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Enhanced photostability of PVC using benzenesulfonamide derivatives

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Abstract

This study investigated the effect of Schiff bases photostabilizers from benzenesulfonamide on PVC films. Benzenesulfonamide derivatives (1-5) were prepared by reacting 4-aminobenzenesulfonamide with five substituted benzaldehyde, the structural properties of all compounds were confirmed by ¹HNMR. These compounds were incorporated into the composition of polyvinyl chloride (PVC) membranes at a weight concentration of 0.5%. The optical stability of PVC was studied by several techniques including finding the photodegradation constant of PVC as well as weight loss and scanning electron microscopy (SEM) of the polymer films. The photodegradation rate constants (kd) of the modified PVC films, which included these Schiff bases, decreased significantly ($3 \times 10^{-4} \text{ sec}^{-1}$ to $39 \times 10^{-4} \text{ sec}^{-1}$) compared to the control PVC films ($88 \times 10^{-4} \text{ sec}^{-1}$). Benzenesulfonamide derivative 5 showed the strongest photostabilizing activity. Irradiated films containing benzenesulfonamide compounds (1-5) showed a weight loss of 35%. Scanning electron microscopy (SEM) of the additive-treated PVC after 250 h of irradiation showed a heterogeneous surface morphology resembling broken rocks, indicating the structural changes induced by radiation. These results demonstrate the effectiveness of benzenesulfonamide derivatives as photostabilizers for PVC.

Keywords: PVC films, photostabilization, benzenesulfonamide, schiff bases and photodecomposition rate constant

Introduction

Polymers are manufactured annually in vast quantities due to their diverse and significant industrial and medical applications [1-3]. The widespread utilization of synthetic polymers is attributed to their facile and cost-effective production via various convenient manufacturing processes. Approximately 10,000 companies in the United States are involved in the production, processing, and fabrication of polymeric materials [4]. Polyvinyl chloride (PVC) is one of the most widely produced and used polymers worldwide [5], with extensive applications in outdoor environments [6], particularly in construction [7-9]. However, prolonged exposure of PVC to solar radiation and/or high temperatures causes photodegradation [10], which leads to changes in its physical and mechanical properties [11]. Structural defects and impurities within the PVC polymer backbone constitute the primary cause of dehydrochlorination and/or photo-oxidation reactions, culminating in the formation of unsaturated centers [12-14]. Consequently, protecting PVC from harsh environmental conditions is crucial. Photostabilization of PVC can be accomplished through the incorporation of a diverse array of additives, notably including plasticizers [15], aromatic compounds [16, 17], heterocyclic compounds [18], Schiff base complexes [19-21], metal complexes, and inorganic salts. Due to their drug-like properties, sulfonamides remain a subject of interest for structural and pharmaceutical chemists. Benzene sulfonamide derivatives represent a significant class of organic chemical compounds, combining a benzene ring with the sulfonamide functional group (SO_2NH_2) [22-27]. This combination endows them with unique chemical and physical properties, rendering them of considerable importance in diverse fields, ranging from pharmaceutical and agricultural industries to materials science and analytical chemistry [28-30]. Benzene sulfonamide derivatives are widely used as stabilizers in polymers (e.g., PVC, polyurethane, polyolefins) and for protecting biomaterials, dyes, and pharmaceuticals from photodegradation.

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Their unique chemical structure, featuring a sulfonamide group (SO_2NHR) linked to aromatic rings and heteroatoms (N, O, S), provides them with unique chemical and physical properties^[30]. The electronic conjugation between the sulfonamide group and aromatic rings enhances UV absorption and efficient conversion of absorbed energy into heat, minimizing harmful photochemical reactions. These properties make them promising light stabilizers.

Materials and Methods

Chemicals (solvents and reagents) were used as received from Sigma-Aldrich (UK) without further purification. Polyvinylchloride (PVC) with a K value of 67 and a degree of polymerization of 800 was obtained from Petkim Petrokimya (Turkey).

Procedure

Preparation of Polyvinylchloride (PVC) Films

Commercial Polyvinylchloride (PVC) was dissolved in tetrahydrofuran (THF) (5 g/100 mL), reprecipitated with ethanol (EOH), and dried under reduced pressure at 20 ($^{\circ}\text{C}$) for 24 (h). Organotin complexes (0.5 wt/wt %) were then incorporated into the PVC at 20 ($^{\circ}\text{C}$). The resulting mixture was cast onto aluminum (Al) plate stands (Q-Panel-Company, Homestead, FL, USA)^[31] to produce 40 μm thick films. A digital vernier caliper was used to measure (Kevelaer, Germany).

Accelerated Testing Method

PVC films were irradiated using a QUV tester (Germany) following a standard procedure for 250 hours. The light source provided an intensity of 6.43×10^{-9} Einstein ($\text{dm}^{-2}\text{s}^{-1}$) at a wavelength of 365 nm.

Measurement of the photodegradation rate constant (kd) of PVC films by UV-vis spectroscopy.

The UV-visible spectral changes of PVC films under light exposure at $\lambda_{\text{max}} = 365$ (nm) were measured using a Shimadzu UV-VIS 160A Spectrophotometer (Japan). Using equation (1), the photodegradation rate constant (kd) for PVC films was determined.

$$\ln(a-x) = \ln a - k_d t \quad (1)$$

Where,

$$a = A_0 - A_{\infty}, x = A_0 - A_t \quad (2)$$

The variable A represents the initial PVC concentration, while x represents the change in concentration at any time t during irradiation. A_0 denotes the PVC absorption intensity at the beginning of the experiment (t_0), while A_{∞} denotes the absorption intensity at the end of the reaction (t_{∞}). At gives the absorption intensity after an irradiation time of t. Equation (3) is derived by substituting the value of Equation (2) into Equation (1).

$$\ln(A_t - A_{\infty}) = \ln(A_0 - A_{\infty}) - k_d t \quad (3)$$

Analysis of the above correlation yielded a linear relationship, with the slope (k_d) indicating first-order decomposition kinetics for PVC^[32,33].

Determination of the degree of photolysis by measuring weight loss:

To measure the stabilization efficiency, the percentage weight loss of the photodegraded PVC films, in both the presence and absence of additives, was calculated using the following equation:

$$\text{Weight loss \%} = [(W_1 - W_2) / W_1] \times 100 \quad (4)$$

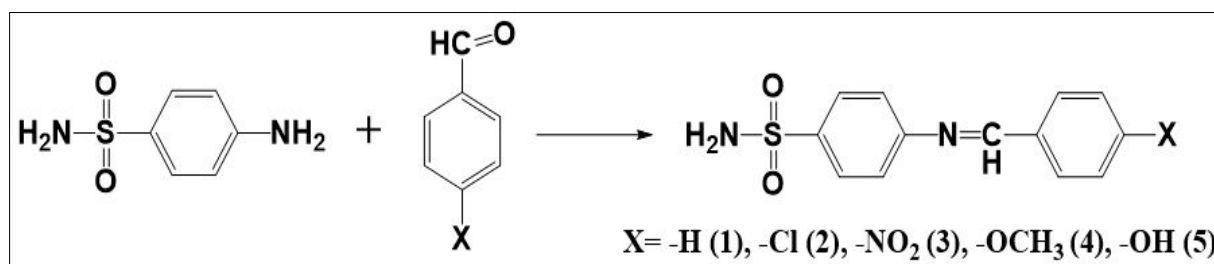
Where W_1 represents the mass of the initial sample (before irradiation), and W_2 refers to the mass of the sample after the irradiation process is complete^[34].

Technology of Scanning Electron Microscopes (SEM)

Scanning electron microscope (SEM) observations were performed using two instruments: a KYKY-EM 3200 microscope operating at an accelerating voltage of 26 kV, and an Inspect S50 (FEL, Czech Republic) operating at an accelerating voltage of 15 kV at Tarbiat Modares University, Tehran, Iran.

Synthesis of Benzene Sulfonamide Derivatives

The preparation of benzene sulfonamide derivatives was carried out by reacting 0.172 (g) of 4-aminobenzenesulfonamide with 0.106 (g), 0.14 (g), 0.151 (g), 0.136 (g), and 0.122 (g) of benzaldehyde, 4-chlorobenzaldehyde, 4-nitrobenzaldehyde, 4-methoxybenzaldehyde, and 4-hydroxybenzaldehyde, respectively. The reaction steps involved using 30 (mL) of absolute ethanol as a solvent and adding a few drops of glacial acetic acid as a catalyst. The reaction mixtures were then refluxed for 3-4 (h) at 80-90 ($^{\circ}\text{C}$), and all products were recrystallized and dried to obtain pure products, as shown in the reaction scheme 1.



Scheme 1: Synthesis of Schiff bases of benzenesulfonamide (1–5)

Table 1: Physical properties of benzenesulfonamide derivatives (1-5)

Compounds	Mol.F	M.P C ⁰	Yield %	Color
1	C ₇ H ₆ O	193-196	79	Yellow
2	C ₇ H ₅ ClO	187-190	81	Light yellow
3	C ₇ H ₅ NO ₃	201-104	75	Brown
4	C ₈ H ₈ O ₂	177-180	68	Gray
5	C ₇ H ₆ O ₂	212-215	87	Orange

Results and Discussion

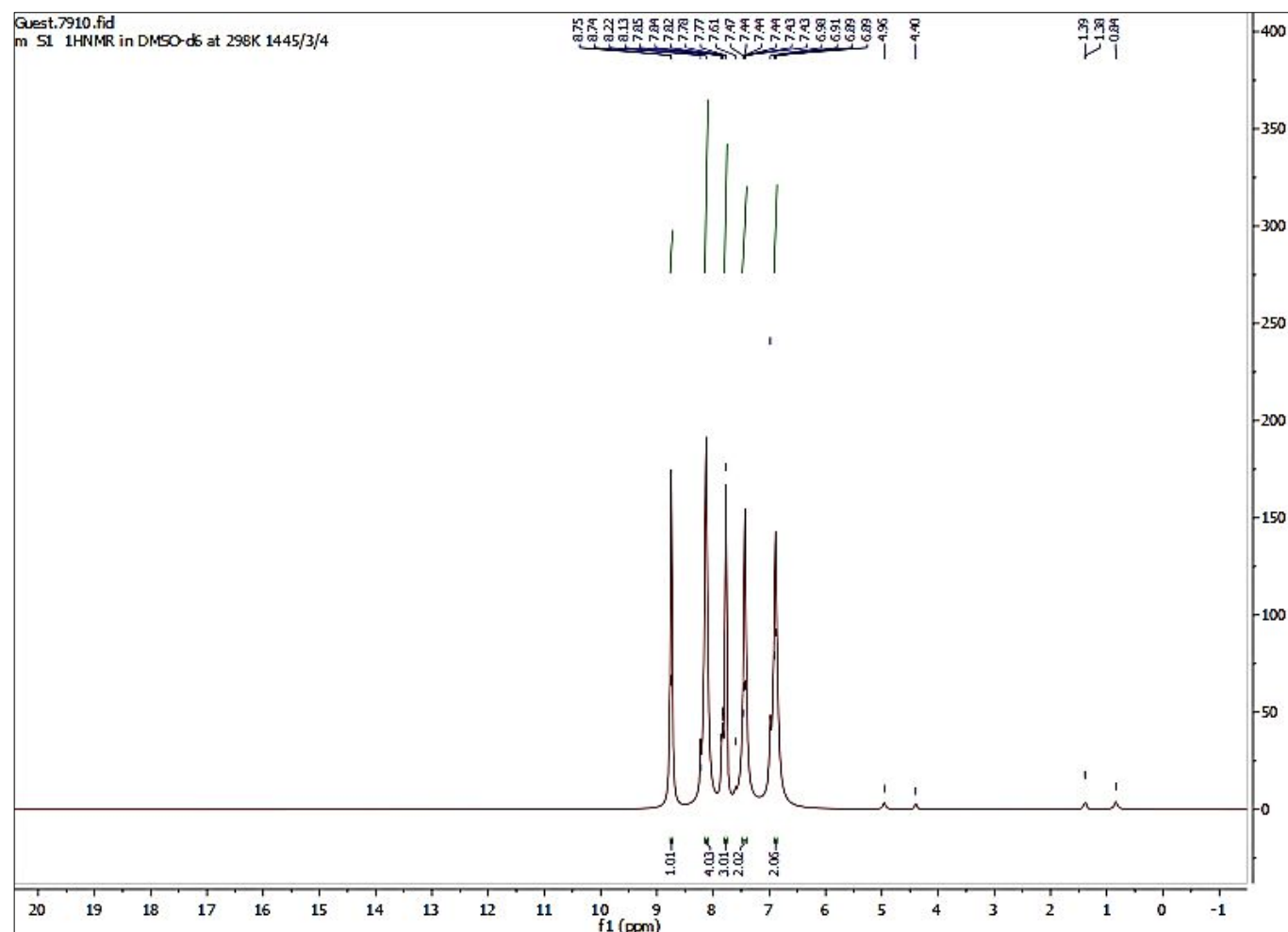
¹H-NMR spectroscopy

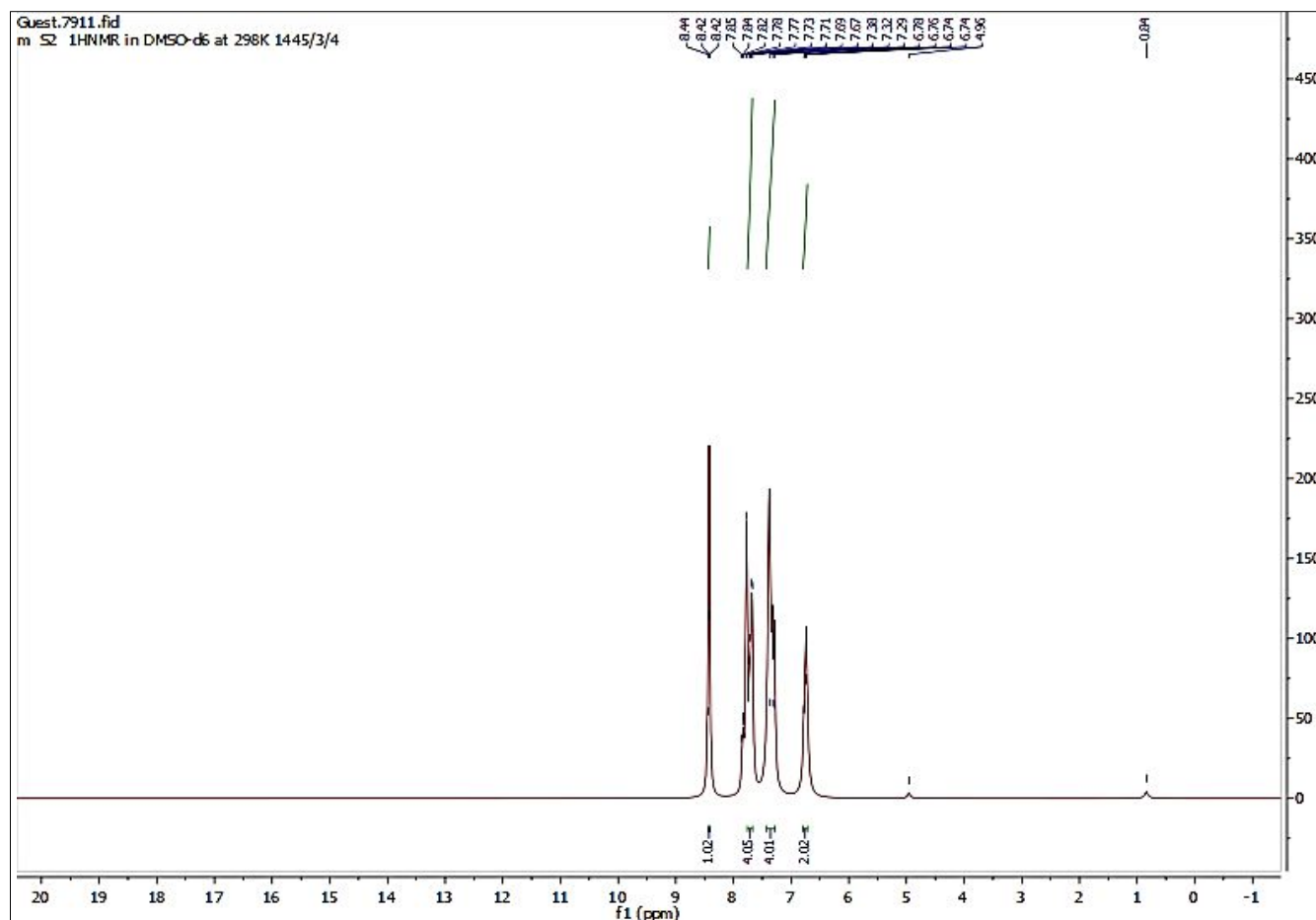
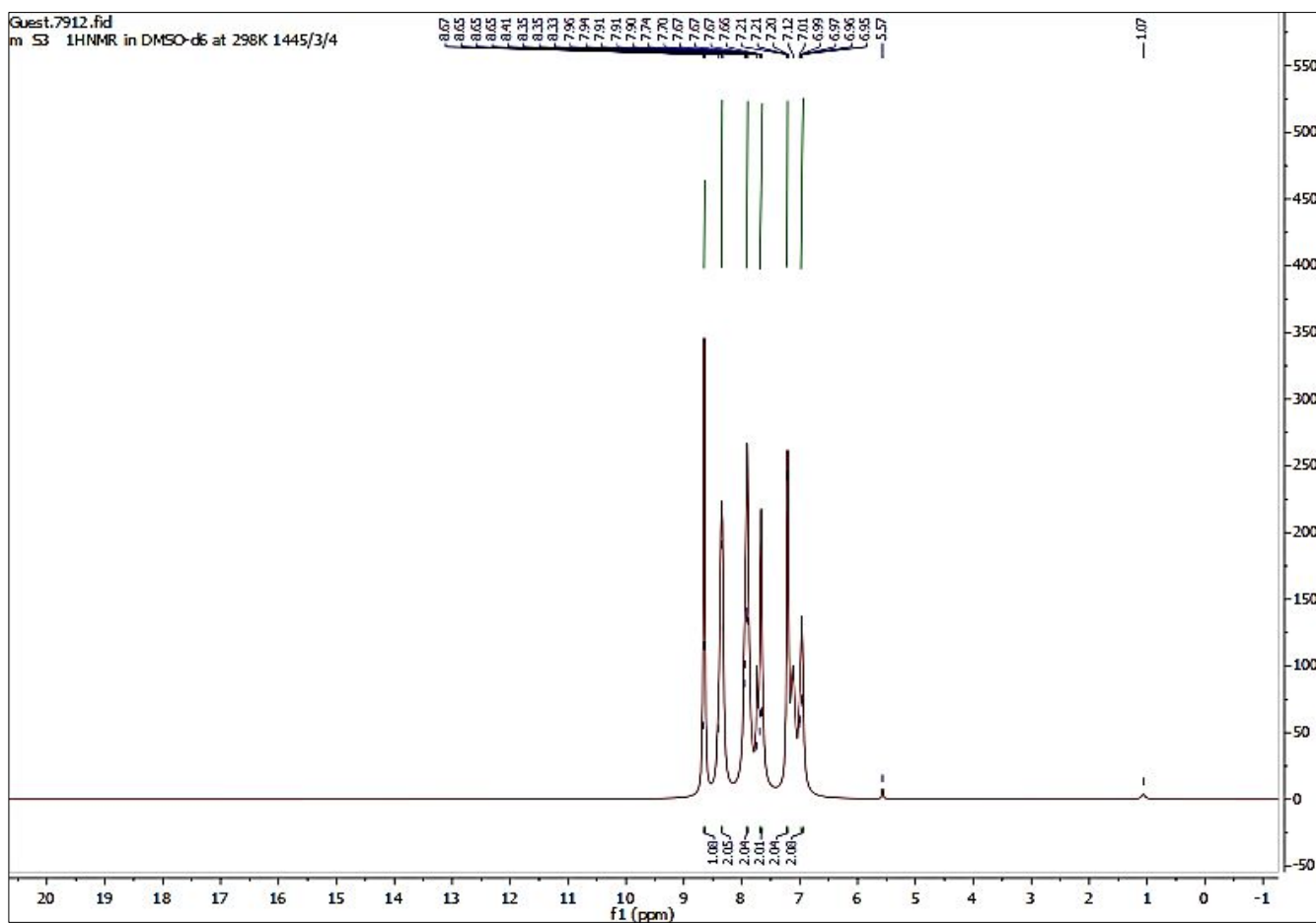
In the proton nuclear magnetic resonance (¹H-NMR) spectra of the synthesized benzenesulfonamide derivatives 1-5, the characteristic signal of the formyl proton (H-C=O) at 10.01 ppm was observed in all benzaldehyde derivatives, and a

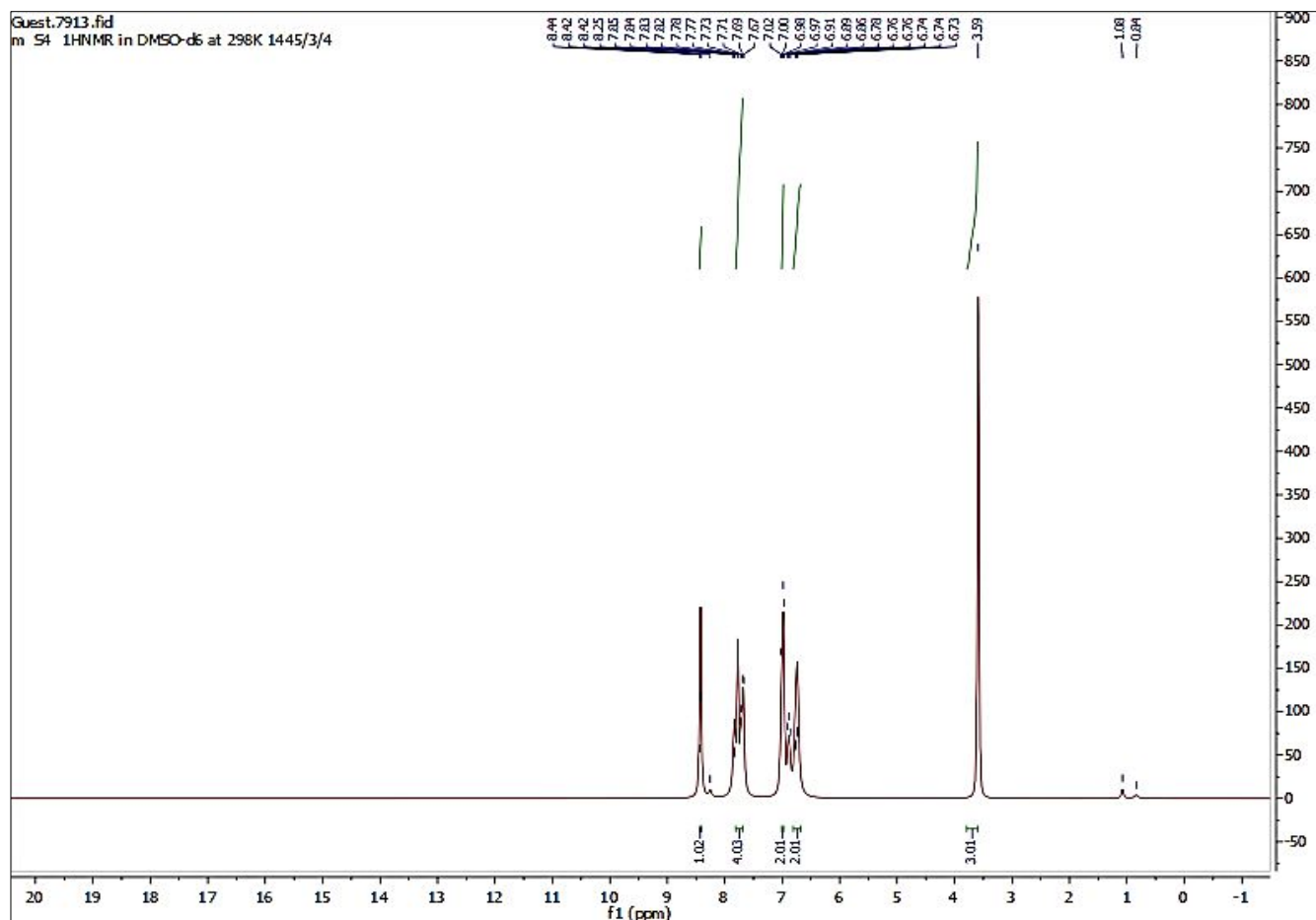
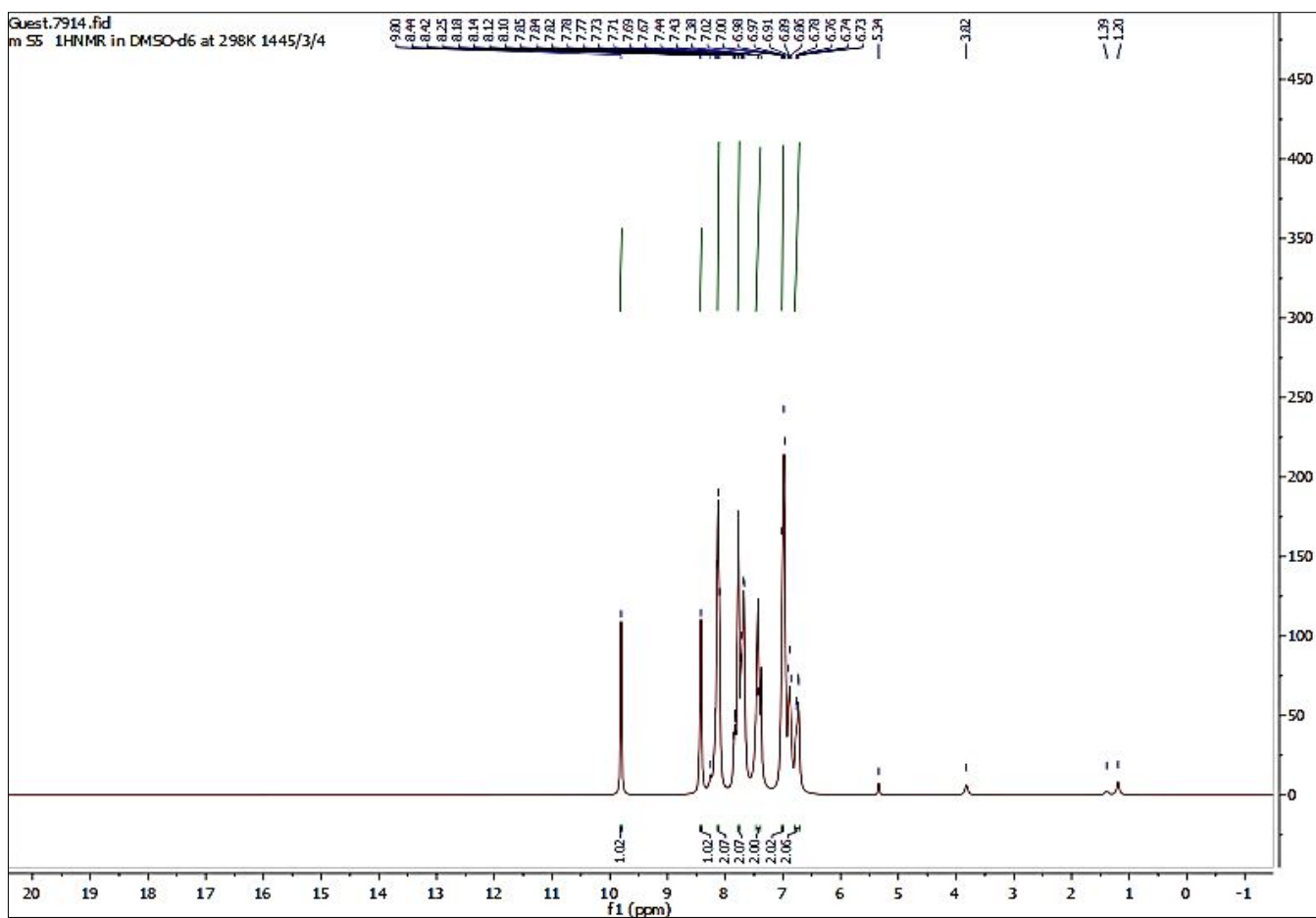
new signal attributed to the imine proton (N=C-H) appeared in the range of 8.41-8.76 (ppm). This can be seen in Figure (1-5) and summarized in Table 2. In addition, the signal of the hydroxyl proton (OH) of compound 5 appeared at 9.80 (ppm), and the signal of the three methoxy group protons (O-CH₃) of compound 4 appeared at 3.59 (ppm).

Table 2: ¹H-NMR data for benzenesulfonamide derivatives (1-5)

Comp	¹ H NMR (500 MHz, DMSO-d ₆)
1	δ 8.76 – 8.72 (m, 1H), 8.13 (s, 4H), 7.77 (d, <i>J</i> = 8.1 Hz, 3H), 7.48 – 7.40 (m, 2H), 6.92 – 6.86 (m, 2H).
2	δ 8.43 – 8.41 (m, 1H), 7.70 (dd, <i>J</i> = 20.5, 8.3 Hz, 4H), 7.33 (d, <i>J</i> = 44.9 Hz, 4H), 6.79 – 6.72 (m, 2H).
3	δ 8.70 – 8.64 (m, 1H), 8.35 (s, 2H), 7.94 – 7.89 (m, 2H), 7.73 – 7.61 (m, 2H), 7.22 – 7.19 (m, 2H), 7.03 – 6.91 (m, 2H).
4	δ 8.46 – 8.41 (m, 1H), 7.75 (dd, <i>J</i> = 27.7, 8.8 Hz, 4H), 6.99 (d, <i>J</i> = 9.9 Hz, 2H), 6.81 – 6.68 (m, 2H), 3.58 – 3.60 (s, 3H).
5	δ 9.80 (s, 1H), 8.43 (d, <i>J</i> = 10.4 Hz, 1H), 8.12 (s, 2H), 7.77 (d, <i>J</i> = 8.1 Hz, 2H), 7.47-7.39 (m, 2H), 7.00 (s, 2H), 3.57-6.72 (m, 2H).



Fig 2: ^1H -NMR data for Compound 2Fig 3: ^1H -NMR data for Compound 3

Fig 4: ^1H -NMR data for Compound 4Fig 5: ^1H -NMR data for Compound 5

Results and-Discussion

Using UV-vis spectroscopy, the photodegradation rate of PVC films was studied: The prepared PVC films 40 (μm) thick containing 0.5 wt% of Schiff bases derivatives of benzenesulfonamide were exposed to UV light to study the photodegradation and evaluate the effect of these additives, whether present or absent (benzenesulfonamide derivatives (1-5). The observed changes in the irradiated PVC films indicated decomposition. Plotting the data according to

Equation 3 yielded a straight line, the slope of which represents the decomposition rate constant of the PVC films. After exposure of pure PVC films (Without additives) to UV radiation, Figure 6 shows the change in $\ln(A_t - A_\infty)$ as a function of irradiation time (t). Similarly, Figures 6 to 11 show the corresponding changes in $\ln(A_t - A_\infty)$ versus irradiation time for PVC films containing 0.5 wt% of benzenesulfonamide derivatives as stabilizers after such exposure.

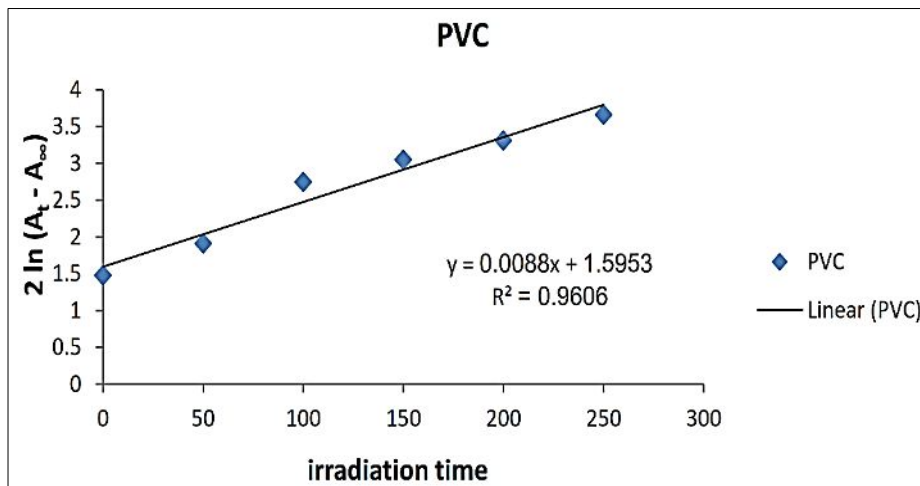


Fig 6: The correlation between $2 \ln (A_t - A_\infty)$ and irradiation time for PVC film (Without additives)

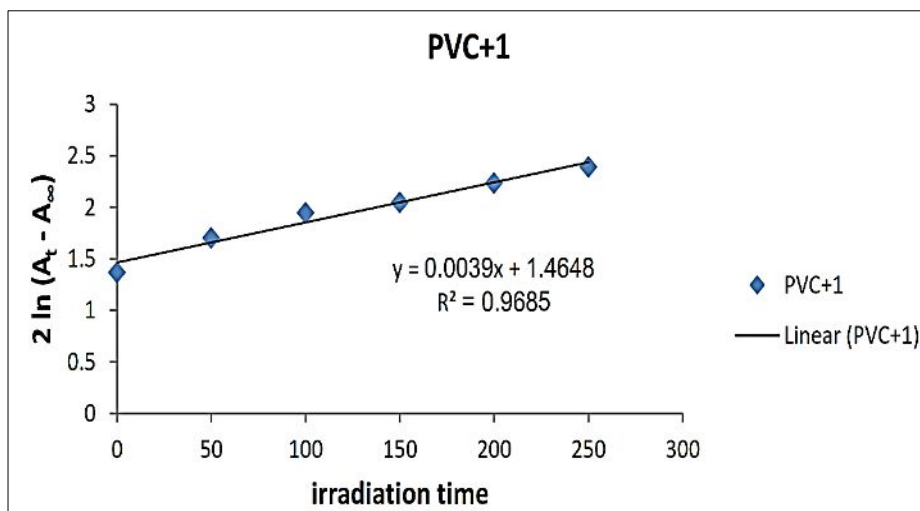


Fig 7: The correlation between $\ln (A_t - A_\infty)$ and irradiation time for PVC+1 film

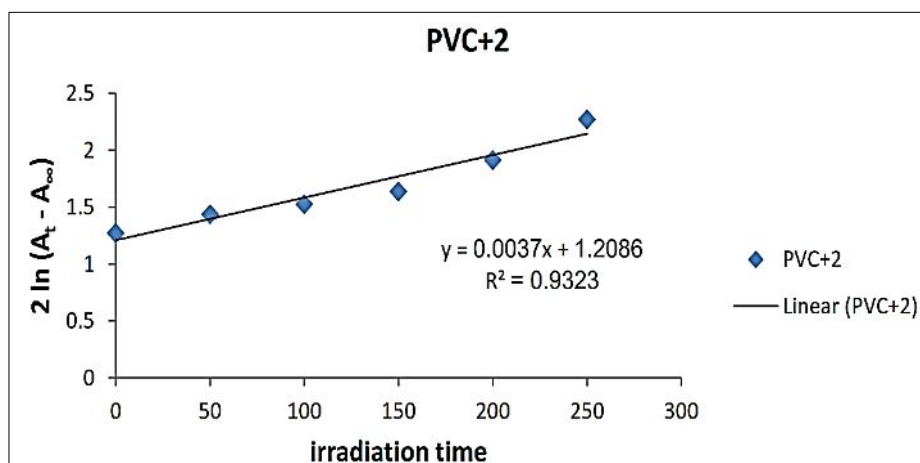


Fig 8: The correlation between $\ln (A_t - A_\infty)$ and irradiation time for PVC+2 film

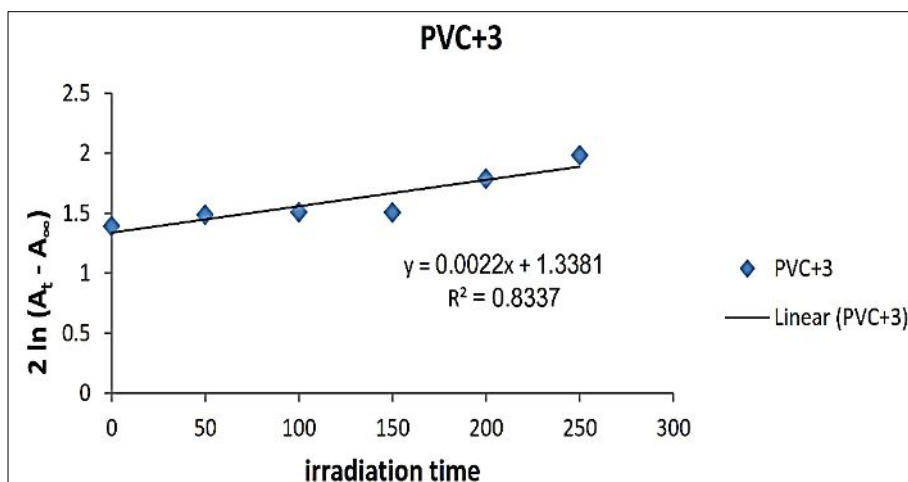


Fig 9: The correlation between $\ln (A_t - A_\infty)$ and irradiation time for PVC+3 film

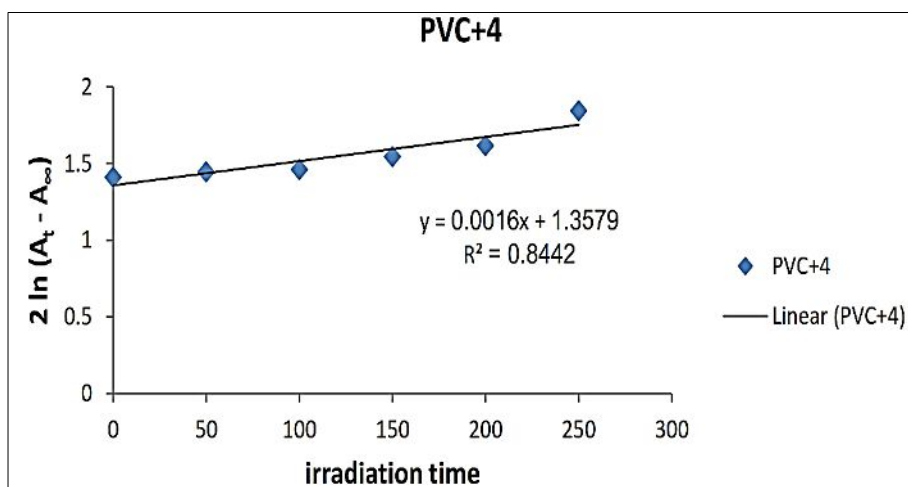


Fig 10: The correlation between $\ln (A_t - A_\infty)$ and irradiation time for PVC+4 film

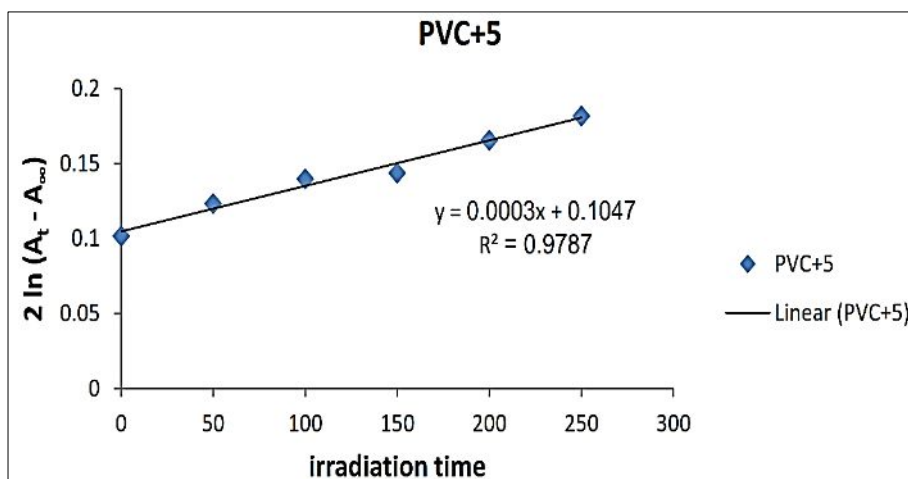


Fig 11: The correlation between $\ln (A_t - A_\infty)$ and irradiation time for PVC and PVC(1+5) film

Table 3 presented the rate constants for the films that were blank and those that had additives. The breakdown rate's sensitivity to the type and presence of benzenesulfonamide Schiff bases is shown by k_d values [31]. The k_d for PVC (Blank) film, for instance, was high $88 \times 10^{-4} (\text{sec}^{-1})$, but it drastically decreased $39 \times 10^{-4} (\text{sec}^{-1})$ when benzenesulfonamide derivatives were used as additives. When benzenesulfonamide derivatives are present, PVC photostabilization follows this order: $5 > 4 > 3 > 2 > 1$.

Table 3: Calculation of k_d for PVC films after 250 hours of UV exposure.

Films	k_d value (sec^{-1})
PVC (blank)	88×10^{-4}
PVC + 1	39×10^{-4}
PVC + 2	37×10^{-4}
PVC + 3	22×10^{-4}
PVC + 4	16×10^{-4}
PVC + 5	3×10^{-4}

Investigation of the impact of UV exposure on weight loss

Photo-oxidation of PVC leads to the formation of free radicals, which promote cross-linking and the elimination of hydrogen chloride (HCl), resulting in a decrease in polymer mass. To evaluate the photostabilizing efficacy of benzenesulfonamide derivatives (1–5) in PVC, the weight loss resulting from photo-oxidation and photodegradation processes was measured. The percentage weight loss was calculated using Equation 4, where W_1 and W_2 represent the initial and final PVC masses, respectively, post-irradiation.

Figure 12 shows that the rate of PVC weight loss was rapid during the initial 100 hours of irradiation and continued to increase in the absence of benzenesulfonamide 1–5. This confirms the significant impact of these additives on PVC stabilization. After 250 hours of irradiation, the control (blank) PVC film exhibited a 61% weight loss (measured after 50 hours from the start of the experiment). In contrast, PVC films containing benzenesulfonamide compounds 1–5 showed lower weight losses of 53%, 47%, 38%, 30%, and 26%, respectively. The figure illustrates that benzenesulfonamide 5 demonstrated the highest stabilization efficacy for PVC [35].

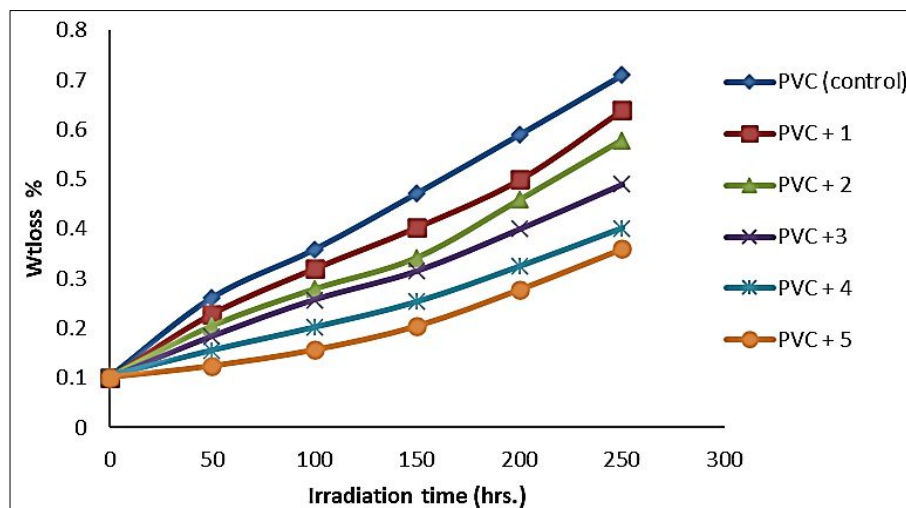


Fig 12: Change in weight loss (%) of PVC and PVC containing compounds (1-5) after 250 hours of irradiation.

The SEM micrographs

Scanning electron microscopy (SEM) images of irradiated PVC films (neat PVC, surface of PVC/benzenesulfonamide films) taken at 15.00 kV after 250 hours of irradiation are shown in Figure 13. These light patches are activation areas that contribute to the deterioration of aging. Both clean PVC and PVC/benzenesulfonamide films developed many surface fractures [36]. After the films were exposed to UV light for 300 hours, the dehydro-chlorination and oxidation

processes on their surface caused such fissures. The PVC/Schiff basis films' surface cracks, however, are hardly noticeable, but the apparent cracks on those samples' surfaces are more numerous than on blank PVC. An ice-rock-like structure is created as a result of hydrogen chloride and volatile chemicals cross-linking and evolving [37]. Good compatibility and homogeneous dispersion are two important factors in improving the performance of PVC composites [38].

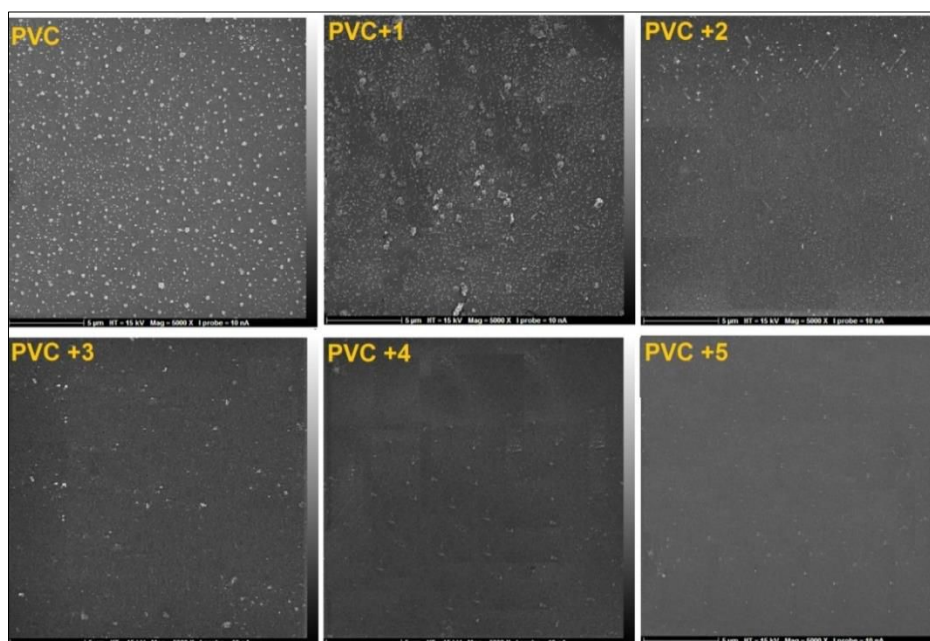


Fig 13: SEM images 10 (nA) of PVC and PVC (1-5) films after irradiation for 250 (h).

Conclusions

PVC films incorporating benzenesulfonamide-derived Schiff bases showed significantly reduced photodegradation rate constants (k_d) compared to the control (Pure) PVC films. Specifically, the k_d values of the modified PVC films ranged from $3 \times 10^{-4} \text{ (s}^{-1}\text{)}$ to $39 \times 10^{-4} \text{ (s}^{-1}\text{)}$, compared to $88 \times 10^{-4} \text{ (s}^{-1}\text{)}$ in the control film. Among the derivatives tested, benzenesulfonamide 5 showed the highest light-stabilizing activity. These results clearly demonstrate the light-stabilizing effect of benzenesulfonamide derivatives on PVC films. Weight loss tests of irradiated films confirmed a weight loss of 35% in the presence of benzenesulfonamide compounds (1-5). Scanning electron microscopy (SEM) of additive-treated PVC after irradiation for 250 h revealed an inhomogeneous surface morphology resembling broken rocks, indicating structural changes induced by the irradiation process.

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