Enhanced UV Resistance of Poly(methyl methacrylate) Through Schiff Base and Metal Oxide Nanoparticle Incorporation

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Abstract: *In this work, a novel poly(methyl methacrylate) (PMMA) material was prepared by incorporating Schiff base and metal oxide nanoparticles (NPs). Hence, different polymer derivatives were produced, and their resistance to light degradation was improved. PMMA was subjected to chemical modification, first by reacting PMMA with an excess of ethylene diamine to reduce the occurrence of cross-linking. Second, it underwent a reaction with the amino group of 4-methoxybenzaldehyde to produce Schiff base molecules. These molecules were subsequently infused with various metal oxide NPs, namely: TiO_2, CuO, Cr_2O_3, Co_2O_3, and NiO to work as photostabilizers and prepare modified PMMA films. The synthesized polymers were analyzed using Fourier transform infrared (FTIR) and NMR spectroscopy to confirm their structures and determine the extent of structural alterations. FTIR and weight loss measurements assessed modified PMMA's UV stabilization. The effectiveness of functional groups was assessed by monitoring their growth. Additional analyses were done included atomic force microscopy (AFM), scanning electron microscopy (SEM), and microscopic imaging. Unmodified PMMA performed worse than Schiff base polymers. The modified PMMA photodegraded less than the blank films after 300 h of UV exposure. The unit's strong internal conjugation absorbs UV light, which improves performance.*

Keywords: *polymethylmethacrylate; UV irradiation; photo-chemical degradation; thin films; nanoparticles*

■ INTRODUCTION

Polymers are extensively utilized in various industries, including medicine, building, energy, water treatment, and electronics [1]. Attributed to their transparency and biocompatibility, thermoplastic polymers, such as polymethyl methacrylate (PMMA), cyclic olefin copolymer, and polystyrene, are often used in microfluidic devices [2]. Comprehensive investigations were carried out to alter and improve the surface characteristics for specific purposes while maintaining the inherent benefits [3]. Hence, surface modification techniques have significantly broadened the range of applications for these materials [4]. However, PMMA is a commonly used material in microfluidic apparatus for large-scale manufacturing because of its exceptional biocompatibility, scalability, and superior optical transparency [5]. The wettability of polymers plays a vital role in determining the qualities of adhesion, collocability, biocompatibility, and electrical characteristics. A range of surface treatment techniques, including physical, chemical, plasma, corona, annealing, and patterning, were created to augment the surface energy of PMMA [4]. In turn, this improves the polymer's capacity to be wetted and enhances its adhesive qualities. Treatments on the morphology upgrade the surface energy of PMMA by either decreasing or increasing its value depending on the desired uses.

Photochemical oxidation is a method that uses ultraviolet (UV) radiation to eliminate pollutants, particularly organic ones, from plastic surfaces and enhance adherence. UV irradiation causes a chemical reaction between PMMA and oxygen or ozone, which results in the formation of carbonyl and carboxyl hydrophilic groups on the surface of PMMA [6]. This procedure enhances the hydrophilicity of the hydrophobic PMMA surface; the degree of hydrophilicity is contingent upon the length of UV irradiation and the storage duration after exposure [7]. The integration of nanoparticles (NPs) into the polymer matrix became imperative to enhance the characteristics of the polymer. Different carbon-based, metallic, minerals, and NPs were employed to enhance the electrical, mechanical, and thermal characteristics of polymers [8].

Over the past decade, PMMA has been investigated as a prominent polymer in publications related to polymer optical fiber. Hamdalla and colleagues [9] produced and studied doping PMMA with different rare earth elements. In comparison, Saris et al. [10] conducted a study on europium aluminum-incorporated polymer composites for optical waveguide amplifiers. Also, Nafee et al. [11] investigated the optical and structural characteristics of PMMA infused with Rhodamine B. Lyutakov et al. [12] conducted a study to analyze the effectiveness of PMMA thin films infused with silver ions, NPs, and a silver-imidazole polymer complex. Finally, Sáez-Rodríguez et al. [13] investigated the photosensitivity mechanism of undoped PMMA under UV radiation.

This work aimed to synthesize and analyze newly modified PMMA composites that contain different metal oxide NPs, with a focus on investigating the impact of UV radiation on the modified PMMA materials with NPs. The structure and physical characteristics of the films were analyzed by applying multiple techniques, including Fourier transform infrared (FTIR) spectroscopy, optical microscopy, scanning electron microscopy (SEM), and atomic force microscopy (AFM). The films were evaluated for weight loss to compare the durability of the modified films against sunlight exposure with the plain ones. Beyond material stabilization, this work offers substantial environmental potential by potentially reducing plastic use, which is a main contributor to the increasing damage to marine ecosystems.

■ EXPERIMENTAL SECTION

Materials

PMMA, with an average molecular weight of 74,315 g/mol, ethylenediamine (≥ 99%), and 4-methoxybenzaldehyde (98%) were acquired from Merck (Gillingham, UK). The metal oxide NPs, namely CuO (58.43 nm), Cr_2O_3 (18.51 nm), TiO_2 (48.82 nm), Co_2O_3 (15.14 nm), NiO (10.00 nm), chloroform ($CHCl_3$) and tetrahydrofuran (THF) solvents were obtained from the same supplier

Instrumentation

The FTIR spectra of the PMMA films were obtained using a Shimadzu spectrophotometer Model 8400 (Japan) with a frequency range of 400–4000 cm^{-1}. The QUV tester, which is equipped with UV-B 365 lamps, was purchased from Philips company in Saarbrücken, Germany. The PMMA films were analyzed using a Bruker DRX300 NMR spectrophotometer (Bruker, Zürich, Switzerland) to get the ^1H-NMR and ^{13}C-NMR spectra. The PMMA film's surface, which had undergone radiation exposure and subsequent modification, was analyzed using SEM of model SIGMA 500 VP microscope from ZEISS Microscopy (Jena, Germany). The films' morphology was further examined using a Veeco model AFM. In order to mitigate distortion during the AFM test, the materials were positioned within a nitrogen atmosphere. The tapping mode was applied to acquire three-dimensional images at a 0.8 Hz scanning rate. Finally, a MEIJI TECHNO microscope was employed to conduct a thorough analysis of the surface morphology.

Procedure

Preparation of PMMA containing ethylenediamine

An amount of 5 g of PMMA were agitated in a solution of 15 mL of a 1:1 mixture of 2-propanol and water for 10 min at 25 °C. After drying, the polymer was immersed in a solution containing 29.5 g of ethylenediamine ($H_2N(CH_2)_2NH_2$) and 15 mL of dimethyl sulfoxide (DMSO). This polymeric mixture was refluxed for 1 h with continuous stirring, then allowed to air dry at 25 °C for 24 h.

Fabrication of PMMA films containing Schiff base

A small aliquot, 15 mL, of $CHCl_3$ was introduced to the aminated PMMA (2.5 g, 25 mmol), and the resulting combination was agitated for 5 min at 25 °C. Afterward, 3.7 g (30 mmol) of 4-methoxy benzaldehyde was introduced, and the combination was agitated at 55 °C for an extra 2 h. Afterward, the mixture was allowed to undergo natural evaporation for 72 h, creating the PMMA-Schiff base combination.

Preparation of Schiff base-NPs PMMA films

The PMMA sheets were filled with metal oxide NPs (Cr_2O_3, CuO, NiO, TiO_2, and Co_2O_3) by sonicating 5.00 g of PMMA and 0.35 mg of metal oxide NPs in 100 mL of $CHCl_3$ for 1 h at room temperature. The resultant mixture was thereafter subjected to 3 h heating with uninterrupted agitation. The final solution was cast on a glass plate at 40 µm thickness to prepare the polymeric sheets subjected to UV examination.

Sheets irradiation by UV light

The plain and modified PMMA films were subjected to UV light with a wavelength of 365 nm and the intensity was 6.2×10^{-8} ein/dm^{3}s. The exposure took place for 300 h at 25 °C. This test aimed to evaluate the films' ability to withstand photodegradation and determine the improvement achieved by adding those NPs. UV irradiation test was performed using a QUV tester manufactured by Q-Panel Company (Homestead, FL, USA), equipped with two 40 W UV-B 365 fluorescent lamps mounted on a stainless-steel plate. The sheets were placed 10 cm away from the tester, with a vertical orientation, and parallelly lined to the lights to achieve consistent exposure on all surfaces. The films were regularly turned throughout the 300 h duration to guarantee uniform exposure. The testing intervals were 50 h to observe the impact of irradiation. Fig. S1 depicts the experimental arrangement used to obtain data on the PMMA surface under various surface treatment conditions.

■ RESULTS AND DISCUSSION

Structural Characterization of Modified PMMA

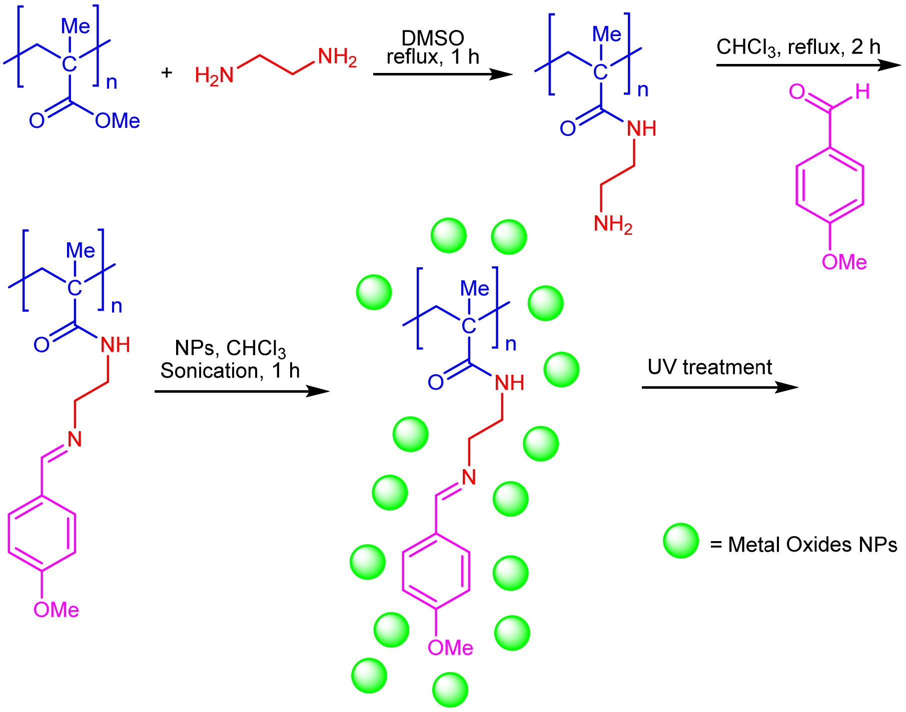
The process of altering PMMA is described in Scheme 1, adhering to recognized protocols. The initial stage involved the attachment of ethylenediamine to the polymer backbone via aminolysis of PMMA in $CHCl_3$ at its boiling point. This technique utilizes an S_N2 reaction mechanism. During the second stage, a Schiff base group is incorporated into the polymer chain by reacting 4-methoxybenzaldehyde with the amino-functionalized PMMA in $CHCl_3$ at its boiling point. The third and last stage is integrating metal oxide NPs into the PMMA chains, enabling the formation of a tangible connection between the polymer and NPs. Table 1 presents a summary of the physical characteristics of the PMMA composites.

The chemical structure of PMMA was analyzed during its alteration process using FTIR spectroscopy. During the aminolysis process of PMMA with ethylenediamine, novel bonds were detected. These bonds were identified at 3298 and 1245 cm^{-1}, which indicate the presence of NH_2 and C–N stretching vibrations. Then, when PMMA reacted with 4-methoxybenzaldehyde, a distinct peak emerged at 1637 cm^{-1}; this peak suggests the creation of a C=N bond and Schiff base production.

Table 1. Physical properties of PMMA materials

PMMA film	Color	M.P. (°C)
PMMA/SB	Orange	185–187
PMMA/SB/Co_2O_3 NPs	Light orange	197–199
PMMA/SB/Cr_2O_3 NPs	Light yellow	177–179
PMMA/SB/CuO NPs	Light gray	154–156
PMMA/SB/NiO NPs	Gray	238–240
PMMA/SB/TiO_2 NPs	Light yellow	135–137

*SB = Schiff base



Scheme 1. Synthesis of modified PMMA/NPs

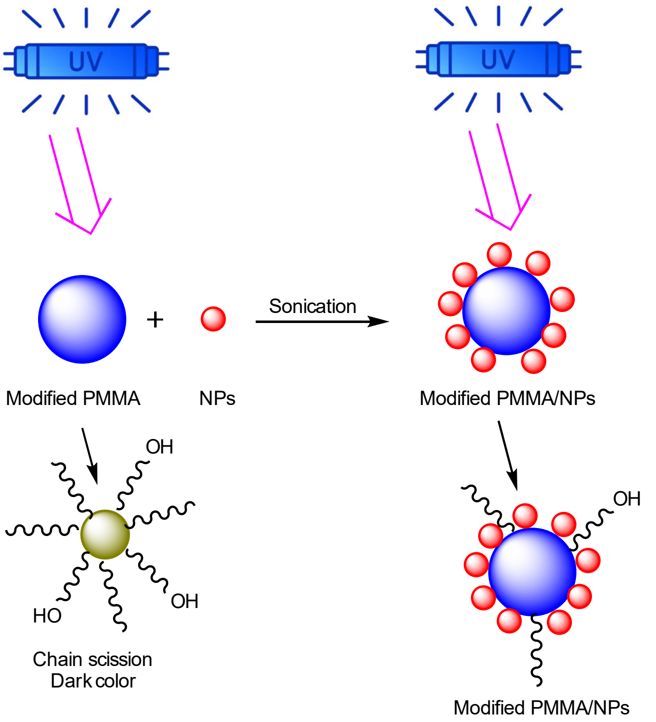
The confirmation of the structures of PMMA modified with Schiff base was conducted through the analysis of ^{1}H-NMR and ^{13}C-NMR data. The ^{1}H-NMR spectrum displayed singlet signals at around 8.04 ppm, indicating the presence of the NH proton. This observation supports the occurrence of aminolysis reaction with ethylenediamine. In the PMMA/Schiff base spectra, singlet signals were observed at around 8.20 ppm, which can be attributed to the CH=N proton. This suggests the formation of the Schiff base. The ^{13}C-NMR spectra displayed singlet signals at 55.29 ppm, which were attributed to the methoxy group. This observation provides more evidence for the structural identification of PMMA modified with the Schiff base.

UV Surface Treatment

Treating the surface with UV is a cost-effective and efficient method for altering polymer surfaces without direct contact, using only a few processing steps. The degree to which UV radiation infiltrates and modifies the surface characteristics of PMMA is contingent upon variables, including wavelength, intensity, and duration of exposure [14]. UV radiation triggers breakdown processes within PMMA, leading to the shortening of polymer chains and increased mobility. This effect promotes local rearrangements and conformational alterations. However, these reactions can result in the formation of oxidized groups, such as OH, that have a substantial impact on the characteristics of the material. The efficiency of deterioration in the sample is influenced by the movement and spread of radicals, oxygen, and other reactive substances, which are facilitated by surface imperfections, minor fractures, and tiny pores. The significant decrease in both stress and strain upon failure can be ascribed to these structural defects [15], as illustrated in Scheme 2. The UV radiation effect was monitored by different techniques, as detailed in the following sections.

FTIR Spectra of Irradiated PMMA Sheets

PMMA undergoes photochemical breakdown, resulting in the generation of crumbs that possess specific functional groups, such as hydroxy (OH) and carbonyl groups. The reaction forms free radicals through the homolytic breakage of C–Me and C–CO_2Me bonds, as shown in Fig. S2. The free radicals can undergo



Scheme 2. The efficiency of degradation inside polymeric chains after UV exposure

a reaction with oxygen, resulting in the formation of peroxy radicals. Subsequently, peroxyl radicals attached to the polymer can extract hydrogen radicals from adjacent polymeric chains, resulting in the formation of hydroperoxides. The O–OH bonds in hydroperoxides undergo homolytic cleavage, resulting in the formation of alkoxy radicals that are linked to the polymer. The alkoxy species remove hydrogen radicals, which leads to the production of corresponding alcohols.

The FTIR spectra monitored the enhancement in the absorption band intensity associated with the OH group under irradiation. More precisely, the increase in the strength of the OH absorption band at 3204 cm^{-1} was observed by comparing it to a reference band that is minimally influenced by radiation, such as C–H at 1443 cm^{-1} or C–C at 750 cm^{-1}. Eq. (1) was used to calculate the OH group (I_{OH}) index during the irradiation. This calculation included the comparison between the absorption values of the OH band (A_s) and the reference band (A_r) [16].

$$I_s = A_s / A_r \quad (1)$$

The variations in peak intensity associated with the vibration of OH group stretching were observed and compared to C–H as a reference peak. The augmentation in the strength of the OH absorption band while exposed to UV light was a sign of PMMA photodegradation. The I_{OH}, denoting the index of the OH group, was determined for each modified PMMA by applying Eq. (1), which measures the absorption intensities of the OH band (A_s) and the reference band (A_r). The I_{OH} readings were graphed against the duration of irradiation in Fig. 1 to demonstrate the degree of photodegradation. The I_{OH} value for the PMMA/Schiff base film containing NiO NPs

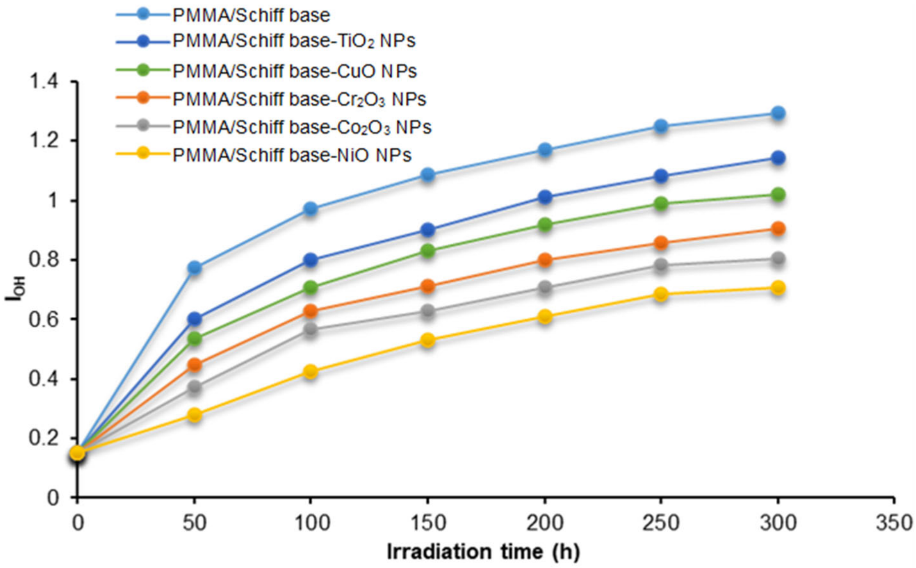


Fig 1. Hydroxyl index (I_{OH}) of PMMA films during the irradiation process

after 300 h of irradiation was 0.705, whereas that of the PMMA/Schiff base film without these NPs was 1.294.

The I_{OH} experienced an initial quick spike at the start of irradiation, which was then followed by a slower but consistent rise over a period of time. The unaltered PMMA film had the most notable augmentation in I_{OH} compared to the changed versions. More precisely, PMMA that was altered with Schiff base exhibited reduced variations in I_{OH}. On the other hand, films that were altered with Schiff base metal complexes displayed minimal alterations in I_{OH}. These findings indicate that the presence of aromatic groups, heteroatoms, HC=N linkages, and metal ions had a role in decreasing the photodegradation of PMMA. Significantly, NiO demonstrated the most pronounced stabilizing effect among the investigated metal complexes [16-17]. Nevertheless, the precise mechanisms underlying these results are still uncertain.

Weight Loss of Irradiated PMMA Sheets

The process of photodegradation in polymers results in the creation of minute volatile particles as a result of the rupture of chemical bonds. The weight loss (%) can serve as a reliable measure of the degree of photodegradation of PMMA. To examine this, we observed the decrease in weight of PMMA films that were exposed to radiation. The weight loss % of the PMMA films was determined at various irradiation durations (Eq. (2)), which compares the initial weight of the sheets (W_0) to their weight after irradiation (W_t) [17-18].

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

Hence, we evaluated the photostability of modified PMMA films by quantifying the weight reduction they experienced following irradiation. The weight loss was determined using Eq. (2). Fig. 2 was drawn to depict the weight loss percentage over the irradiation period. As the time of irradiation increased, there was a proportional rise in weight loss. The unmodified PMMA films exhibited the most significant increase. The weight loss percentage of the PMMA/Schiff base film was the lowest in the presence of NiO NPs and the highest for the PMMA/Schiff base film without NPs.

Surface Morphology Analysis

When PMMA films were exposed to radiation, they experienced surface defects and damage, and they were examined using several microscopic techniques [19-20]. Optical microscopy analysis of the PMMA films exhibited surface imperfections, roughness, and various flaws including dark spots, fractures, and grooves. However, PMMA film treated with only Schiff bases and those grafted with Schiff bases and nano-metals had uniformly smooth surfaces without any visible holes,

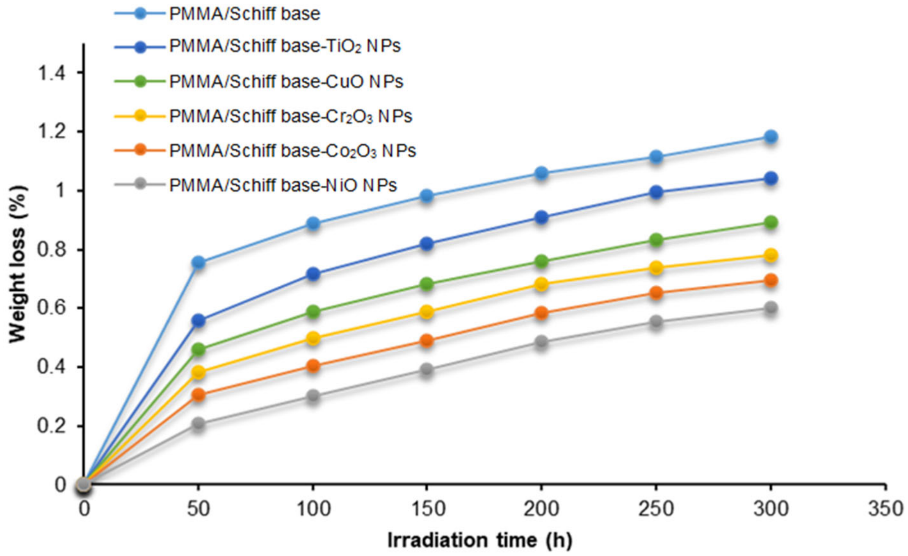


Fig 2. Weight loss percentage of PMMA sheets throughout the irradiation process

fissures, or dark areas and showed no noticeable differences, as illustrated in Fig. 3. The photographs emphasize the difference between the flawless, unharmed surfaces of non-irradiated PMMA sheets and the modified surfaces of those subjected to radiation. However, the surface irregularities in the PMMA films containing metal oxide NPs were less pronounced than those observed in the PMMA/Schiff base films. This suggests that the metal oxide NPs may reduce the photodegradation rate, thereby enhancing the photostability of the irradiated PMMA films.

Further examination of the structures of the films was thoroughly analyzed using a three-dimensional AFM, which offers useful insights into surface characteristics and unevenness. Extended exposure to radiation leads to the breaking of chemical bonds and results in an elevation of surface roughness in films. Fig. 4 exhibits AFM pictures of the surfaces of the polymeric film before and after being exposed to 300 h of irradiation. Remarkably, the irradiation-modified PMMA films exhibited a smoother surface than the blank PMMA film. This finding indicates that the general structure of the PMMA matrix undergoes alterations when it is doped with metal oxide NPs. The results showed that the irradiated PMMA/Schiff base film exhibited a significantly higher surface roughness than the PMMA/Schiff base blends containing metal oxide NPs.

Another morphology test was the SEM, which was used to acquire high-resolution pictures of the surfaces of polymeric films. SEM images of the PMMA film surfaces following irradiation [21-23] are shown in Fig. 5. The provided photos, taken at a magnification of 50×, display the physical structure of the PMMA films that have been exposed to radiation and transformed. The surface of the modified PMMA films exhibits a substantial improvement in smoothness and cleanliness when compared to the plain PMMA film. The inclusion of particles (which may manifest as pores) in the modified PMMA films introduces novel morphological characteristics to the surface. These findings suggest that the addition of metal oxide NPs altered the surface structure of PMMA, leading to a noticeable surface morphology that can be detected by SEM.

From the SEM images, it is clear that the NPs adhered to the surface. However, we were unable to visibly the difference in the surfaces, potentially because of the distinctive surface properties of PMMA [24]. In fact, PMMA is known for its high porosity, resulting in a significant surface area. The surface roughness is likely to be similar in size to the NPs, which means that the surface topography does not simply indicate the presence of nanoparticles. This indicates that the presence of metal oxide NPs-based additives significantly reduced the structural damage caused by photodegradation at the surface of the PMMA material.

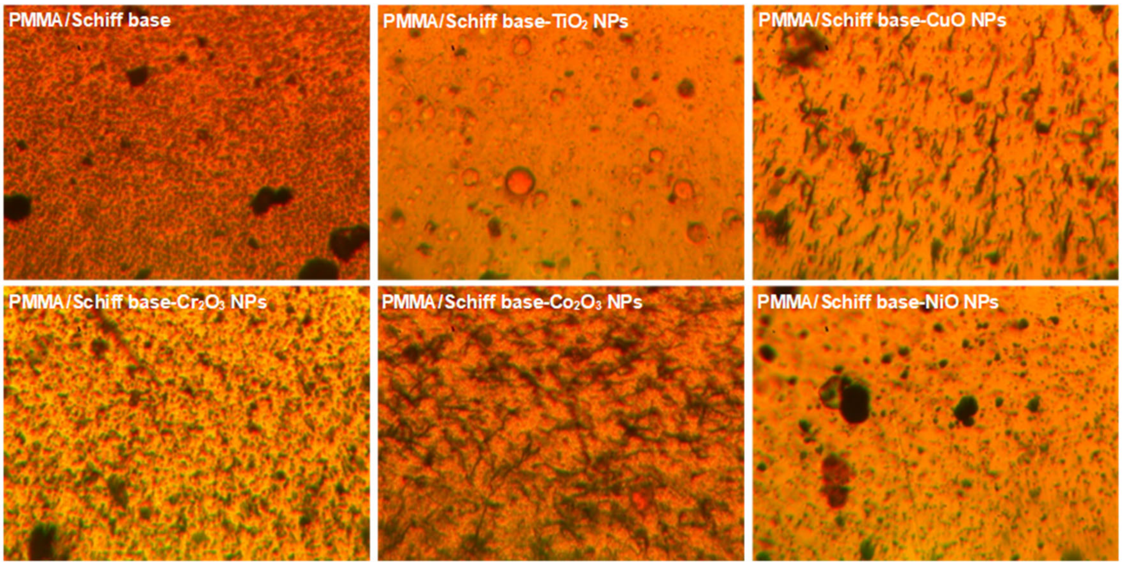


Fig 3. Optical images of irradiated PMMA films

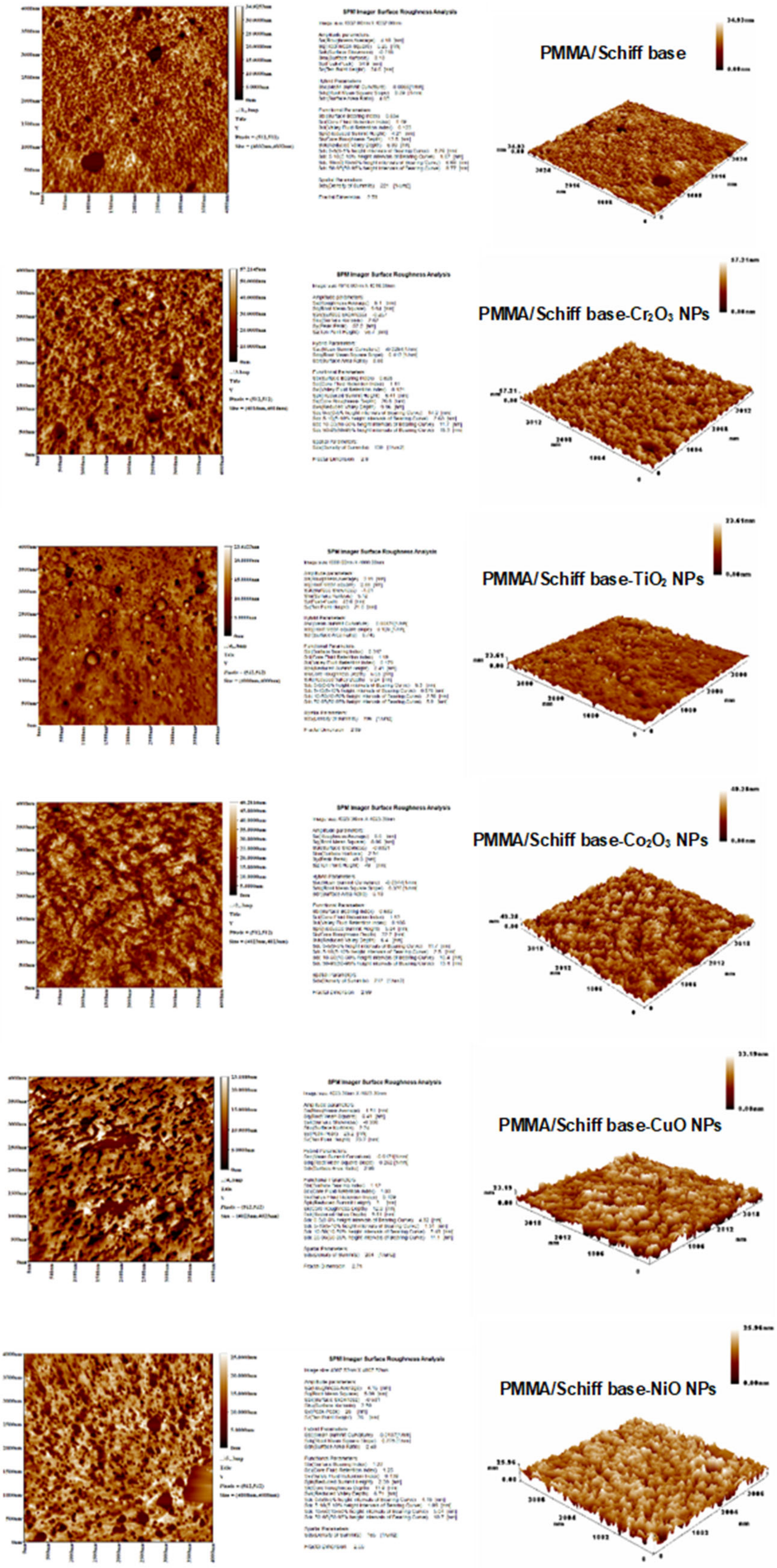


Fig 4. AFM images of irradiated PMMA films

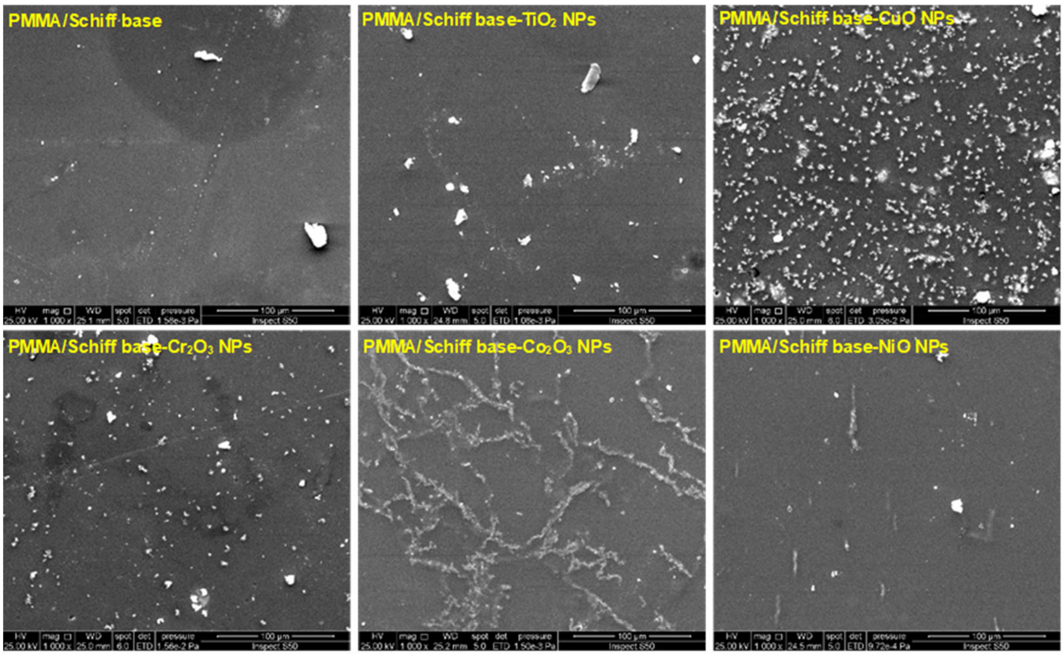


Fig 5. SEM images for PMMA films

CONCLUSION

The addition of Schiff base and metal oxide NPs to PMMA chains resulted in successful surface modification. This alteration allowed PMMA to reduce the harmful impact of UV light with a wavelength of 365 nm over a prolonged duration of up to 300 h. The treated films exhibited substantial enhancement in the photostability of the polymer in comparison to the untreated films. PMMA samples including Schiff base and NiO NPs exhibited minimal surface degradation. Furthermore, modifying the surface of PMMA led to a significant decrease in the rate of photodegradation of the polymer. The Schiff base and metal oxides both contributed to the stabilization of the polymer against photochemical degradation. The Schiff base functioned as a molecule with the ability to absorb UV light, contributing to the overall process of photostabilization. Comparative data analysis showed that the recent PMMA/Schiff base films containing different metal oxide NPs demonstrated superior photostabilizing abilities.

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CONFLICT OF INTEREST

The authors declare there is no known conflict in this work.

AUTHOR CONTRIBUTIONS

Ahmed Hussein conducted the experimental work; Emad Yousif supervised the project; Malath Rasheed wrote the first draft; Dina Ahmed analyzed the data; Muna Bufaroosha administrated the project and visualization; Mohammed Kadhom revised and edited the manuscript.

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