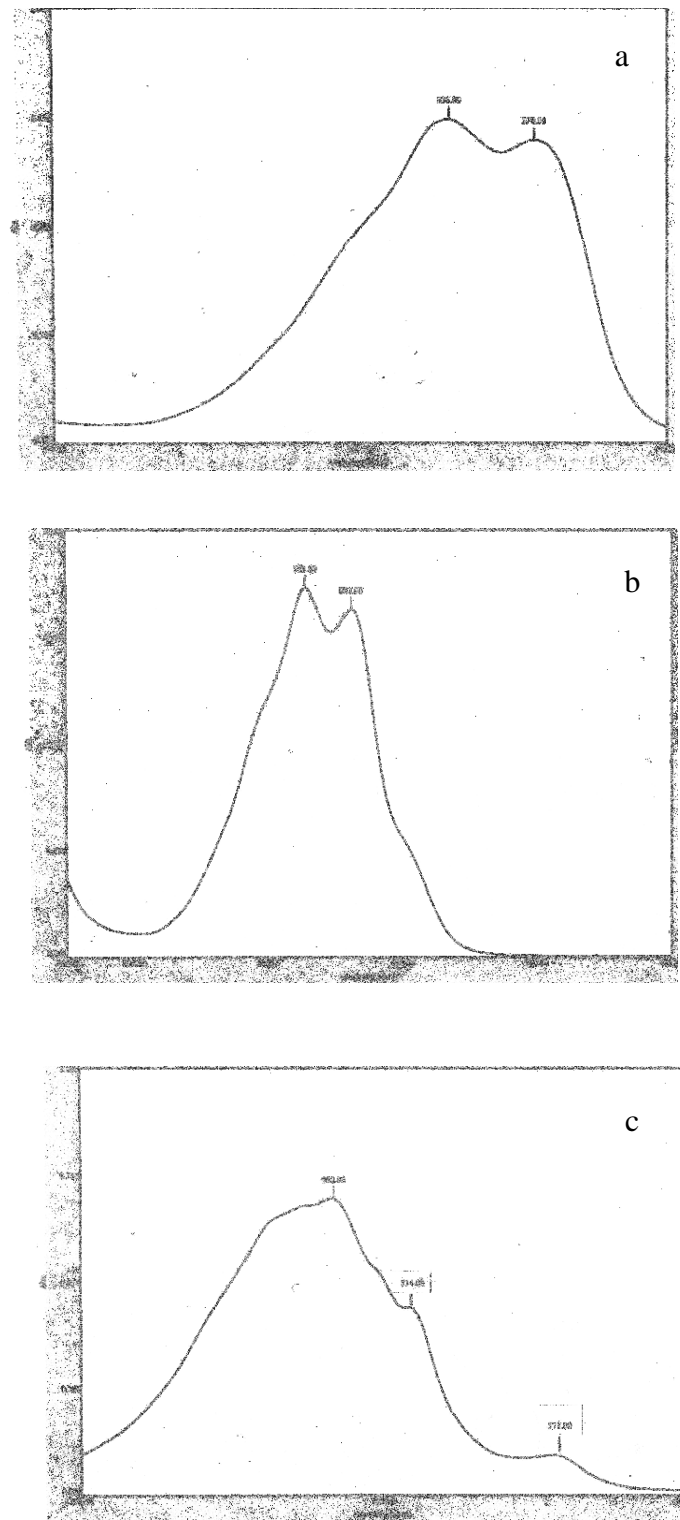
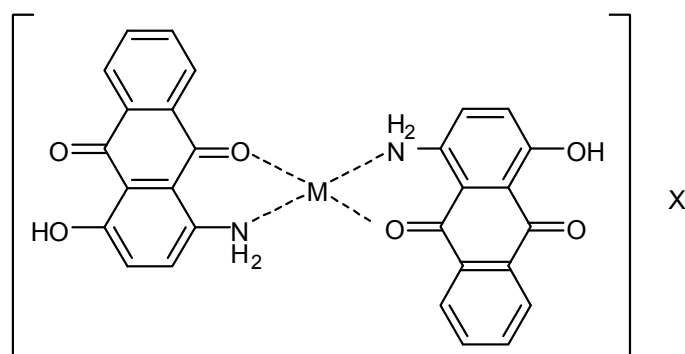
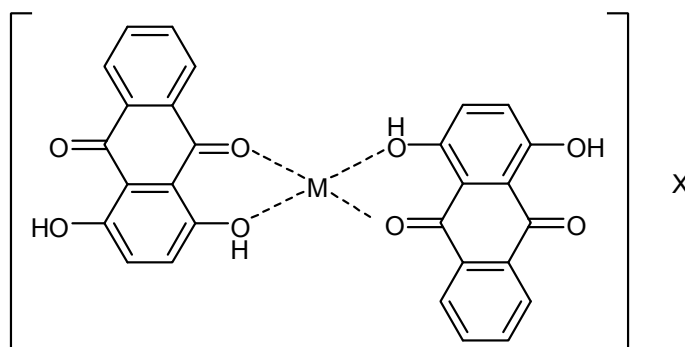


Figure (4): Electronic spectra for (a): BCu, (b): BVo, (c): BNi

According to IR spectra and UV-Visible spectra for (1,4-dihydroxy anthraquinone and 1-amino-4-hydroxy anthraquinone) and their complexes with Cu (II), VO (IV) and Ni (II) ions, it is possible to

suggest the geometrical structure for them, as shown below:

2 :1 (ligand : metal)



M=Cu, X=Cl₂

M=Vo, X=SO₄

M=Ni, X=(CH₃COO)₂

3-3- FTIR spectroscopy characterization

The changes in the chemical structure produced by photoirradiation in the PS films were monitored by FT-IR measurements as a consequence of chemical degradation, characteristic bands appeared and increased in the infrared spectra and it was possible to follow their development as a function of the exposure time. In PS films, the main changes of spectra were found in the 1900-1500 cm⁻¹ regions corresponding to the domains of C=O stretching vibrations, several maxima or shoulders were observed in the carbonyl band (at 1732, 1725, 1704, 1698 and 1688 cm⁻¹) and were well observed when the initial spectrum was subtracted from the spectra of the irradiated film [13]. As a measure of photodegradation of the PS film, we used the band at 1725 cm⁻¹ corresponding to carbonyl groups [14], and the carbonyl index was estimate from the ratio

of $A_{1725_{(t)}} / A_{1725_{(t_0)}}$, in which $A_{1725_{(t)}}$ and $A_{1725_{(t_0)}}$ were the absorbencies at 1725cm⁻¹ after and before photo-irradiation respectively, carbonyl index are plotted as a function of the degradation time in Fig (5).

Figure (5a) shows the change in carbonyl index after several times of irradiation, shows that a rapid increase in carbonyl index for unstabilized PS especially at 300 hrs. and up, this increase is less in PS stabilized with compound (A) and (B), and from the figure we observe that compound (B) is more effective as a stabilizer than(A). Figures (5a and 5b) were a comparison between A and B and their complexes for stabilization of polystyrene, from this figures we noted that all complexes are more the effective in the stabilization than anthraquinones by a little change in carbonyl index,

and we noted that nickel complexes with both A and B give good results for decrease the degradation of polymer, also copper complexes came in the second order for stabilization, then

3-4- UV-Visible spectroscopy characterization

Concerning the UV-vis. results, it is known that conjugated double bonds are formed during the photo-degradation of PS [15]. Usually, a suitable number of conjugated double bonds can be attributed to different bands in the spectra [16]. However, in our study and from figure (6) all complexes show low increase in absorption with comparison with unstabilized PS. It could be observed that yellowing was enhanced in unstabilized PS which can be attributed to the formation of chromophore groups. Figure (6a)

3-5- Viscosity characterization

The viscosity of a polymer solution depends on the concentration and the size (i.e. molecular weight) of the dissolved polymer. By measuring the solution viscosity we should be able to get an idea about molecular weight. Viscosity techniques are very popular because they are experimentally simple [17]. Several methods exist for characterizing the solution viscosity, or more specifically, the capacity of the solute to increase the viscosity of the solution. That capacity is quantified by using one of several different measures of the solution viscosity. The most common solution viscosity terms are: relative viscosity, specific viscosity, inherent viscosity and intrinsic viscosity [18]. In the present work we use the intrinsic viscosity as a measure of degradation of polymer, where if the degradation of polystyrene increases the viscosity decreases because the degraded polystyrene becomes low molecular weight than undegraded polymer because degraded polymer undergoes chain scission that decreases the molecular weight [19].

3-6- Weight loss and the decrease of molecular weight

Fig. (8a) show the photodegradation weight loss curves of the pure PS film and stabilized PS film by derivative A and B. The weight loss of poly styrene film increased gradually with the increasing degradation time [20], the increasing very small with PS film modified by A and B. fig. (8b and 8c) show the change in weight for polystyrene stabilized with A B and their complexes, this characterization proved that the complexes of

oxovanadium came third but all complexes are good stabilizers when compare with anthraquinones derivative.

shows that the effect of inthraquinone derivative (A) and (B) on the absorbance at degradation times, that compound (B) shows low absorbance than (A).figures (6b and 6c) shows the effect of complexes for compound (A) and (B) with nickel, copper and oxovanadium on the change of absorbance, that the complexes prepared are more effective of lowering the conjugated double bonds and less the yellowing especially with complexes A(Ni) and B(Ni).

Fig.(7) shows the change of the intrinsic viscosity with degradation time for PS with anthraquinone derivative, the slight decrease of viscosity observed for PS containing A and B stabilizers indicates that, under the same experimental conditions, the variation of molecular weight was very small with comparison with PS alone. On the other hand, the decrease observed for PS free from stabilizers is due to the formation of low molecular weight products after chain scission.

It could also be noted from figs. (7b and 7c) that the complexes of nickel, copper and oxovanadium with anthraquinones derivative A and B show low change in viscosity (especially with nickel) with comparison of A and B when added to PS. Thus, the results indicate that photodegradation of PS induces the scission of C-C bonds, which is strongly decreased by the presence of anthraquinones complexes and derivative.

nickel, copper and vanadium give stabilization more than derivative A and B.

From the characterization of carbonyl index, absorbance, viscosity and loss in weight we conclude that the prepared complexes have a good stabilizer and we can arrange it according to the increase capability to the stabilization of polystyrene as below:

Complexes of Ni> Cu> VO> B> A.

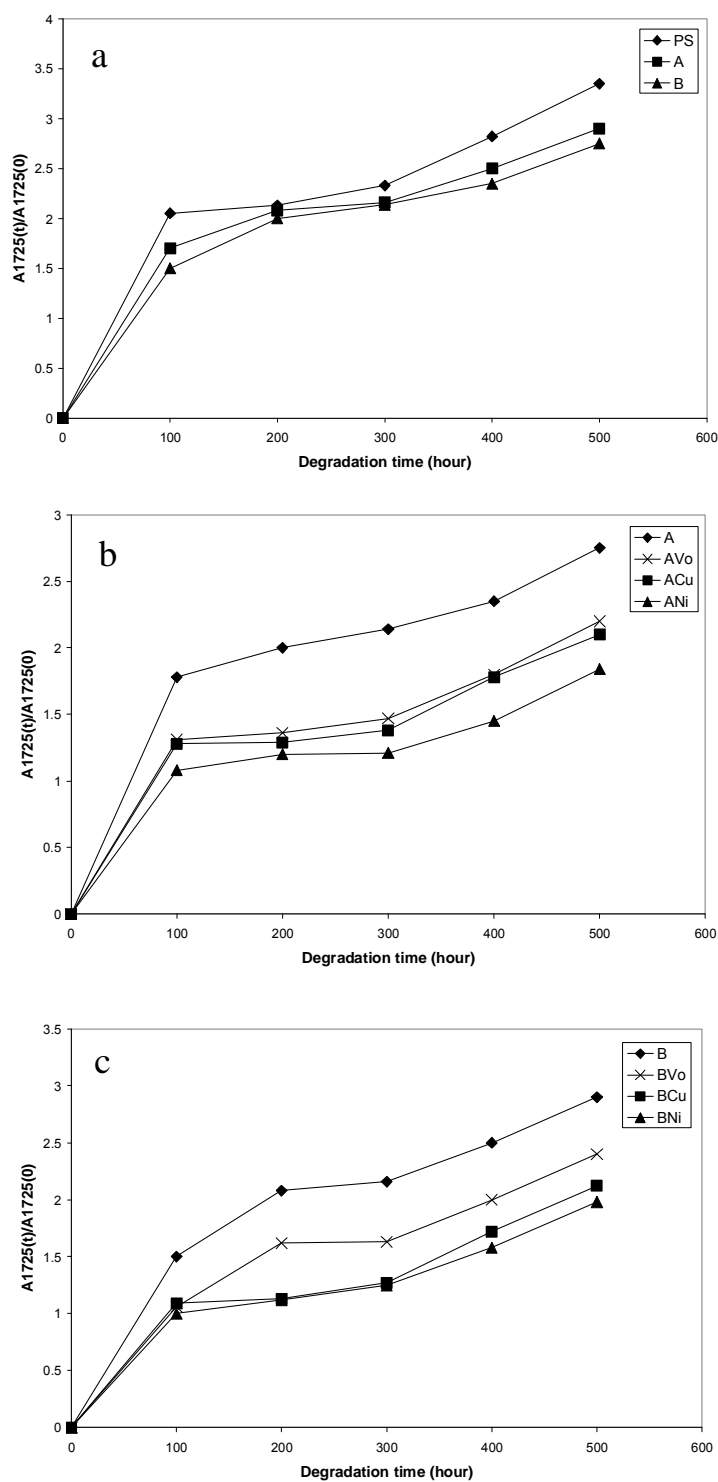


Fig. (5): Change of carbonyl index vs. degradation time for (a) PS and compounds A and B, (b) compound A and its complexes, (c) compound B and its complexes

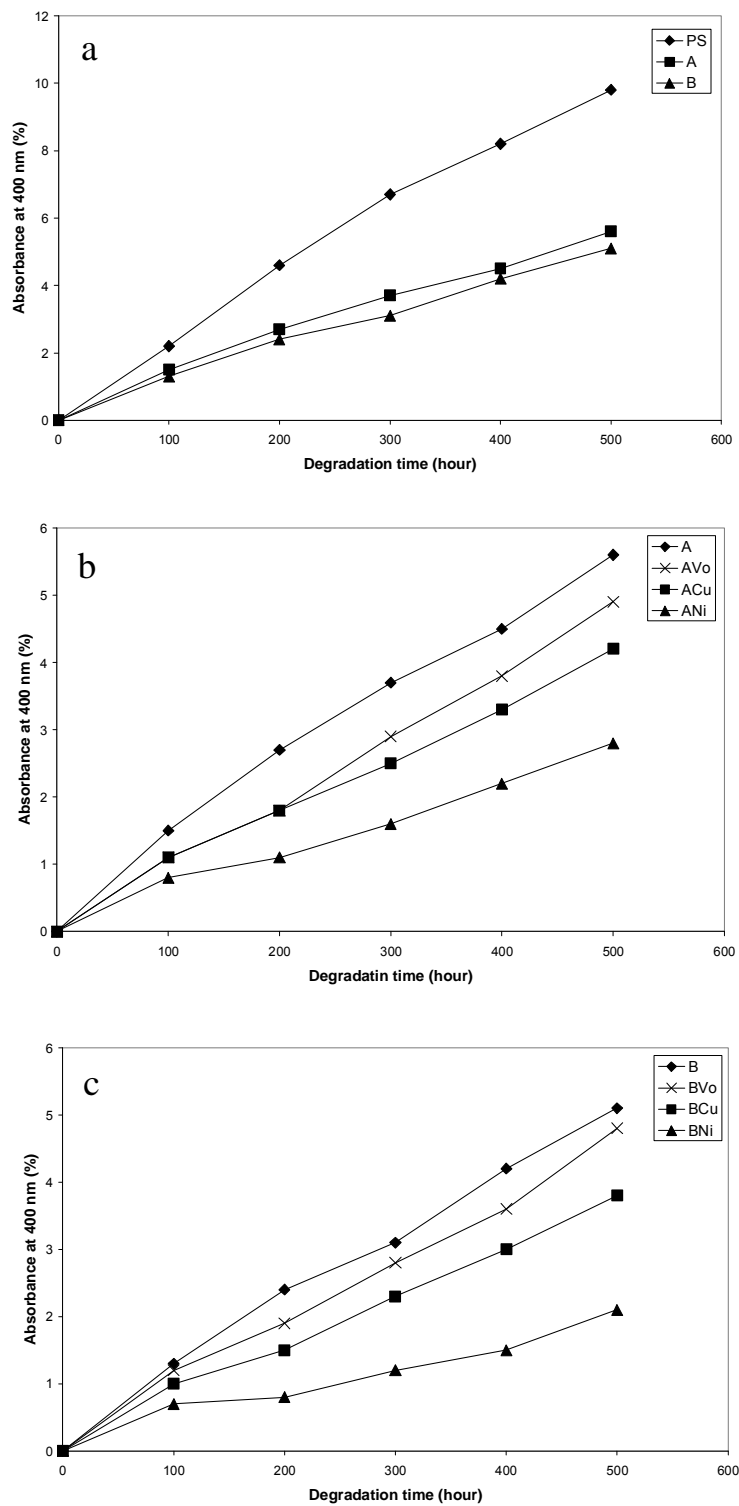


Fig. (6) : Absorbance at 400 nm vs. degradation time for (a) PS and compounds A and B, (b) compound A and its complexes, (c) compound B and its complexes

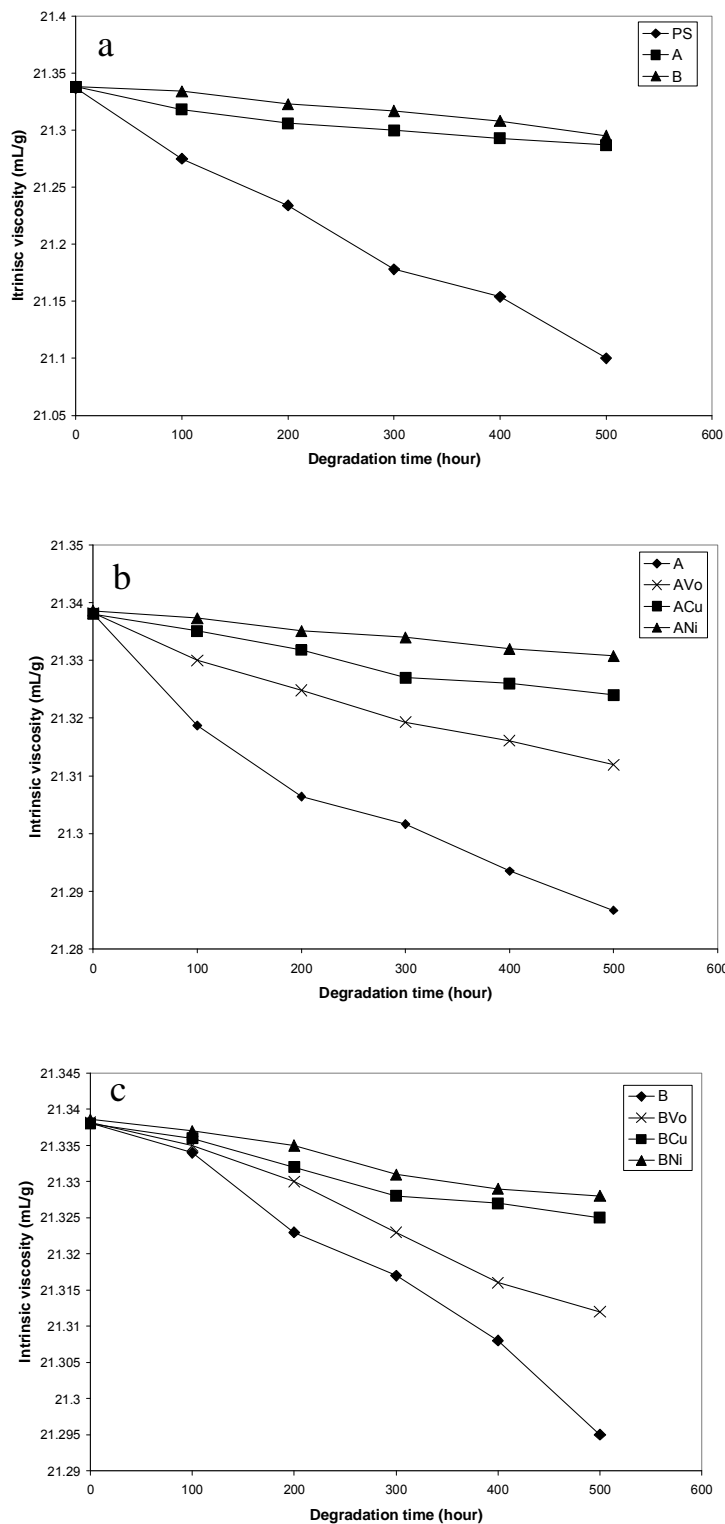


Fig. (7): Intrinsic viscosity vs. degradation time for (a) PS and compounds A and B, (b) compound A and its complexes, (c) compound B and its complexes

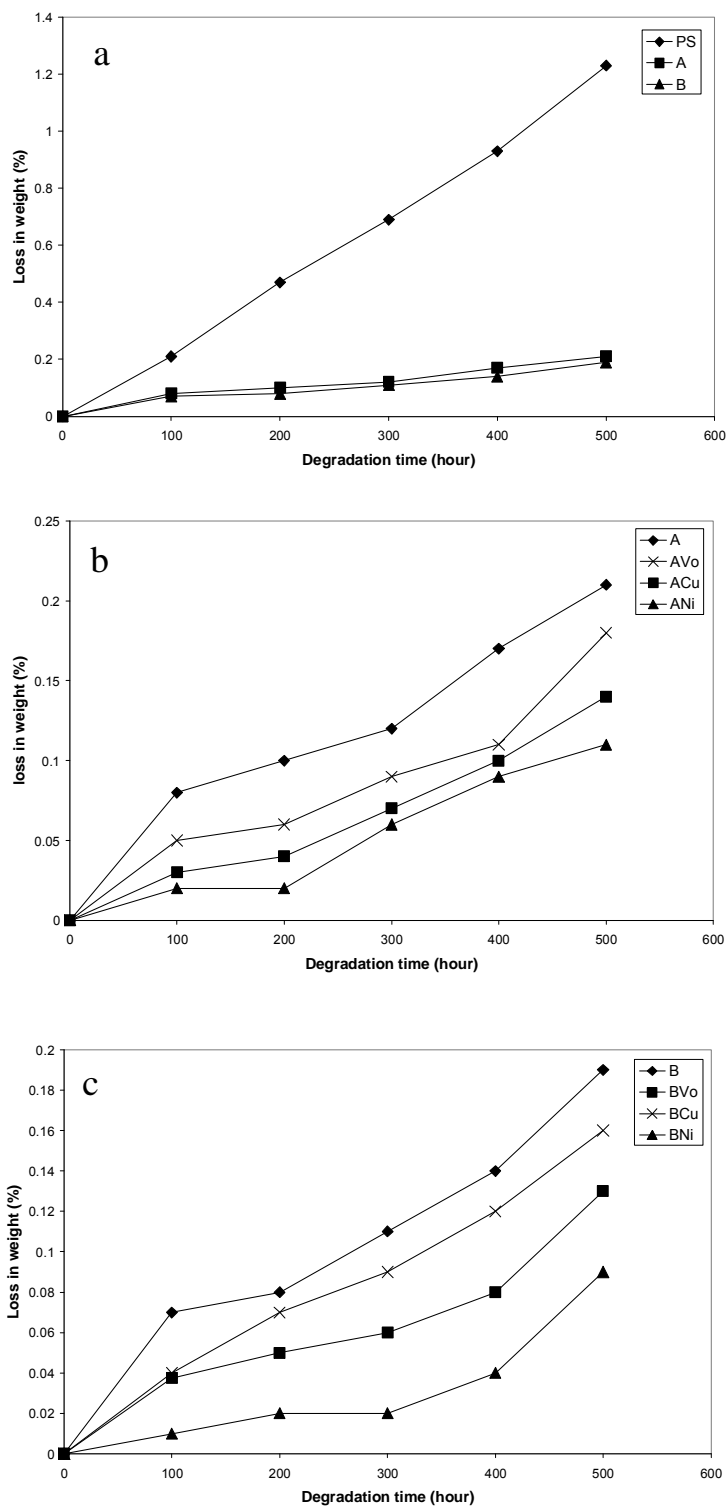


Fig. (8):Loss in weight vs. degradation time for (a) PS and compounds A and B, (b) compound A and its complexes, (c) compound B and its complexes

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المخلص

تم في هذا البحث دراسة التثبيت الضوئي للبولي ستايرين (سمك $150 \mu\text{m}$) باستخدام نوعين من مشتقات الانثراكوينون A,B ومعقداتها الست مع ثلاثة ايونات وهي : النحاس (II) الاوكسوفناديوم (IV) , النيكل (II). هي :

- 1.[Bis (1,4-dihydroxyanthraquinone)] Copper (II).
- 2.[Bis (1,4-dihydroxyanthraquinone)] Oxovanadium (I).
- 3.[Bis (1,4-dihydroxyanthraquinone)] Nickel (II).
- 4.[Bis (1-amino-4-hydroxyanthraquinone)] Copper (II).
- 5.[Bis (1-amino-4-hydroxyanthraquinone)] Oxovanadium (IV).
- 6.[Bis (1-amino-4-hydroxyanthraquinone)] Nickel (II).

لقد شخصت جميع مشتقات الانثراكوينون ومعقداتها باستخدام مطيافية الاشعة تحت الحمراء والاشعة فوق البنفسجية , كما تم تعيين المحضره و تم تتبع درجة التفكك او التفسير الحراري باستخدام تقنيات الاشعة تحت الحمراء وفوق البنفسجية درجة التفكك للمعقدات بالاضافه الى فقدان بالوزن والزوجه ولوحظ من هذه الدراسه ان المعقدات المحضره اعطت تثبيطاً جيداً وخصوصاً معقدات النيكل (II) والنحاس (II) اكثر من مشتقات الانثراكوينون A,B اكثر من لوحدها