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RESEARCH ARTICLE

Aging effects on polystyrene thin films containing high aromatic moiety compounds under UV irradiation

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Abstract

Objectives. Exposure to ultraviolet (UV) radiation initiates a photo-oxidative degradation process in plastics that causes broken chemical bonds, weight loss, and weakened crosslinking, as well as the formation of free radicals and release of volatile substances. The work aims to study the influence of UV radiation on polystyrene films containing low concentrations of highly aromatic Schiff bases.

Methods. Utilizing the described casting technique, polystyrene films containing a small quantity of strong highly aromatic Schiff bases were produced. These films were then subjected to 300 h of UV irradiation. Following irradiation, analyses were conducted on the infrared spectra, weight loss, and surface morphology of the polystyrene.

Results. The surface of the sheets was initially smooth and free of wrinkles. However, following irradiation, remarkable morphological changes were observed as the surface became more uneven. Intentional perforations and imperfections introduced into the examined surfaces were used to indicate the degradation of the polymer matrices.

Conclusions. Highly aromatic Schiff bases act as photostabilizers to significantly reduce the extent of photodegradation in polystyrene films when compared to films with no additives.

Keywords

polystyrene films, carbonyl group index, photooxidation, photodegradation, surface roughness, Schiff's foundations

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НАУЧНАЯ СТАТЬЯ

Влияние ультрафиолетового облучения на старение тонких пленок полистирола с высоким содержанием ароматических компонентов

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Аннотация

Цели. Воздействие ультрафиолетового (УФ) излучения инициирует процесс фотоокислительной деградации пластмасс, сопровождающийся разрушением химических связей, выделением летучих веществ, как следствие, потерей массы, а также образованием свободных радикалов. Цель работы — установление влияния УФ-излучения на полистирольные пленки, содержащие низкие концентрации оснований Шиффа с расширенной цепью сопряжений.

Методы. С использованием технологии литья получены пленки из полистирола, содержащие небольшое количество оснований Шиффа с расширенной цепью сопряжений. Прослежено влияние УФ-облучения полученных пленок (время воздействия — 300 ч) на их строение, изученное методом инфракрасной спектроскопии, массу и морфологию поверхности.

Результаты. Изначально гладкая и не содержащая дефектов поверхности полистирольная пленка после облучения претерпевала значительные морфологические изменения и становилась неровной. Сделан вывод о разрушении полимерной матрицы.

Выводы. Показано, что основания Шиффа с расширенной цепью сопряжений действуют как фотостабилизаторы, обеспечивающие уменьшение степени фотодеградации пленок из полистирола по сравнению с системами, не содержащими добавок этих соединений.

Ключевые слова

пленки из полистирола, индекс карбонильной группы, фотоокисление, фотодеградация, шероховатость поверхности, основания Шиффа

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1. INTRODUCTION

Polystyrene (PS), with a molecular weight of 100000 to 400000 is one of the most commonly used thermoplastic polymers [1]. It is derived from styrene polymerization to form either foam or solid final structures. One of its primary benefits lies in its cost-effectiveness due to being based on inexpensive feedstock materials. Furthermore, its low weight and chemical inertness make it impervious to interact with other chemicals, including acids and bases [2]. As documented in previous reports, PS is adaptable to various deposition types [3]. However, it is also non-biodegradable, flammable, and soluble in various chlorinated solvents. Despite these drawbacks, PS has been extensively applied in many commercial products, from simple packaging to intricate engineering tasks [4]. The arrangement of phenyl groups along the polymer chain influences PS's crystallinity [5]. Due to random phenyl group positioning, commercially valuable atactic PS is amorphous, while syndiotactic PS is crystalline with phenyl groups formed alternately on both sides of the polymer chain [1].

The main environmental variables affecting the durability of polymers used in outdoor settings are solar irradiation, weather patterns, temperature, humidity, and pollutants. When these factors are compared, ultraviolet (UV) radiation is generally considered to be the most significant [6]. Due to high oxygen levels and the UV sensitivity of microplastics (MPs), multiple aging processes occur upon their discharge to landfills or the environment. Some of the steps in these processes include direct exposure to UV radiation from the sun, thermo-oxidative breakdown, physical stress, and biodegradation. The high commercial value of atactic PS is due to its amorphous structure, which derives from the randomly positioned phenyl groups. In contrast, syndiotactic PS has an alternating arrangement of phenyl groups along the polymer chain, resulting in a crystalline structure [1].

As a result of aging processes, cracks and fragmentation are developed within plastic polymers [7, 8]. MPs constitute tiny fragments of synthetic polymers discovered in freshwater, marine ecosystems, sediment, and within living organisms. These particles significantly threaten aquatic environments and organisms, causing ecological harm through ecotoxicological pathways [9].

By altering the surface properties of plastic debris, the above-described processes escalate the release of intermediary chemical additives including pigments, antioxidants, flame retardants, stabilizers, and plasticizers. As a consequence of their discharged from MPs, such additives introduce harmful chemicals into marine ecosystems [10]. Moreover, due to their diminutive size, high hydrophobicity, rugged particle surfaces, and enhanced mobility, MPs can absorb various organic pollutants in aquatic environments. Consequently, there has been a surge in interest in investigating the potential ecotoxicological ramifications of MPs as carriers of pollutants [11]. The present work sets out to demonstrate the efficiency of adding highly aromatic Schiff bases at low concentrations as photostabilizers as a means of mitigating the photodegradation of PS films under extended UV irradiation. This provides another means to enhance the photostability of polymeric materials in addition to those presented previously [12–14]. The described Schiff bases, which function as UV absorbers and stabilizers, can be incorporated into PS to improve its resistance to photodegradation. In order to prevent PS photodecomposition, such stable compounds are required to have a high concentration of heteroatoms and aromatic moieties such as phenyl and heterocycles.

In the present study, we demonstrate the effective use of highly aromatic Schiff bases at low concentrations as photostabilizers to significantly reduce the photodegradation of PS films under 300 h UV exposure. This work contributes to ongoing research on the photostabilization of polymeric materials.

2. MATERIALS AND METHODS

2.1. Chemicals and devices

The PS used in this study was obtained from *Sigma-Aldrich* chemical company (*Gillingham*, United Kingdom). Fourier transform infrared (FTIR) spectra spanning the range of 4000–400 cm^{-1} were acquired using a FT/IR-4200 spectrometer (*Jasco*, Tokyo, Japan). The PS films underwent irradiation at a temperature of 25°C, a maximum wavelength of 365 nm, and an intensity of $6.43 \cdot 10^{-9}$ $\text{ein/dm}^3\cdot\text{s}$. This irradiation process was conducted using a QUV tester, an accelerated weather meter sourced from the *Q-Panel* company (*Homestead*, FL, USA). The surface of the PS films was analyzed using a *Veeco Instrument* atomic force microscope (AFM) (*Plainview*, NY, USA). Scanning electron microscopy (SEM) was also employed to examine the PS surface. This process, which utilized an Inspect S50 microscope from the *FEI Company* (*Czechia*, Czech Republic), was operated at an accelerating voltage of 15 kV. Microscopic images of the PS surface were captured using a *Meiji Techno* microscope (Tokyo, Japan). The thickness of the PS films was measured to be approximately 40 µm using a Digital Vernier Caliper 2610 A micrometer from *Vogel GmbH* (*Kevelaer*, Germany). The films were supported using aluminum plate stands with a thickness of 0.6 mm from *Q-Panel* company (*Homestead*, FL, USA).

2.2. Synthesis of the compounds

2.2.1. Synthesis of compound **1**

A solution consisting of 5 mL of hydrazine hydrate (*Merck*, Germany) and 10 mL of 0.079 mol methyl benzoate *(Merck*, Germany) was refluxed for 4 h. Following this, 8 mL of ethanol was added, and the mixture was refluxed for an additional hour. After cooling, the product was separated via filtration and left to dry in readiness for the next step [15].

2.2.2. Synthesis of compound **2**

3.4 g of benzohydrazide (0.024 mol, *Merck*, Germany) was dissolved in 30 mL of absolute ethanol (*Alfa Chemical Co*., China) along with 2 g potassium hydroxide (0.035 mol, *Merck*, Germany) and stirred for 15 min. Following this, 2 mL of a 0.033 mol carbon disulfide solution (*Fisher Scientific*, MA, USA) was added and the mixture was continuously stirred for 18 h until it became homogeneous. Subsequently, 10 mL of hydrazine hydrate (*Merck*, Germany) was added to the potassium xanthate (*Merck*, Germany) product. The resulting mixture underwent reflux with stirring, resulting in a homogeneous solution that is associated with the release of hydrogen sulfide gas. The reaction was stopped when the release of hydrogen sulfide ceased (as indicated by the absence of a reaction with lead acetate paper). After filtration, the reaction mixture was diluted with 10 mL of cold water and acidified using concentrated hydrochloric acid (*Alfa Chemical Co*., China), resulting in the formation of a white precipitate [15].

2.2.3. Synthesis of compound **3**

The compound **2** (2 mol, 1 g) was combined with terephthaldehyde (1 mol, 0.3 g, *Merck*, Germany) in 10 mL of dimethylformamide (DMF, *Alfa Chemical Co*., China), along with two drops of glacial acetic acid (*Alfa Chemical Co*., China). The mixture was then refluxed for 7 h. Following this, the solution was cooled to prompt the precipitation of the product, which was then washed with ethanol [15].

2.2.4. Synthesis of compounds **4** and **5**

0.5 g of sulfamethoxazole (*Merck*, Germany, to prepare compound **4**) or methyldopa (*Merck*, Germany) to prepare compound **5**) along with 0.2 mL triethylamine (0.001 mol) were dissolved in 10 mL of DMF in two 50 mL round bottom flasks each fitted with a magnetic stirring bar. The flask was cooled to 0°C for 30 min while stirring, and then 0.1 mL of 0.001 mol chloroacetyl chloride was slowly added in small portions. The mixture was stirred overnight at room temperature (25°C). Following reaction completion, the mixture was transferred into ice water, filtered, and the product was washed with water. The yields were purified using ether [16].

2.2.5. Reaction of compound **3** with compounds **4** and **5**

Compound **3** was dissolved in 10 mL of DMF in a round bottom flask utilizing 1 mol (0.1 g) of the compound. Following complete dissolution, 2 mol (0.05 g) of K_2CO_3 was added. Subsequently, compounds **4** and **5** were added in a total amount of 4 mol (0.2 g). The mixture was stirred for an extended period and then transferred into a container of ice water. Following this, the mixture was filtered and the resulting solid was allowed to desiccate in readiness for the subsequent reaction [17]. By this means, compounds **6** and **7** were prepared.

2.3. Preparation of PS films

5 gram of PS was stirred in 100 mL of chloroform (*Merck*, Germany) for 90 min at 25°C. Compounds **6** and **7**, weighing 25 mg and constituting 0.5% of the total weight, were added to the PS solution. The resulting mixture was stirred for 30 min at 25°C. The homogeneous mixture was then poured onto clean glass slides to a thickness of approximately 40 µm and allowed to dry for 24 h at 25°C.

2.4. Accelerated UV weathering

The films were subjected to 300 h of accelerated UV weathering testing using the QUV tester from *Q-Panel* company (*Homestead*, FL, USA). During each weathering cycle, the films were continuously exposed to UV radiation having a maximum wavelength of 365 nm and intensity of $6.43 \cdot 10^{-9}$ ein/dm^3·s. Testing intervals were set at 0, 50, 100, 150, 200, 250, and 300 h of exposure at a temperature of 25°C. After each duration of exposure, every group underwent extraction for measurements.

2.5. Monitoring UV degradation by FTIR spectrophotometry of PS films

Under photolytic conditions, PS degradation occurs, resulting in the generation of small polymer fragments containing carbonyl (C=O) groups. The FTIR spectra of the irradiated PS films were measured at different irradiation durations. The functional group index ($I_{\mathrm{C=O}}$) was determined by calculating the absorbance of the functional group ($A_{\mathrm{C=O}}$) and the reference peak (A_{r}) using Eq. (1) [18, 19].

$$I_{\mathrm{C=O}} = \frac{A_{\mathrm{C=O}}}{A_{\mathrm{r}}}. \quad (1)$$

2.6. Weight loss of PS films

The sheets were weighed before irradiation (W_0) and after irradiation at different time intervals (W_t). The estimation of the polymer weight loss percentage due to irradiation was calculated using Eq. (2) [19].

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

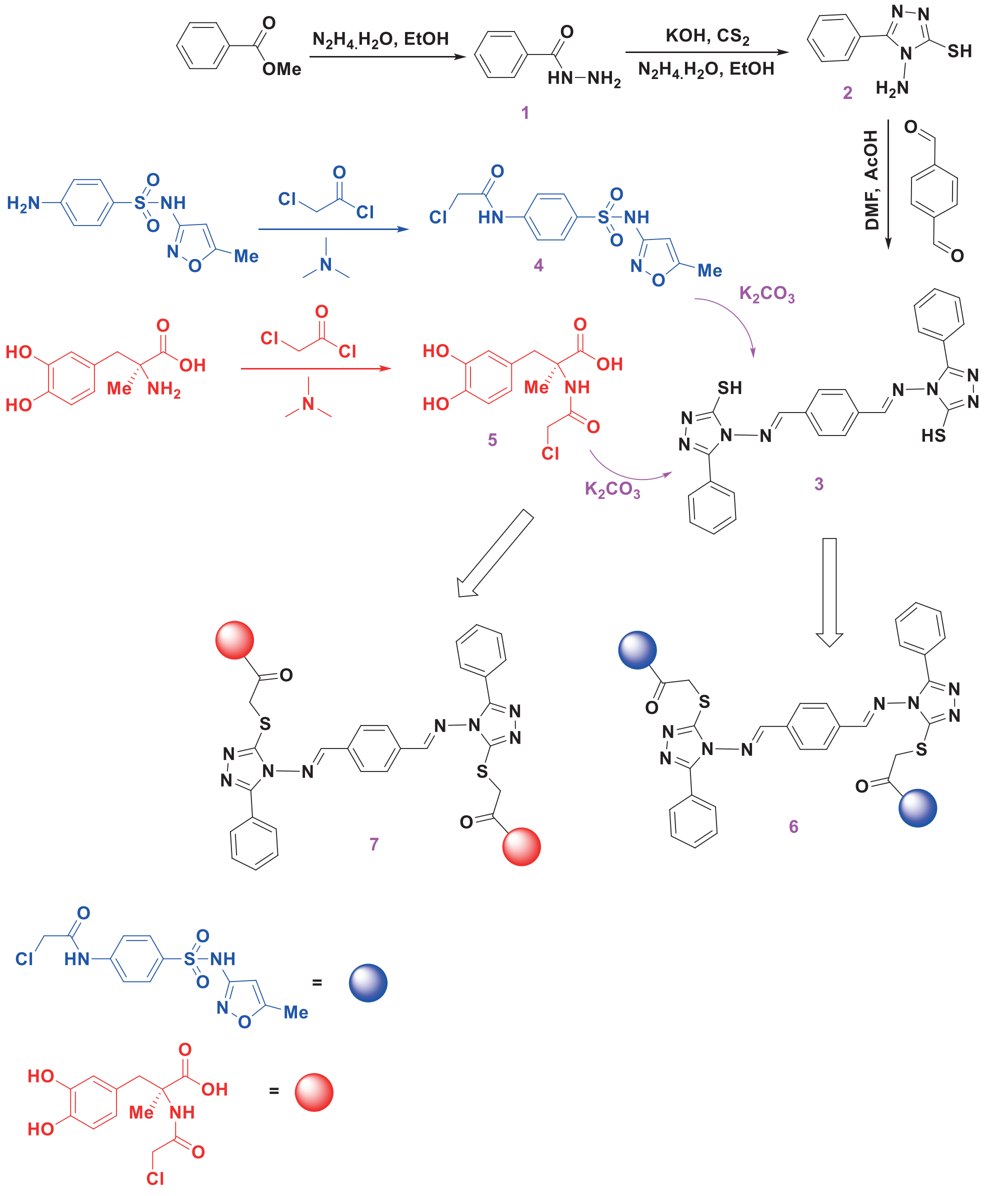
3. RESULTS AND DISCUSSION

This synthesis process yielded compounds **1**–**7**, which were utilized in the fabrication of PS films. The compounds were obtained with yields ranging from 40% to 96% (Scheme). Table 1 presents the physical characteristics of compounds **1** to **7**.

Table 1. Physical properties of compounds **1**–**7**

No.	*M*, wt %	Yield, %	Color	Melting point, °C
1	136.15	50	White	112–114
2	192.24	96	White	200–202
3	482.58	61	Orange	300
4	329.50	40	Orange	110–112
5	287.50	70	Brown	250
6	1069.18	50	Yellow	250
7	985.06	65	Brown	130–131

Compound **1** was synthesized by reacting salt with hydrazine hydrate. The synthesis was confirmed using an FTIR device, which detected a peak at



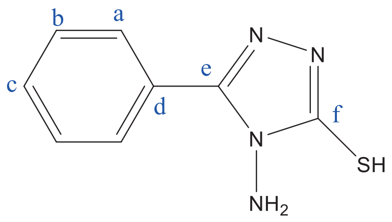
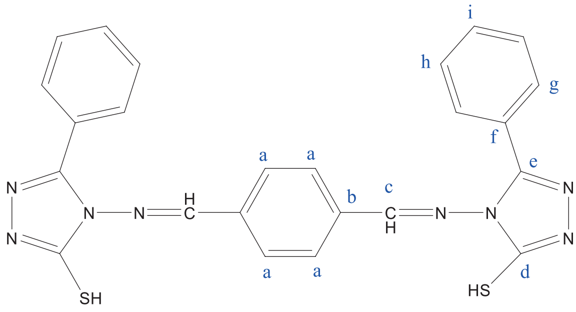
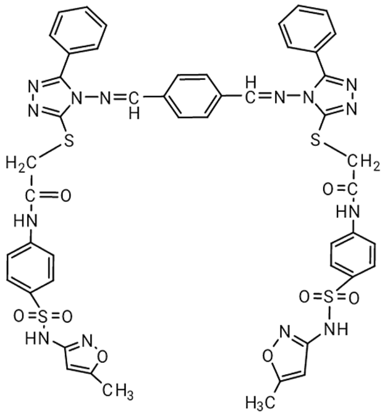
Scheme. Synthesis of compounds **1**–**7**

1632 cm^{-1} corresponding to the C=N group of the triazole ring. Two distinct stretching vibration bands additionally observed at 3294 and 3189 cm^{-1} were attributed to the NH_2 group. The FTIR spectra of the prepared Schiff base indicated the disappearance of the stretching band of the primary amine NH_2 at 3294 and 3189 cm^{-1}. In contrast, a stretching band at 1657 cm^{-1}, associated with the creation of the imine group HC=N, appeared. Table 2 shows the FTIR analysis for compounds **2**–**7**, and Fig. 1 shows FTIR spectra for compounds **2**–**7**. Furthermore, Table 3 and Figs. 2 and 3 present the ^{1}H nuclear magnetic resonance (NMR) and ^{13}C NMR characteristic spectra, respectively, for compounds **2**, **3**, and **6**.

Table 2. FTIR characteristic spectra for compounds **2**–**7**

No.	Frequency numbers, cm^{-1}
2	3294 and 3189 cm^{-1} for asymmetric and symmetric [NH_2], 3002 cm^{-1} for [C–H] aromatic, 2663 cm^{-1} for [S–H], 1632 cm^{-1} for [C=N], and 662 cm^{-1} for [C–S]
3	3067 cm^{-1} for [C–H] aromatic, 2932 cm^{-1} for [C–H] aliphatic, 2752 cm^{-1} for [S–H], 1657 cm^{-1} for [C=N], and 688 cm^{-1} for [C–S]
4	3040 cm^{-1} for [C–H] aromatic, 1620 cm^{-1} for [C=O] of amide, 3332 cm^{-1} for [NH], 2982 and 2930 cm^{-1} for [C–H] aliphatic, and 715 cm^{-1} for [C–Cl]
5	1691 cm^{-1} for [C=O] of amide, 3050 cm^{-1} for [C–H] aromatic, 2983 cm^{-1} [C–H] aliphatic, 1655 cm^{-1} for [C=N], and 763 cm^{-1} for [C–Cl]
6	1612 cm^{-1} for [C=N] of Schiff base, 1518 cm^{-1} for [C=N] of triazole ring, 1661 cm^{-1} for [C=O] of amide, 1149 cm^{-1} for symmetric [S=O], 1302 cm^{-1} for asymmetric [S=O], 3058 cm^{-1} for [C–H] aromatic, 2965 and 2931 cm^{-1} [C–H] aliphatic
7	1613 cm^{-1} for [C=N] of Schiff base, 1502 cm^{-1} for [C=N] of triazole ring, 1663 cm^{-1} for [C=O] of amide, 3051 cm^{-1} for [C–H] aromatic, 2991 and 2931 cm^{-1} [C–H] aliphatic

Table 3. ^{1}H NMR and ^{13}C NMR characteristic spectra for compounds [**2**, **3**, and **6**]

No.	Structure	Chemical shift, ppm
2		^{1}H NMR shows: δ = (7.5 ppm, 8.05 ppm, m) for protons at the aromatic ring, (5.9 ppm, s) for NH_2, and (10 ppm, s) for SH ^{13}C NMR shows: δ = (166.8 ppm) for carbon at f, (149.4 ppm) at e, and (128, 128.4, 128.9, and 130.4 ppm) for carbons at a, b, c, and d
3		^{1}H NMR shows: δ = (8.02 ppm, s) for protons at aromatic ring that bonded to C=N, (8.3 ppm, s) for HC=N, (14.4 ppm, s) for S–H, and (7.65, 7.66, and 8.06 ppm, m) for protons at aromatic ring bonded to 1,2,4-triazole ^{13}C NMR shows: δ = (162 ppm) for carbon at c, (165 ppm) for carbon at d, (148.7 ppm) for carbon at e, and (129.3, 135.5, 130.7, 128.4, and 129.2 ppm) for carbons at aromatic rings (a, b, f, g, h)
6		^{1}H NMR shows: δ = (8.01 ppm, s) for protons at aromatic ring bonded to C=N, (8.3 ppm, s) for <u>H</u>C=N, (4.04 ppm, s) for C$\underline{H}_2$, (2.3 ppm, s) for C$\underline{H}_3$, and (7.5–8.1 ppm, m) for protons at aromatic rings

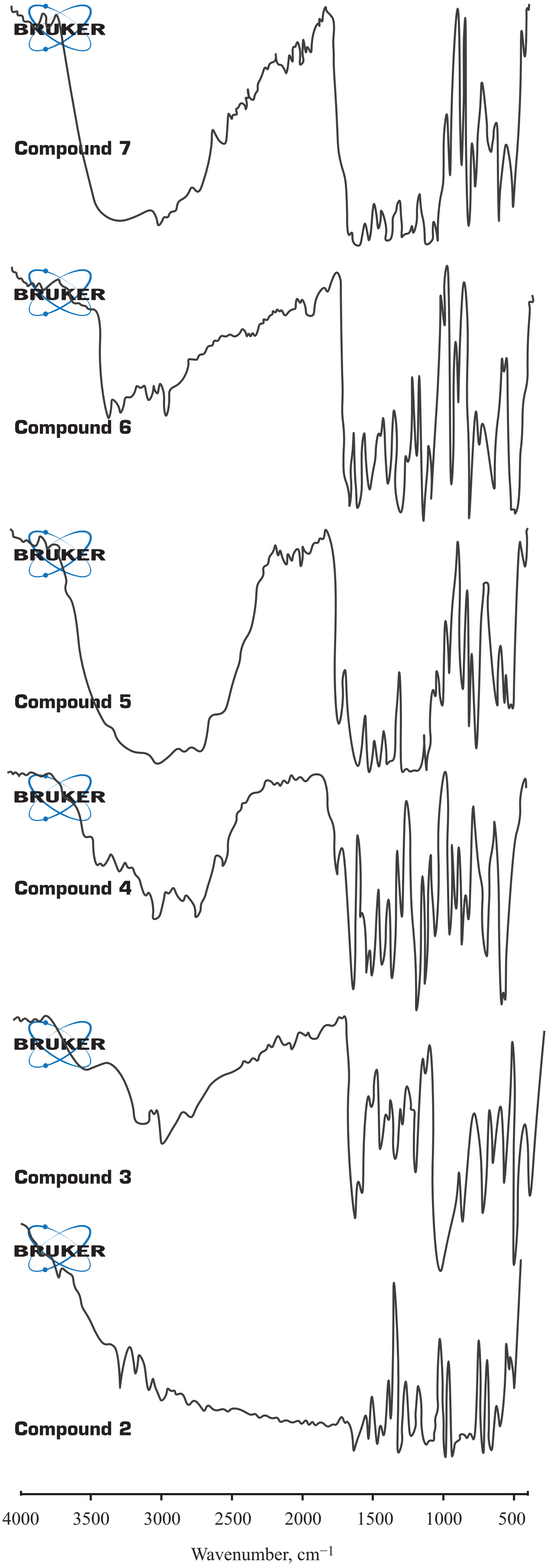
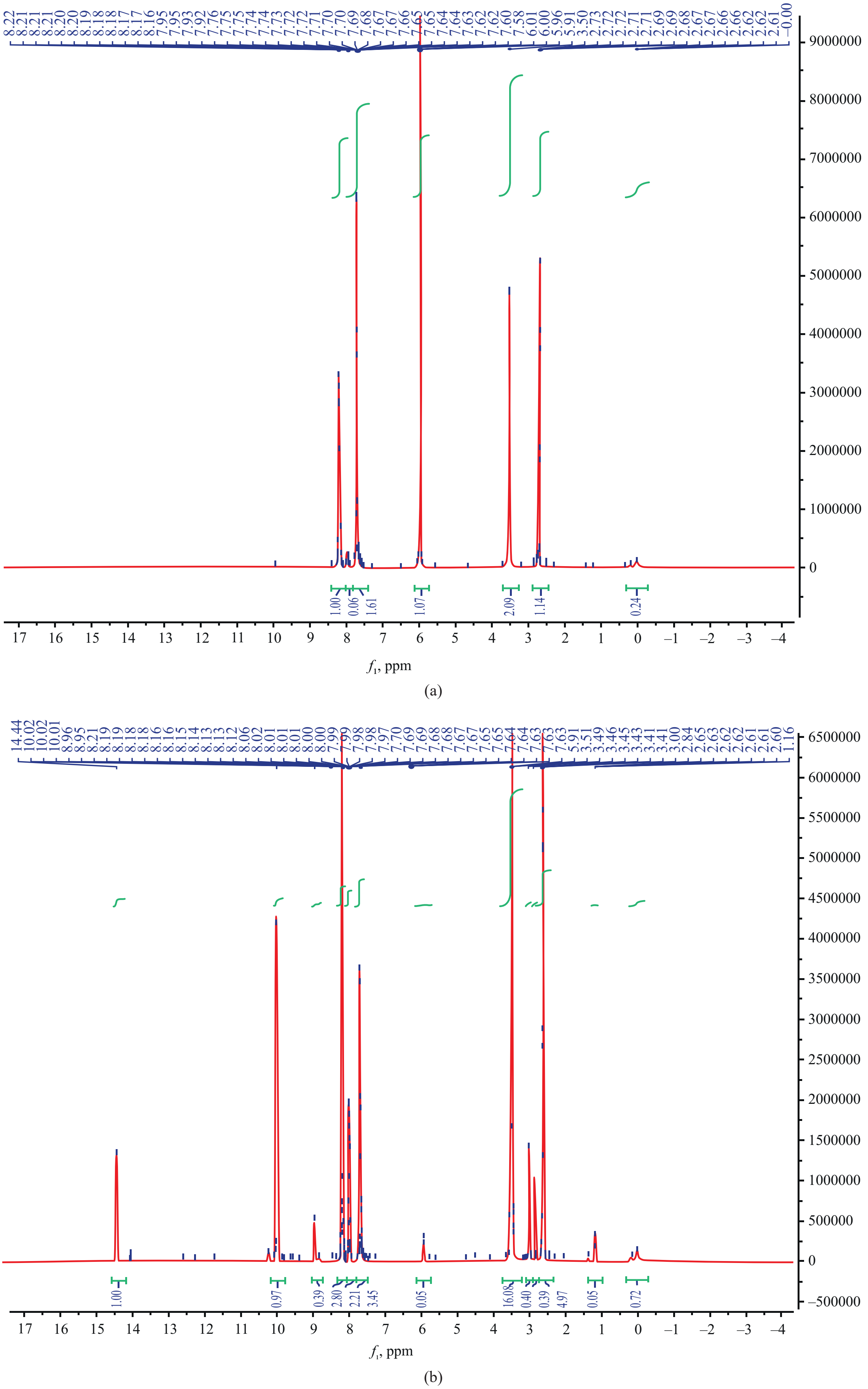


Fig. 1. FTIR for compounds **2**–**7**



(a)

(b)

Fig. 2. ^{1}H NMR for (a) compound **2** and (b) compound **3**

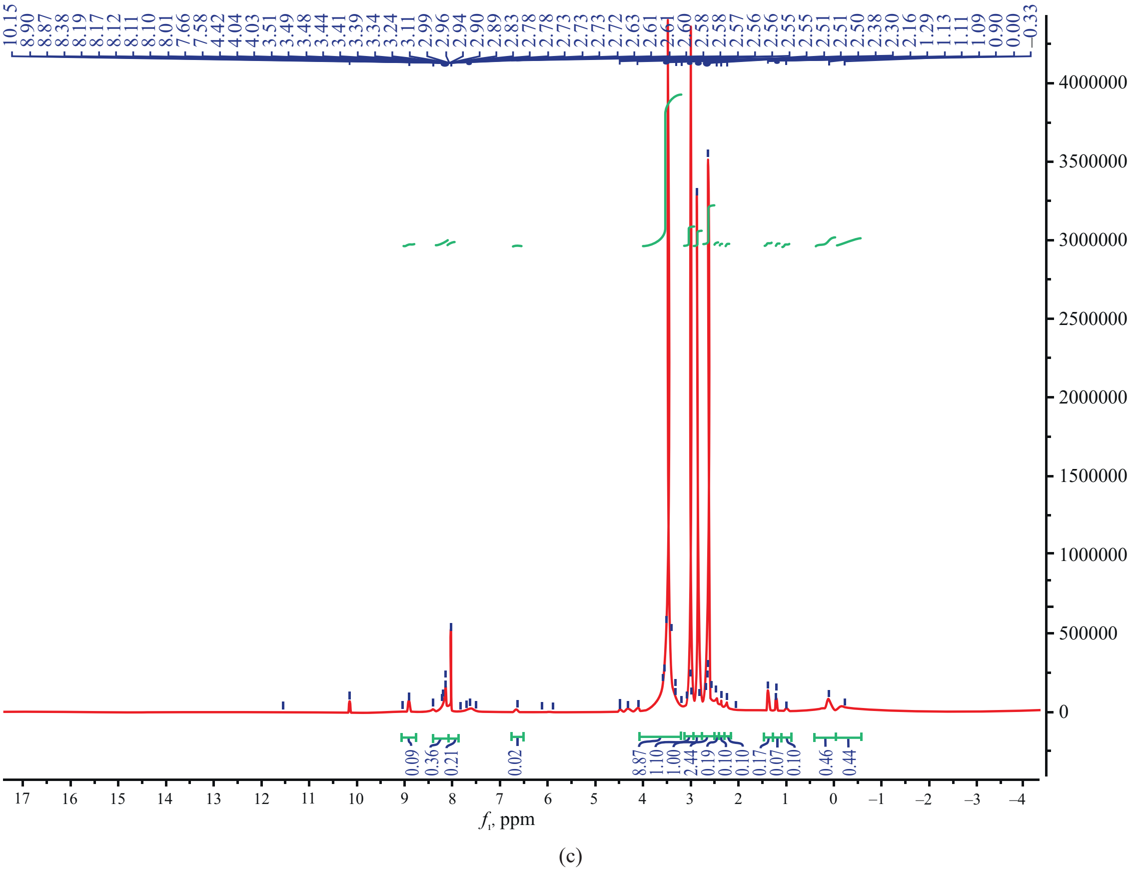


Fig. 2. Continued. ^{1}H NMR for (c) compound **6**

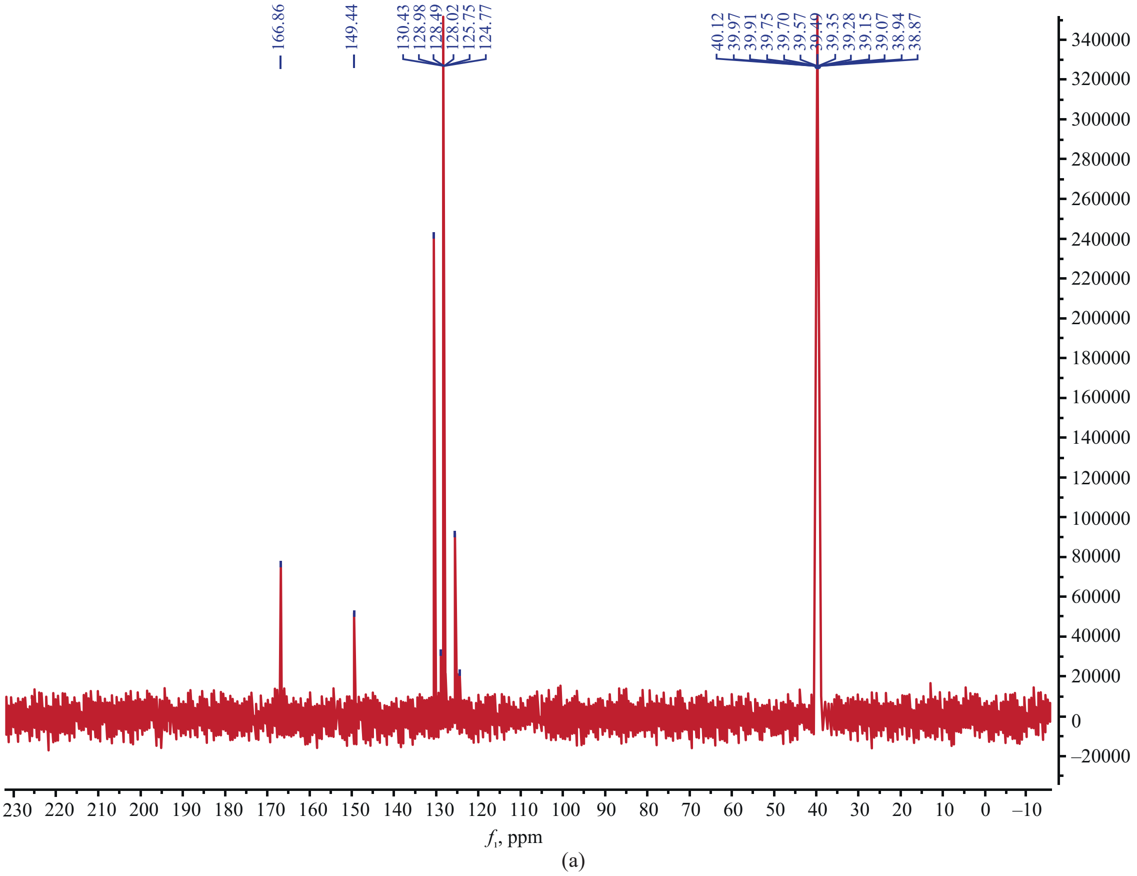
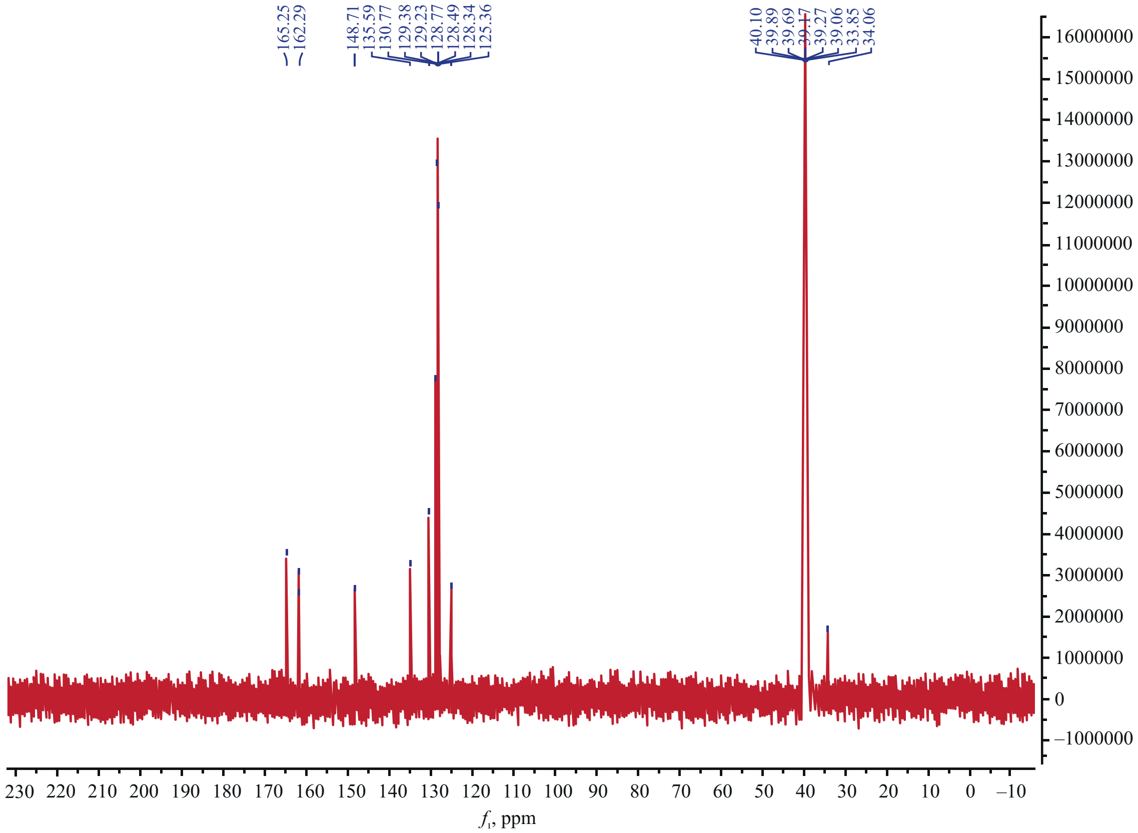


Fig. 3. ^{13}C NMR for (a) compound **2**



(b)

Fig. 3. Continued. ^{13}C NMR for (b) compound **3**

3.1. Chemical structure changes

The aging of polymer materials entails irreversible alterations in their characteristics due to exposure to light, heat, ambient air, oxygen, and sun rays. A heightened overall impact of weathering occurs due to the combination of multiple agents in aging processes. Photodegradation of polymers involves both crosslinking and chain scission reactions to significantly alter their physical properties. FTIR spectroscopy has been successfully employed to analyze the chemical changes occurring during the UV irradiation period as a means of understanding photooxidation processes [20]. The carbonyl group index ($I_{\mathrm{C=O}}$) was determined using Eq. (1). The intensity of the signal corresponding to the absorption of the C=O group at 1720 cm^{-1} was significantly higher following irradiation as compared to before. Figure 4 illustrates the variations observed in $I_{\mathrm{C=O}}$ as the irradiation time ranged from 0 to 300 h. In contrast with the blank PS film, films containing compounds **6** and **7** (0.5 wt %) exhibited reduced carbonyl group indices after irradiation. The results demonstrated the successful utilization of these compounds, particularly compound **6**, to enhance the photostability of the PS films.

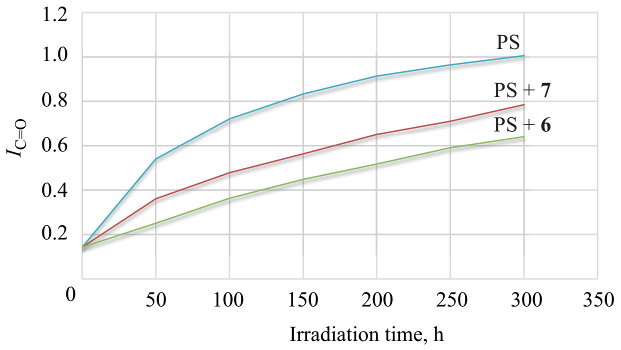


Fig. 4. Carbonyl index during UV exposure

3.2. Weight loss of PS films

When subjected to high temperatures and prolonged UV exposure, polystyrene undergoes a rapid color change from colorless to yellow and gradually becomes more brittle. This results in the formation of tiny fragments and a reduction in overall polymer weight [21]. The weight loss of PS films exposed to UV irradiation for a maximum of 300 h was determined using Eq. (2). Figure 5 illustrates the changes in the weight of the PS material after exposure to radiation for 300 h. The polymer experienced significantly greater weight loss in the blank PS film compared to the PS films containing compounds **6** and **7**. Compound **6** exhibited the lowest weight loss.

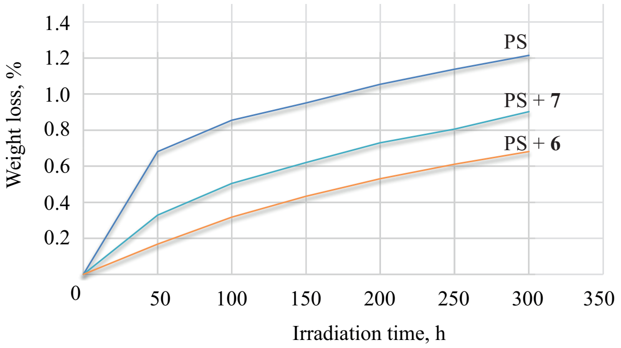


Fig. 5. Weight loss percent during UV exposure

3.3. Morphology analysis

An SEM study was conducted on the surface of PS films to identify any alterations caused by photooxidation in the various samples. The surface of the films was initially smooth and devoid of wrinkles. However, significant morphological changes were observed following irradiation as the surface of the films became more uneven [22]. Intentional perforations and imperfections were additionally incorporated into the surfaces under investigation to reveal the degradation of the polymer matrices. Typically, the evaluated specimens grew more fragile after being exposed to 300 h of irradiation (Fig. 6).

AFM is a commonly used method for studying the surface structure of polymeric materials [23, 24]. Figure 7 shows the AFM images of the PS film

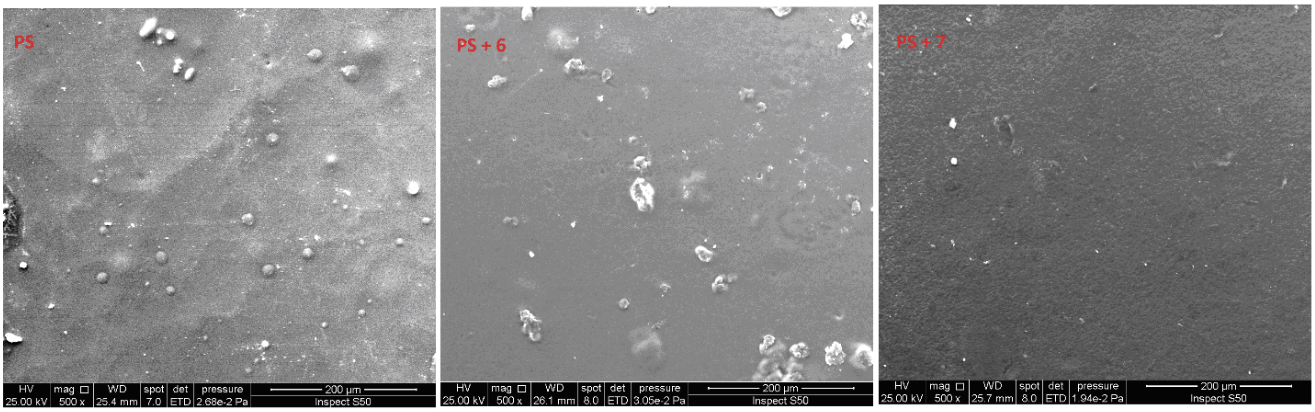


Fig. 6. SEM micrographs of PS films after 300 h of UV exposure

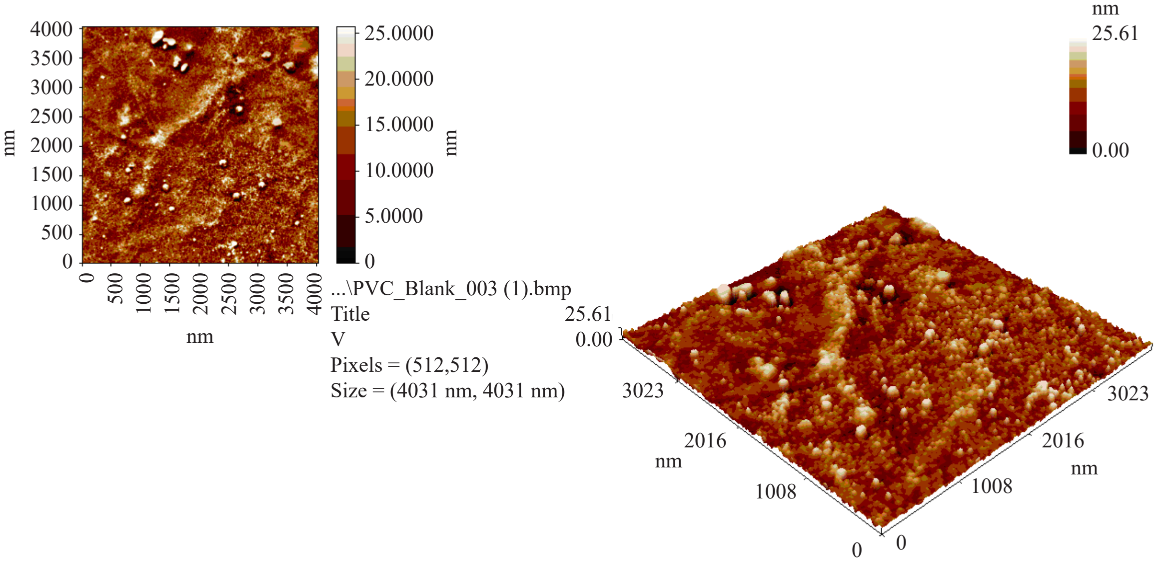


Fig. 7. AFM images for PS blank film after 300 h of UV exposure

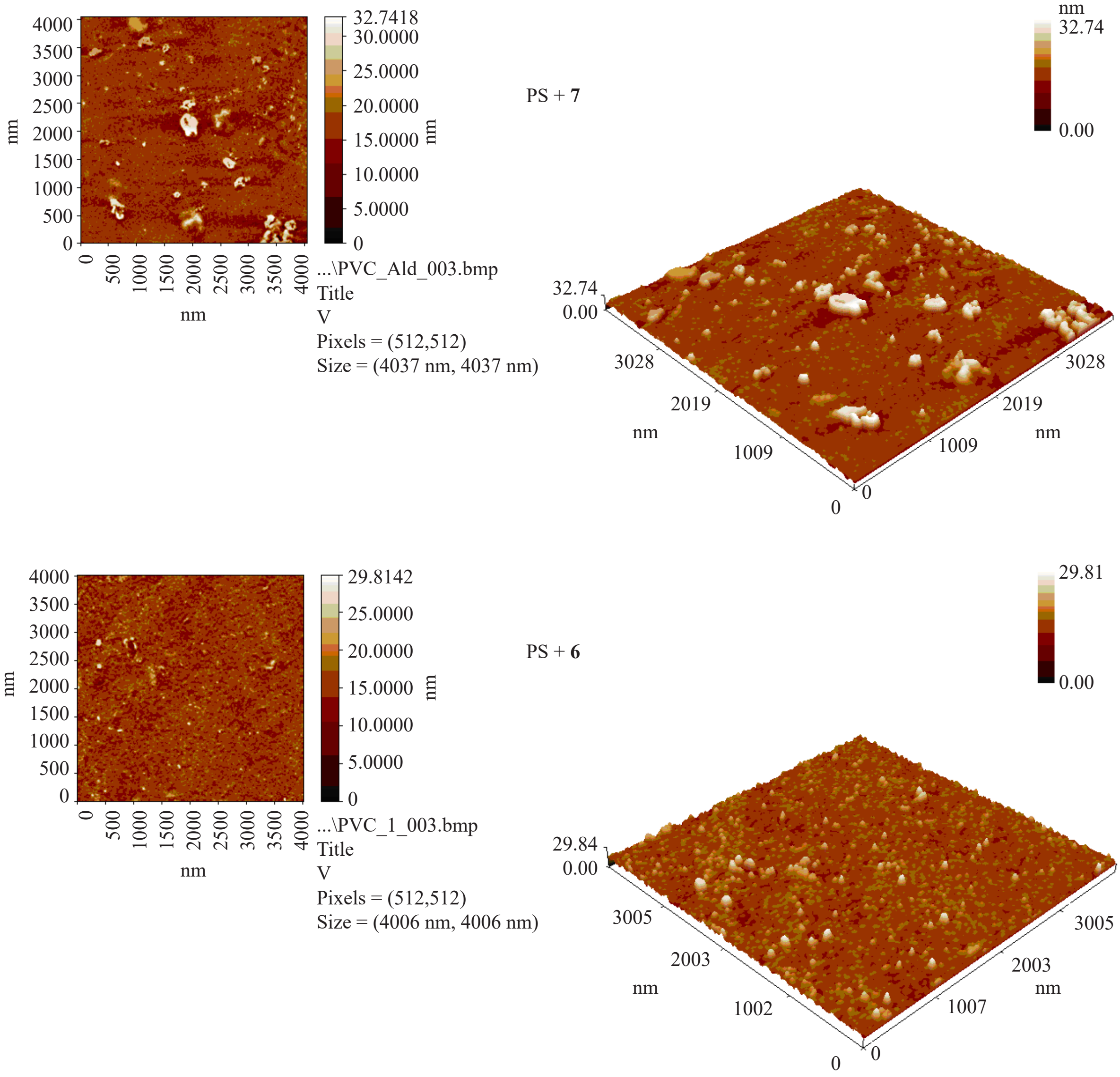


Fig. 8. AFM images for PS films after 300 h of UV exposure

without any compounds, while the PS film with compounds **6** and **7** after exposure to radiation for 300 h are depicted in Fig. 8. The presence of black specks can be attributed to residue originating from the PS matrix. The surface of the PS film containing compound **6** after irradiation exhibited a smooth texture.

4. CONCLUSIONS

This study investigated the effects of aging on PS thin films containing highly aromatic Schiff bases under UV irradiation. The analysis of the performance using FTIR spectroscopy, weight loss measurements, and morphology testing revealed several key findings. Photostabilization by the synthesized aromatic compounds, especially components **6** and **7**, reduced PS film photodegradation after 300 h of UV exposure. FTIR spectroscopy revealed a decrease in carbonyl group indices in films containing these compounds, which indicates a reduction in photo-oxidative degradation compared to the blank PS film. Moreover, weight loss measurements corroborated these findings by showing significantly lower weight loss in films with compounds **6** and **7**. In addition to the photostabilization effects, morphological analyses via SEM and AFM showed that films containing the aromatic compounds had smoother surfaces and fewer cracks. These results highlight the potential of highly aromatic Schiff bases for use as photostabilizers to reduce PS film photodegradation and increase UV film durability and longevity.

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