

## Color Changes, AFM and SEM Study of PVC/triorganotin (IV) - Cephalexin Complexes Samples Via UV Radiation

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### ABSTRACT

**T**hree organotin complexes were successfully produced in the presence of sodium hydroxide by the condensation reaction of cephalexin (ligand) and tri-organotin (IV) complexes. The complexes formed were given trigonal bipyramidal geometries based on spectrum measurements. These compounds were applied as additives (0.5 percent by weight) to reduce the photodegradation of polyvinyl chloride films (40  $\mu\text{m}$  thickness) when exposed to ultraviolet light at a normal temperature (max = 313 at an intensity of light =  $7.75 \times 10^{-7} \text{ ein dm}^{-3} \text{ S}^{-1}$ ). The weight changes of films before and after irradiation can be applied to investigate the effectiveness of organotin (IV) complexes as photo stabilizers. After irradiation, the morphology of poly(vinyl chloride) studied by the microscope, atomic force microscopy, and scanning electron microscope was shown to be less damaged in the films containing additives. These additives are effective UV absorbers, hydrogen chloride scavengers, and peroxide quenchers. Prog. Color Colorants Coat. 16 (2023), 283-294© Institute for Color Science and Technology.

### 1. Introduction

Organometallic compounds are substances that have directed greater or lesser polar connections between metallic and carbon atoms [1]. Thousands, if not millions of compounds, have been synthesized during the last five decades, with organotin materials receiving the greatest attention, Organotin compounds are among the most commonly employed organometallic compounds [2]. These compounds have a tin atom and an anion that is often hydroxide, oxide, fluoride, chloride, or thiolate [3]. Due to their diverse structural

makeup and numerous potential uses in a number of industries, organotin complexes are of tremendous interest [4, 5]. The research of various coordination modes, such as chelate, monodentate, or more subtle bridging, which can produce oligomeric or polymeric structures, is made possible by the coordination of carboxyl groups to organotin residues. Organotin (IV) carboxylates have been used in the stability of polyvinyl chloride (PVC), polyurethane synthesis, antifouling coatings, and silicone curing [6-8]. Organotin complexes are used as polyvinyl chloride stabilizers in the common of their applications due to their high reactivity.

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When exposed to ultraviolet light, PVC photo oxidizes, with the autocatalytic dehydrochlorination process causing the majority of the damage [9-11]. Polymeric stretch, cross-links, and chain scissions develop when hydrogen chloride is removed from PVC, as a result, discoloration, fractures, losses in total molecular weight, transparency loss, and mechanical property changes occur [12, 13]. To improve polymer resistance to free radical scavengers, UV absorbers, hydroperoxide decomposes and exactions energy varied quantities are required to utilize effect additives [14, 15]. To successfully absorb ultraviolet light and create excited state molecules that are stabilized by resonance, a broad range of additives has been explored for their protective properties on polyvinylchloride films [16, 17]. Organotin (IV) carboxylates are the most recently developed and widely used PVC additives that are exceptionally stable and excellent HCl scavengers [18]. Cephalixin is a white solid containing aromatic rings and heteroatoms that are stable and non-toxic, when mixed with PVC; it is regarded as an ultraviolet absorbent with no color change or environmental difficulties [19]. They also function as antiviral medications, inflammatory agents, and antibacterial substances [20, 21]. Organotin compound efficiency was revealed as dependent on the kind and amount of substituents added to the tin. In this study, we describe the design, and use of new tin-cephalexin complexes as PVC additives to minimize photooxidation and photodegradation. These complexes are expected to be more efficient PVC photo stabilizers than those previously reported because they contain alkyl and aryl groups in addition to heteroatoms like nitrogen, oxygen, and sulfur.

## 2. Experimental

### 2.1. General

Petkim Petrokimya had an abundance of PVC (MV= about 165,000, K-value = 67, degree of polymerization = 800). (Istanbul, Turkey). The KBr disc method was used to record Fourier transform infrared spectroscopy on the Shimadzu FTIR spectrometer 8400 (Tokyo, Japan) in the spectral region of 4000 - 400  $\text{cm}^{-1}$ . With the use of a Meiji Techno microscope (Tokyo, Japan), the PVC's surface morphology was examined, and Veeco equipment was used to log the atomic force microscope pictures (Plainview, NY, USA).

### 2.2. Synthesis of complexes 1–3

Tri-organotin(IV) complexes **1-3** (Figure 1) were made by reacting cephalixin with chloride triorganotin in MeOH under reflux for five hours, as was previously described [22]. Hence the molar ratios (metal: ligand) utilized to synthesize the complexes are 1:1, thus an suitable quantity of  $\text{Ph}_3\text{SnCl}$  (1.9273 g, 5.0, mmol),  $\text{Bu}_3\text{SnCl}$  (1.6275 g, 5.0 mmol), and  $\text{Me}_3\text{SnCl}$  (0.9964 g, 5.0 mmol) were dissolved in 20 mL of methanol and then added to a stirred solution of cephalixin (1.7369 g, 5.0 mmol) that dissolved in 30 mL of methanol with an equivalent amount of sodium hydroxide, After stirring for 10 minutes at room temperature, the mixture had been refluxed for 5 hours. The resultant solution was filtered, washed, dried, and recrystallized from methanol to produce pure target products (Scheme 1). Many spectroscopic and analytical findings supported the stated structures of synthesized compounds **1-3** and were compatible with them.

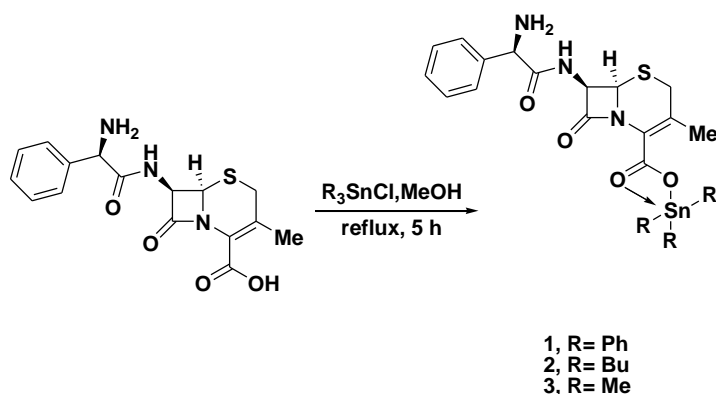


Figure 1: Synthetic route of tin-cephalexin complexes 1–3.

### 2.3. Preparation of PVC films

PVC (4 g) and tin complexes **1-3** (20 mg) were combined with 80 mL of THF and stirred for two hours. The final homogenous mixture was put into a glass plate with 15 holes that were approximately 40 micrometers thick. The plate was dried at room temperature overnight. To confirm that there were no remaining residues of THF, the generated films were dried in a vacuum oven at 50 °C for 6 hours.

### 2.4. IR spectroscopy of PVC

As a result of PVC photodegradation, tiny polymeric fragments with carbonyl (C=O) and alkene (C=C) moieties are produced. Dehydrochlorination, or hydrochloride (HCl) removal from the PVC chains, is the primary cause of this process [23]. PVC films were exposed to radiation at various exposure durations, and their FTIR spectra were captured. Both the C=O (1714 cm<sup>-1</sup>) and C=C (1618 cm<sup>-1</sup>) groups' absorption bands' intensities were measured and contrasted with a reference peak of C-H bonds; at 1332 cm<sup>-1</sup>. Equation 1 was used to determine the functional group index (I<sub>s</sub>) for IC=O and IC=C groups from the absorbance of the functional group (A<sub>s</sub>; AC=O or AC=C) and the reference peak (A<sub>r</sub>; AC-H).

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

### 2.5. Polymeric films weight loss

The PVC polymeric films were weighed before (W<sub>0</sub>) and after (W<sub>t</sub>) irradiation at different times (t). The PVC weight loss (%) as a result of irradiation was

calculated using Equation (2).

$$\text{Weight loss \%} = \frac{W_0 - W_t}{W_0} \times 100 \quad (2)$$

## 3. Results and Discussion

### 3.1. Polyvinyl chloride weight loss

While exposed to ultraviolet and/or high temperatures for longer, polyvinyl chloride (PVC) photodegrades. As a result of the release of hydrogen chloride (HCl) and the generation of molecular chains with double bonds, differences in the physical and mechanical properties of the polymer occur. When PVC is exposed to UV radiation (max = 313 at an intensity of light = 7.75 × 10<sup>-7</sup> ein dm<sup>-3</sup> S<sup>-1</sup>) at ambient temperature, bonding breaks and unsaturated fragments with low molecular weights generate [24]. With time, Poly (vinyl chloride) discolors crosslinks and scissions. PVC weight loss becomes more visible as photodegradation progresses. The obtained and reported findings are depicted in Figure 2. The weight loss percentage is substantially reduced when additives were used in comparison to the blank PVC. After 300 hours of irradiation, the weight loss percentage of blank PVC is about 0.9 while for blended PVC films they are less than 0.3 in all cases. In comparison these results to previous studies [7] when the weight loss approach was used to investigate the efficiency of organotin(IV) complexes as photostabilizers of PVC after irradiation by UV light using Sulfamethoxazole as a ligand. It found that using triorganotin (IV)/cephalexin complexes demonstrated excellent results to reduce the photo-degradation of PVC films after irradiation.

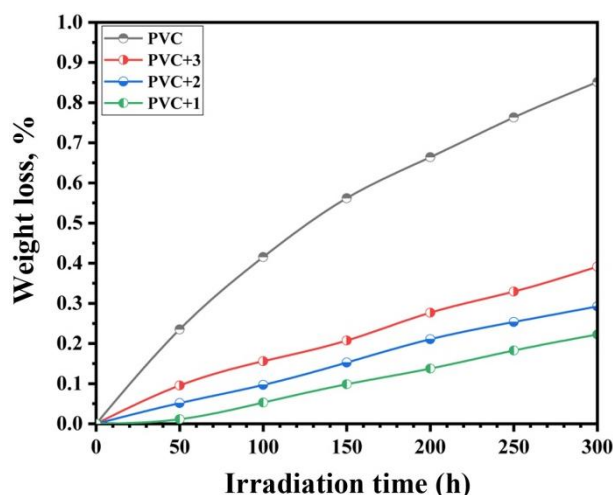


Figure 2: Shows how the UV exposure time affects the amount of weight loss in PVC films.

### 3.2. Fourier transforms infrared spectroscopy of PVC

The physical and chemical characteristics of polymers are impacted by ultraviolet light. Small fragments including hydroxyl, carbonyl, and polyene molecules are created during the photo-oxidation of polyvinylchloride. It is possible to measure and compare the intensity of peaks in the PVC films' fourier transform infrared spectra that are linked to the OH ( $3400\text{ cm}^{-1}$ ), C=O ( $1730\text{ cm}^{-1}$ ), and C=C ( $1620\text{ cm}^{-1}$ ) groups with the intensity of a reference peak ( $1332\text{ cm}^{-1}$ ) associated with the C-H bonds inside

the polymer chains. This kind of comparison offers crucial knowledge regarding the pace at which PVC photodegrades [25].

Figures 3 and 4 illustrate the variations in the infrared wavelengths of PVC (pure) film and PVC+Me<sub>3</sub>SnL film (mixed) after irradiation. The indices for the carbonyl (IC=O) and polyene (IC=C) groups were determined and displayed vs. irradiation time for each PVC film were shown in Figures 5 and 6. These values were substantially lower for the polymer films having organotin (IV) complexes **1-3** than for the blank film.

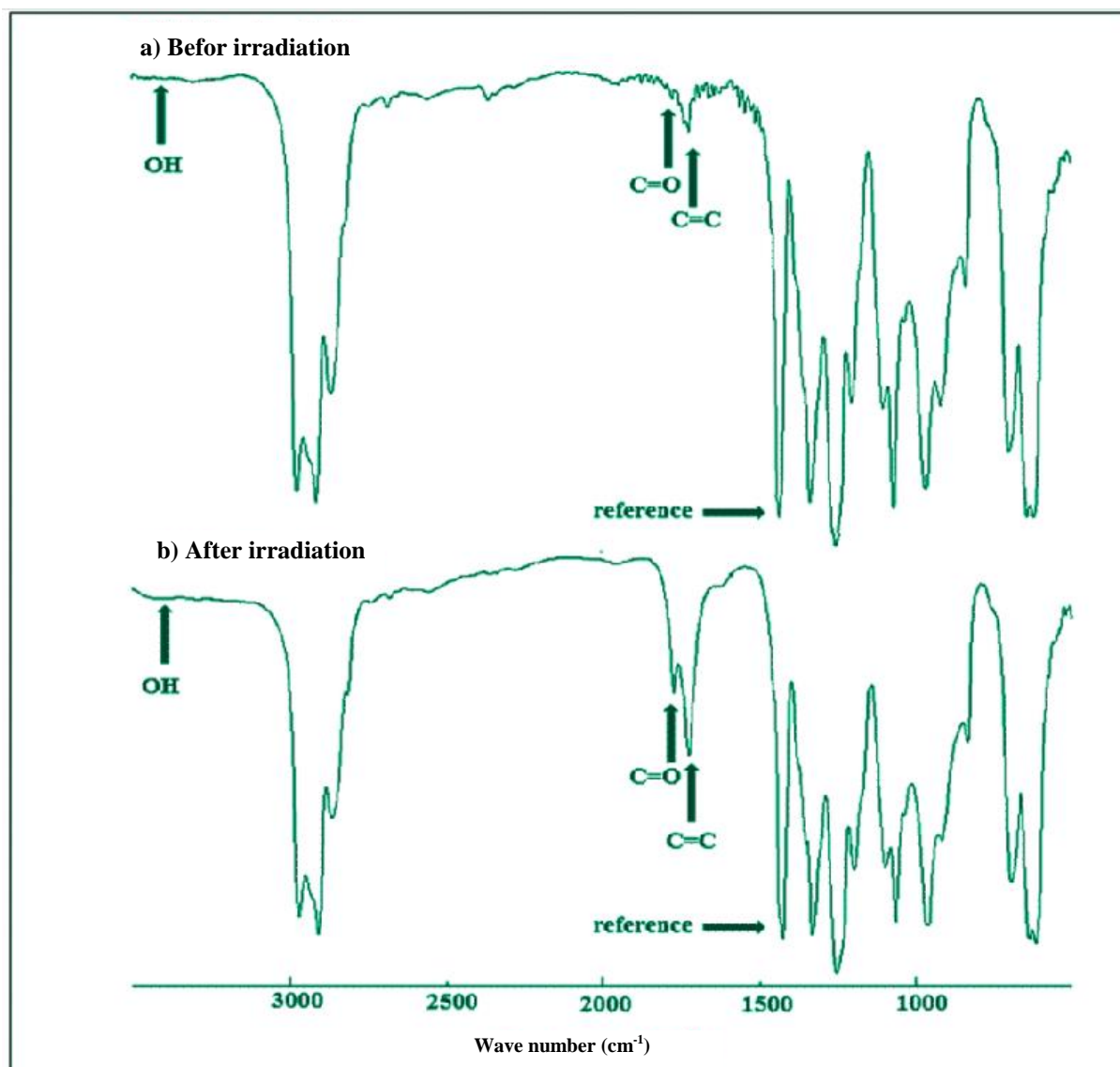


Figure 3: FT-IR spectra of blank PVC films.

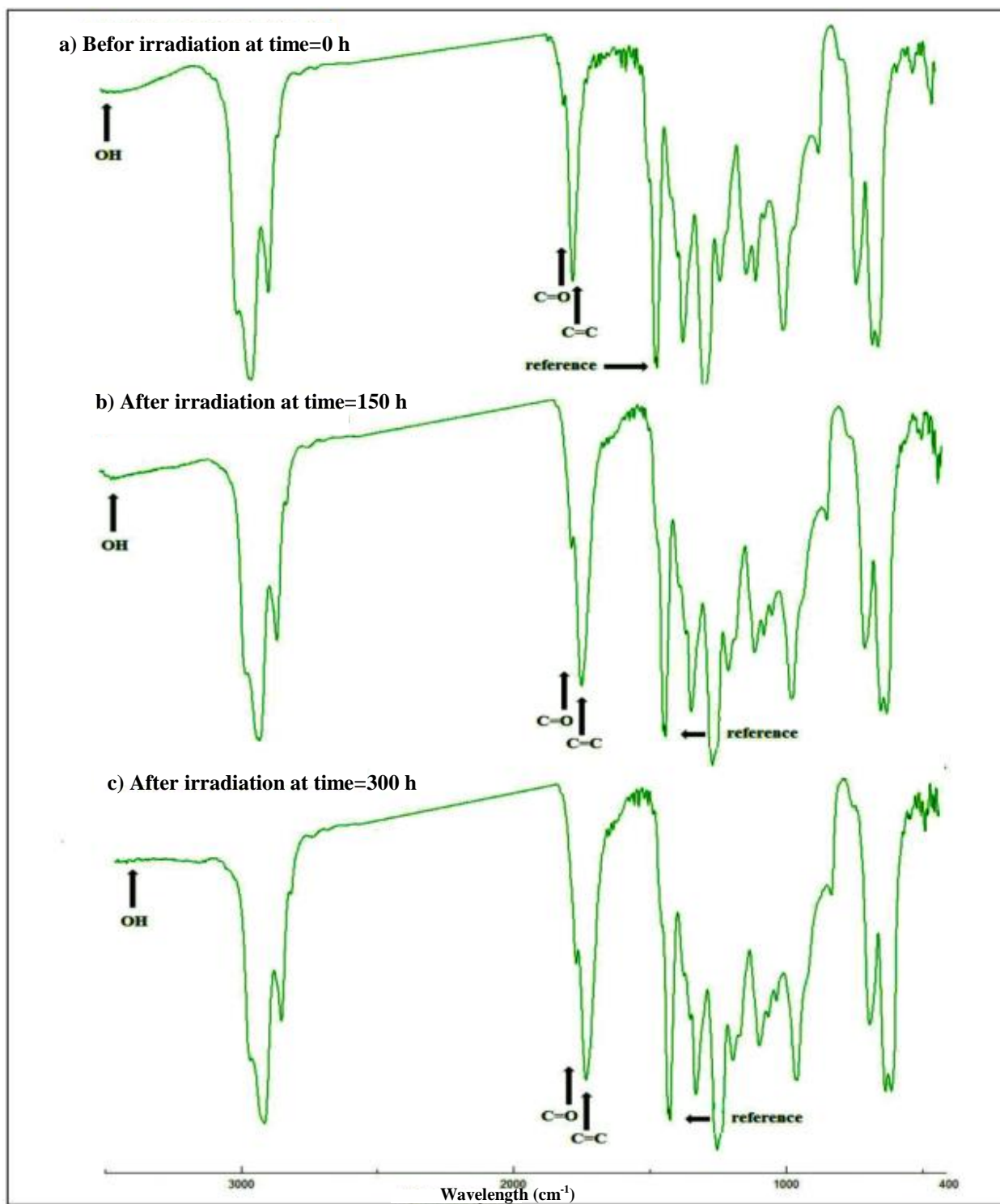


Figure 4: FT-IR spectra of doped PVC film containing Me<sub>3</sub>SnL complex.

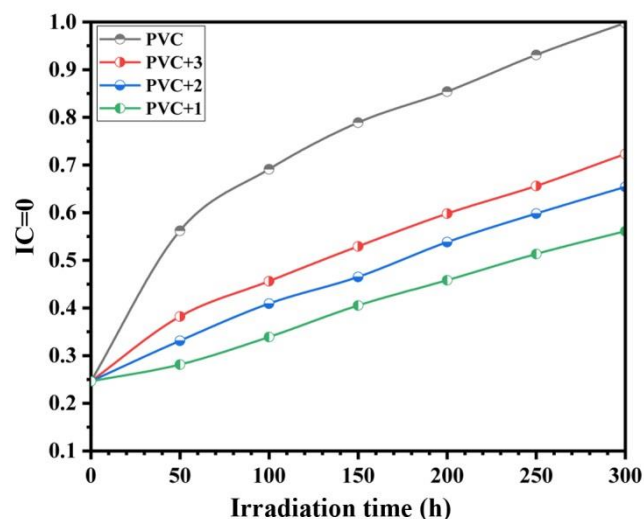


Figure 5: Irradiation's impact on the PVC films and growing of carbonyl group.

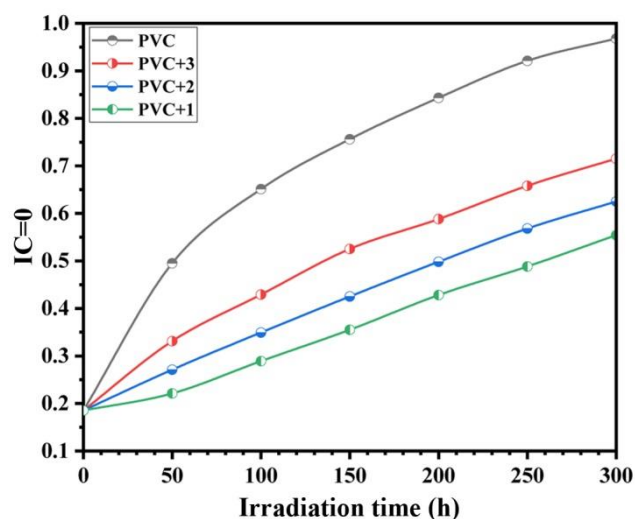


Figure 6: Irradiation's impact on the PVC films and growing of alkene group.

### 3.3. Surface morphology

#### 3.3.1. Microscopic analysis

Morphological microscopy offers information on the roughness and irregularity of the surface of a polymeric film, as well as defects within the surface of a material [26, 27]. It identifies faults, fractures, damages, breakdowns, scission, and other modifications inside the polymer surface after 300 hours of exposure to UV radiation. Optical microscope images of the polyvinylchloride before irradiation revealed very few dark areas and a smooth surface with. While the polymer surface after irradiation had some surface damage, such as holes, and dark spots, as well as color changes due to photo-degradation. However, surface defects in organotin (IV) complexes **1-3** containing

PVC films were less visible as compared to those on the surface of the blank PVC film. Figure 7 depicts the fact that such compounds can slow down the photodegradation process and hence enhance the photostability of irradiated PVC films. The presence of bubbles, especially in the blank sample, is observed, which is probably related to the release of the solvent in the preparation of the sample and before irradiation. These bubbles can be seen in other images. Therefore, it seems that they cannot be attributed to dehydrochlorination.

#### 3.3.2. Atomic force microscopy

According to the atomic force microscope with two- and three-dimensional images, the polymer (PVC) after

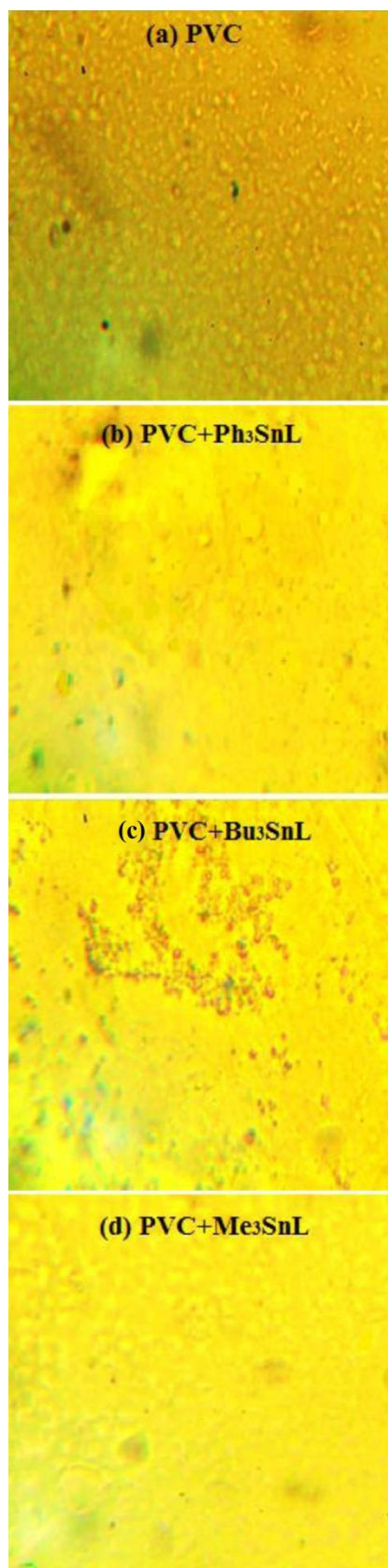
300 hours of irradiation has a rough surface with white spots and fractures. Although the polymer containing organotin (IV) complexes has smooth surfaces with fewer cracks as shown in Figure 8. The roughness factor ( $R_q$ ) may be used to evaluate the roughness of the PVC surface, which measures the degree of photo-degradation produced by volatile reduction in response to irradiation. When  $R_q$  is large, dehydrochlorination and bond breakage results in a rough surface. Dehydrochlorination is usually carried out at high temperatures [28]. Interestingly, the roughness value of blank PVC is 1.08 before irradiation while it is 216.95 after 300 hours of irradiation. However, using the additives showed much lower roughness values especially for the  $\text{Ph}_3\text{SnL}$  complex ( $R_q = 26.18$ ) as illuminated in Table 1. The synthesized three complexes demonstrated significant stability of PVC hence the  $R_q$  was much reduced in comparison to the blank PVC. However  $\text{Ph}_3\text{SnL}$  complex showed the best results, which is expected due to the presence of three phenyl groups that absorb UV light and prevent the photo-degradation of PVC chains.

### 3.3.3. Scanning electron microscope

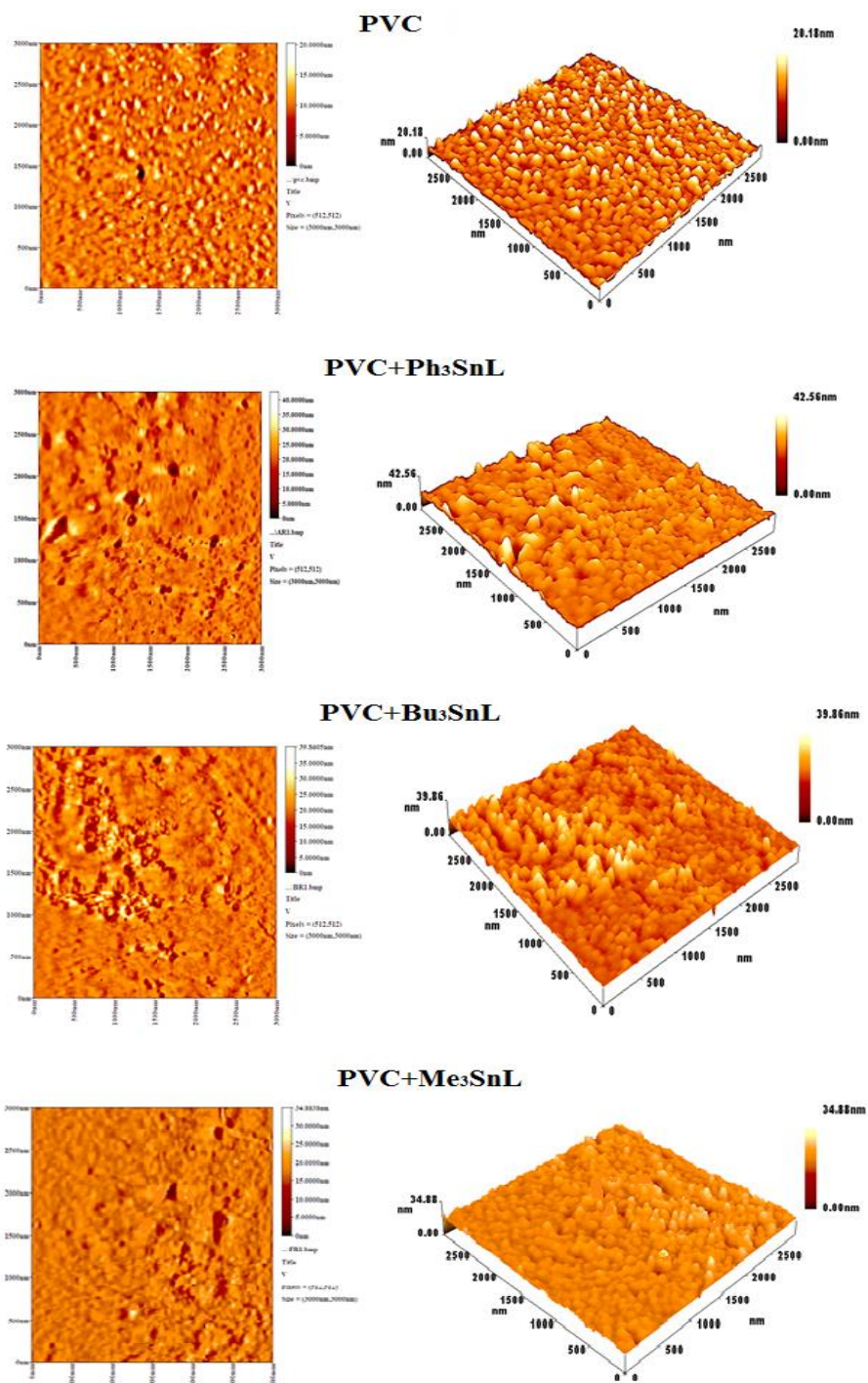
The non-irradiated PVC films' SEM pictures revealed a smooth, clean surface with distinct grain boundaries and good particle homogeneity. Figure 9 shows that the PVC surface was damaged after 300 hours of irradiation. The damage was more obvious on the blank PVC than it was on the PVC with organotin additions. Additionally, the fissures were deeper and longer than they were in the non-radiated material. Such fractures may develop as a result of HCl and other volatile degradation chemicals' chain crosslinking and evaluation [29]. The PVC film with  $\text{Ph}_3\text{SnL}$  complex was clearly the one with the least surface damage.

**Table 1:** PVC roughness factor ( $R_q$ ) following irradiation.

PVC Film	$R_q$
Blank PVC before irradiation	1.08
PVC	216.95
PVC+ $\text{Ph}_3\text{SnL}$	26.18
PVC+ $\text{Bu}_3\text{SnL}$	63.40
PVC+ $\text{Me}_3\text{SnL}$	76.18



**Figure 7:** Microscope images of PVC and (PVC+Complexes) films after 300 h irradiation.



**Figure 8:** Atomic force microscopy images exhibit the effect of irradiation on PVC (pure) and (PVC + complexes).



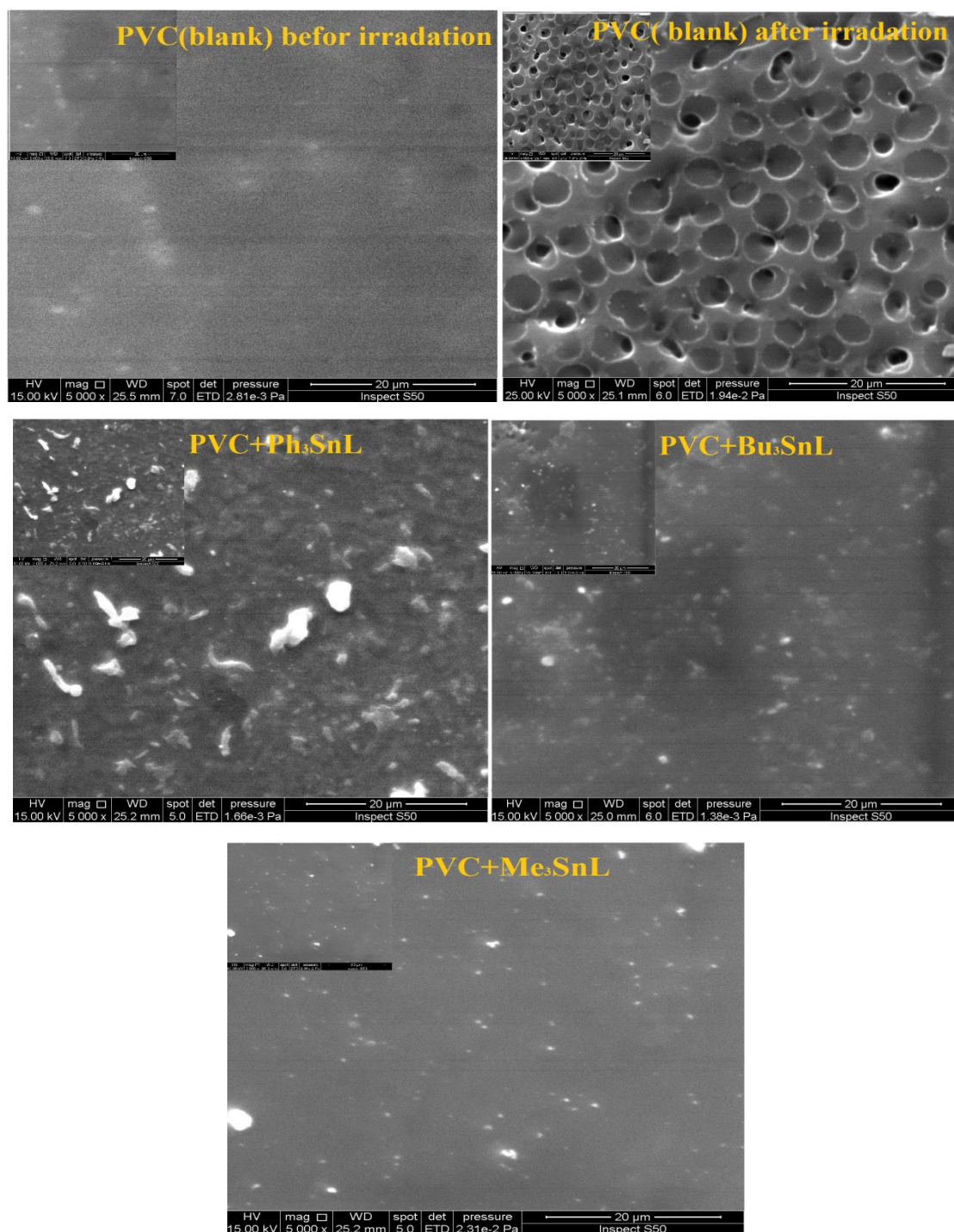
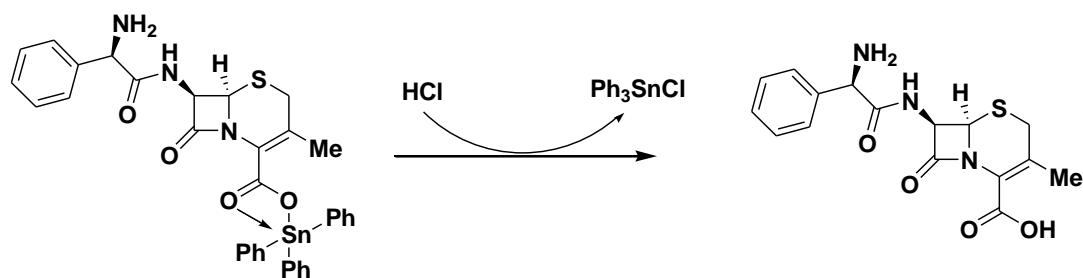


Figure 9: SEM images of PVC films.

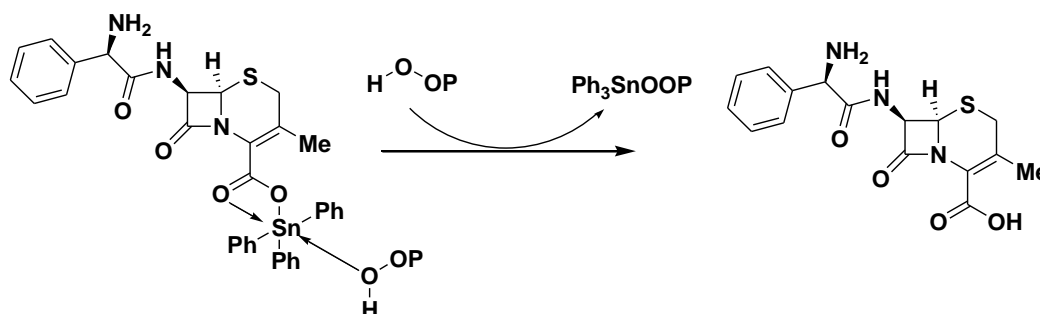
### 3.4. Mechanisms of photo stabilization of PVC by triorganotin (IV)-cephalexin complexes

Compounds **1-3** decreased the amount of defect produced by photoirradiation to PVC and are consequently classified as PVC photo-stabilizers. The complex  $\text{Ph}_3\text{SnL}$  is the best because it includes three phenyl groups in addition to the aryl rings inside the

cephalexin structure. Several approaches have been developed to characterize the activity of triorganotin (IV) complexes as photo stabilizers against PVC film photodegradation. PVC is dehydrochlorination after being exposed to UV light, because the tin atom is a Lewis acid, it can scavenge the hydrogen chloride that has been created, this action would decrease the



Scheme 1:  $\text{Ph}_3\text{SnL}$  complex as hydrogen chloride scavengers.



Scheme 2:  $\text{Ph}_3\text{SnL}$  complex as peroxide decomposers.

harmful effects of HCl on the polymeric chains Scheme 1. Photo-oxidation of PVC forms carbon radicals, that react with oxygen to form peroxide radicals [30]. As a result, complexes are expected to degrade peroxides (such as hydroperoxides) and inhibit PVC photodegradation (Scheme 2).

#### 4. Conclusion

This research explains the production of appropriate quantities of new tin complexes containing cephalexin for photo stabilizers to limit the photodegradation of PVC films. Analytic and spectral techniques have been used to determine the structures of the tin (IV)-cephalexin complexes. Irradiation of PVC thin films causes changes throughout the infrared spectra because of the formation of tiny fragments carrying functional groups, material weight loss, and photodegradation-induced surface defects in the PVC. Such variations

were less distinct in the presence of tin complexes than in the blank PVC film. This additive decreased photodegradation and decreased the loss of weight of PVC film. The tri methyl tin complex also decreased the roughness factor of the irradiated materials by 2.18 the additions worked as UV absorption, chlorine scavengers, and hydroperoxide decomposers. To sum up, following irradiation, it was found that the poly(vinyl chloride) morphology was less harmed in the films containing additives. This was demonstrated by the microscope, atomic force microscopy, and scanning electron microscope. The UV absorbers, hydrogen chloride scavengers, and peroxide quenchers in these additives are all highly effective.

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