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Synthesis of PVA/PEG/ZnO Nanocomposite Films Combined with Rhodamine B Dye for the Protection and Optical Tracking of Cultural Paper Artifacts

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Abstract

To protect and optically monitor the cultural paper artifacts, a multifunctional PVA/PEG/ZnO nanocomposite film doped Rhodamine B (RhB) was developed. A fluorescent indicator, RhB, was added to monitor degradation, and ZnO nanoparticles were added to enhance the UV shielding and mechanical stability. The films were prepared by solution casting, and were characterized by UV–Vis spectroscopy, FTIR, SEM, fluorescence analysis, and mechanical testing. In the results, it was found that there was a significant improvement in UV blocking efficiency at 5 wt.% ZnO, with a UV value of 94% and a high visible transparency. Accelerated UV-aging tests showed good paper preservation, as the coated paper held 89% whiteness after 120 h exposure in comparison with paper with no coating, which was 61% whiteness after 120 h. The fluorescence intensity gradually decreased as the RhB was aged, which verifies the ability of sensing the presence of RhB. The obtained nanocomposite is multifunctional and shows promise as a smart material for the conservation of paper-based culture.

Keyword: PVA • PEG • ZnO nanoparticles • cultural heritage • nanocomposite films • UV protection, optical sensing.

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Introduction

Cultural items are important part of human civilization and historical heritage which are still available as paper-based items. The degradation of ancient manuscripts, maps, archival papers and rare books is continually happening due to environmental conditions. The degradation of cellulose and discoloration of historical paper can occur for several reasons such as UV radiation, temperature variations, humidity, oxidation and biological contamination[1-3]. Ultraviolet radiation is among the leading causes of the deterioration of cultural heritage materials on paper. Prolonged exposure to UV light leads to cellulose chain scission, oxidation reactions, discoloration and mechanical integrity loss. Nanotechnology has become a promising approach for the recent years aimed at the conservation of cultural heritage since nanomaterials are able to offer the cultural heritage better UV protection, transparency, durability and compatibility with historical substrates. Metal oxide nanoparticles, like ZnO, which show high UV absorption and stability, have received a lot of attention from various nanomaterials studied[4-6]. Thus, the use of ZnO nanoparticles in transparent polymer matrices is an attractive solution to create improved protective coatings for historical paper artefacts[7, 8].New technology in nanotechnology is helping to innovate solutions to preserve cultural heritage. Nanocomposite polymeric coatings are expected to have several useful characteristics, such as mechanical stability, shielding from UV radiation, transparency and resistance to the environment at the same time, which makes them good materials[9-11]. Polyvinyl alcohol (PVA) has many applications in conservation because of its excellent film-forming ability, transparency and chemical stability[12, 13]. Pure PVA films, however, may be brittle and lack flexibility. To impart flexibility and minimize crack formation, polyethylene glycol (PEG) is therefore added in.

Incorporation of inorganic nanomaterials such as ZnO nanoparticles is one of the most effective methods for UV-blocking applications due to the large band gap, optical transparency and photo catalytic properties of these materials[14-16]. Furthermore, the nanoparticles of ZnO are able to enhance the optical and mechanical properties of polymeric matrices[17, 18]. A fluorescent dye with a high emission spectrum like Rhodamine B (RhB) can be used as an optical probe to monitor the changes of the environment and the structure occurring inside the polymeric matrix[19, 20].

Although many developments have occurred in the field of polymer-based conservation coatings, the majority of reported systems offer only passive UV protection, and cannot be used to monitor the deterioration of paper in real-time. In order to overcome this problem, the present study aimed at the realization of a multifunctional coating for paper-based cultural heritage based on PVA/PEG/ZnO/Rhodamine B nanocomposite. Both ZnO nanoparticles and Rhodamine B were then added to increase UV-shielding capabilities and mechanical stability, respectively, and to provide an embedded fluorescent indicator of degradation based on changes in fluorescent intensity due to aging. To the best of our knowledge, this is the first conservation coating to incorporate ZnO-reinforced

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PVA/PEG and in-built fluorescent probe for simultaneous protection and condition monitoring during accelerated UV aging.

The nanocomposite films were prepared by the solution casting technique, and systematically characterized with respect to structural, optical, morphological and mechanical properties. Their effectiveness in protecting cellulose paper from accelerated UV induced degradation was thoroughly tested and Rhodamine B was used as a non-destructive optical monitoring tool. This holistic approach offers a single coating platform that enables UV protection, mechanical reinforcement, optical transparency and real-time condition monitoring - an improvement over conventional conservation coatings.

Materials and Methods

Materials

The materials and chemical reagents used for the fabrication of the PVA/PEG/ZnO/RhB nanocomposite films are summarized in **Table 1**.

Table 1. Materials and chemical reagents used for the fabrication of nanocomposite films.

Material	Specification	Supplier	Quantity
Polyvinyl alcohol (PVA)	99% hydrolyzed, MW 85,000–124,000 g/mol	Sigma-Aldrich	50 g
Polyethylene glycol (PEG-400)	Analytical grade	Merck	20 mL
Zinc oxide nanoparticles (ZnO)	30–50 nm	Sigma-Aldrich	10 g
Rhodamine B dye	Analytical grade	Merck	0.5 g
Distilled water	Deionized	Laboratory grade	2 L
Cellulose paper sheets	Acid-free archival paper	Conservation grade	30 sheets

Experimental Design

The prepared PVA/PEG/RhB nanocomposite films were formulated with different ZnO nanoparticle concentrations (0, 1, 3, and 5 wt.%), designated as S0, S1, S3, and S5, respectively. The concentration of Rhodamine B was fixed at 0.02 wt.% in all samples to evaluate the effect of ZnO loading on the performance of the nanocomposite films.

Preparation of PVA/PEG/ZnO/RhB Nanocomposite Films

PVA powder was dissolved in distilled water with 10 wt. % concentration, and the solution was obtained by continuous magnetic stirring at 80°C for 2 h to get homogeneous transparent solution. To increase the flexibility of the film, PEG was then added at 20 wt.% of PVA.

The ultrasonic treatment was used for 30 min to disperse the ZnO nanoparticles separately, preventing them from agglomerating, in distilled water. The ZnO suspension was carefully added to the PVA/PEG solution with concentrations of 0, 1, 3 and 5 wt.%. Then 0.02 wt. % of Rhodamine B dye was added.

The final stirring was continued for 1 h after which they were poured into clean glass Petri dishes. The films were left to dry at room temperature for 48 h and were gently removed from the casting surface.

Overall preparation procedure of the fabricated nanocomposite films is schematically illustrated in **Figure 1**.

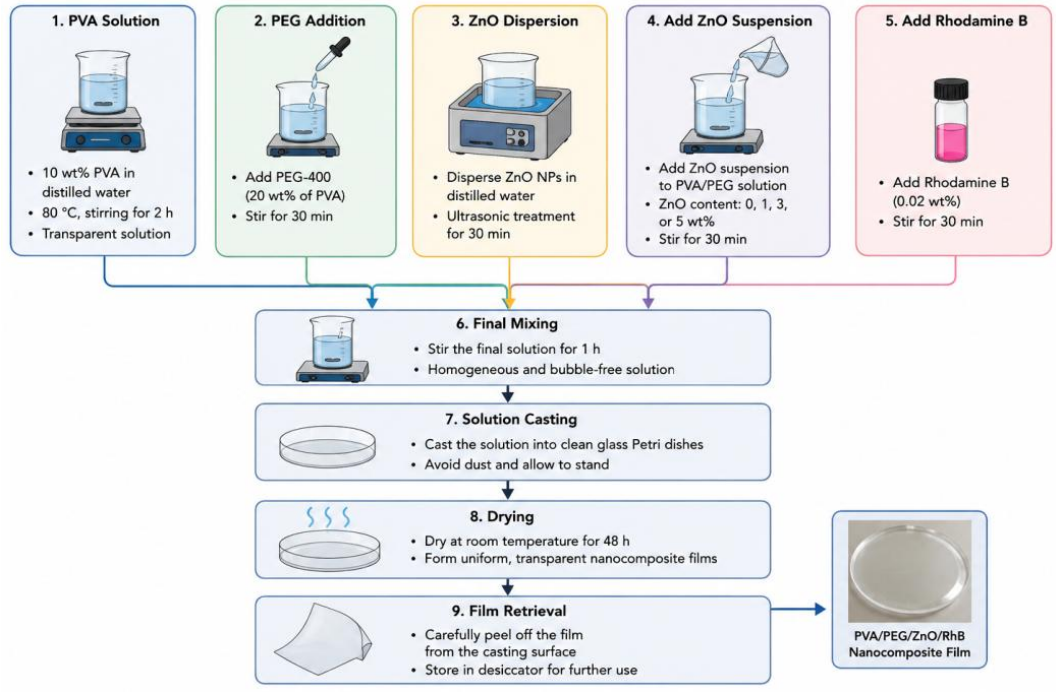


Figure 1. Schematic representation of the preparation procedure for PVA/PEG/ZnO/RhB nanocomposite films.

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Coating Procedure on Paper Artifacts

The prepared nanocomposite coatings were applied onto cellulose paper substrates using a dip-coating method. After drying under ambient conditions for 24 h, the appearance of the fabricated films with different ZnO nanoparticle concentrations was recorded, as shown in **Figure 2**.

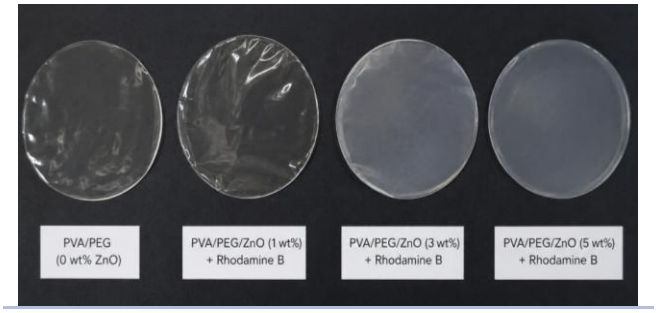


Figure 2. Photographic appearance of fabricated PVA/PEG/ZnO/RhB nanocomposite films with different ZnO nanoparticle concentrations.

Characterization Techniques

The optical properties were analyzed using a UV–Visible spectrophotometer (UV-1800, Shimadzu, Japan) in the wavelength range of 200–800 nm. Fluorescence emission spectra were recorded using a fluorescence spectrophotometer (RF-6000, Shimadzu, Japan) at an excitation wavelength of 540 nm. FTIR spectra were obtained using a Nicolet iS10 FTIR spectrometer (Thermo Fisher Scientific, USA) in the range of 400–4000 cm^{-1}. Surface morphology was examined using a scanning electron microscope (JSM-6510LV, JEOL Ltd., Japan). Mechanical properties were determined using a universal testing machine (Instron 3365, Instron Corporation, USA) according to ASTM standards.

Results and Discussion

Optical Properties

UV–visible spectroscopy was employed to study the optical properties of the nanocomposite films. The absorption spectra obtained and their optical parameters are shown in **Figure 3** and in **Table 2**, respectively. The absorption properties of UV region showed relatively weak absorption for pure PVA/PEG films while there was a significant increase in UV absorption for films containing ZnO nanoparticles below 380 nm which was attributed to the intrinsic band-gap absorption of ZnO nanoparticles. The same UV-shielding properties have been reported in the past for the ZnO/polymer nanocomposite systems[10-13].

The UV-blocking efficiency has been enhanced with increasing ZnO concentration from 34% for pure PVA/PEG film to 94% for the film with 5 wt.% ZnO nanoparticles as mentioned in **Table 3**. Light scattering from particles of ZnO also caused a slight decrease in the visible transparency with increasing ZnO concentration. However, the transparency was over 82%, which means the coating is suitable for cultural heritage conservation applications, where transparency is required[16-18].

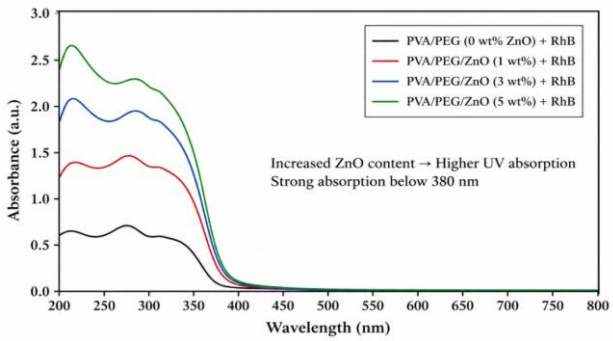


Figure 3. UV–Visible absorption spectra of PVA/PEG/ZnO/RhB nanocomposite films at various ZnO nanoparticle loadings.

Table 2. Optical Properties and UV-Shielding Performance of PVA/PEG/ZnO Nanocomposite Films.

Sample	S0	S1	S3	S5
PVA (wt.%)	90	89	87	85
PEG (wt.%)	10	10	10	10
ZnO (wt.%)	0	1	3	5
λmax (nm)	278	312	336	352
Absorbance	0.41	0.68	1.02	1.35
Visible Transparency (%)	92	89	86	82
UV Blocking Efficiency (%)	34	68	85	94

FTIR Analysis

The intermolecular interactions in the fabricated nanocomposite films were investigated by FTIR. The FTIR spectra are displayed in **Figure 4** and the positions of typical absorption peaks are summarized in **Table 3**.

O–H stretching vibrations linked to hydrogen bonding between PVA and PEG chains were responsible for the wide absorption band seen at about 3300 cm^{-1}. Additionally, the peaks at low frequencies (500 cm^{-1}) confirmed the presence of Zn–O bonds; peaks in the vicinity of 2900 cm^{-1} were attributed to C–H stretching vibrations. Previously, ZnO/polymer nanocomposites have been reported to have similar spectrum properties[15-18].

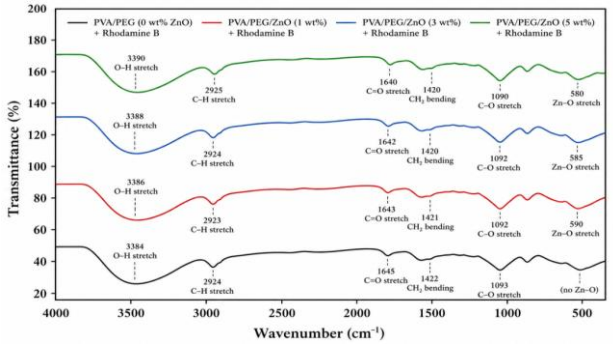


Figure 4. FTIR spectra illustrating intermolecular interactions within the fabricated PVA/PEG/ZnO/RhB nanocomposite films.

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Table 3. Characteristic FTIR absorption peak positions of fabricated nanocomposite films.

Sample	O–H Peak (cm^{-1})	C–H Peak (cm^{-1})	Zn–O Peak (cm^{-1})
S0	3328.0 ± 1.2	2922.0 ± 0.8	—
S1	3316.3 ± 1.0	2919.2 ± 0.9	525.1 ± 1.3
S3	3308.4 ± 1.1	2915.3 ± 0.7	512.4 ± 1.0
S5	3299.2 ± 1.4	2911.1 ± 0.8	503.2 ± 1.2

Table 3 reveals that an additional change was observed in the location of the O-H and Zn-O peaks after the integration of ZnO, which indicates intermolecular interactions between ZnO nanoparticles and the hydroxyl groups in the polymer matrix.

Surface Morphology

The surface shape and dispersion behavior of the nanoparticles in the films of nanocomposite (produced) were examined by scanning electron microscopy (SEM). The SEM micrographs in Figure 5 are representative.

Figure 5 illustrates that at low concentrations of ZnO moderate homogeneous dispersion was achieved and this was moderate nanoparticle agglomeration at high levels of ZnO (5 weight percent). The uniform distribution of the ZnO nanoparticles resulted in better interfacial contact between the polymer matrix and the filler which was similar to previous reports on polymer-based nanocomposite systems[14-16]. Some degree of agglomeration was seen at high concentrations; however, the surface of the films was smooth and continuous and would be appropriate for the wrapping of historical paper objects.

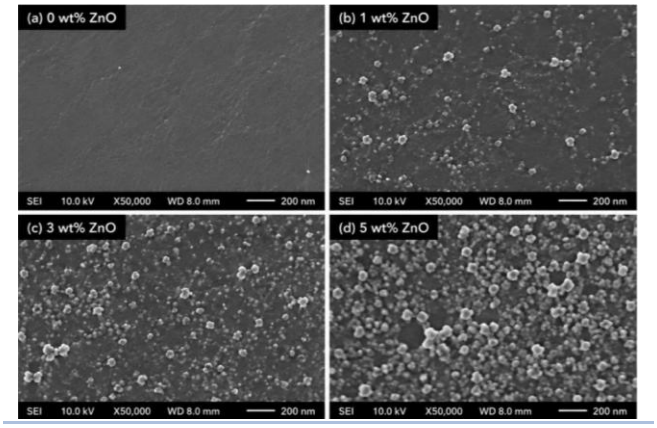


Figure 5. SEM micrographs showing surface morphology and ZnO nanoparticle dispersion within the nanocomposite films.

Mechanical Properties

Tensile testing was used to assess the manufactured nanocomposite films' mechanical properties. The comparative tensile strength behavior is shown in **Figure 6** and the mechanical characteristics found are summarized in **Table 4**.

The findings demonstrate how adding ZnO nanoparticles greatly increased the films' tensile strength, which went from 28.5 MPa for the pure PVA/PEG film to 37.1 MPa for the film with 5 weight % ZnO nanoparticles. Based on previous studies of polymer nanocomposites, this enhancement has been attributed to better stress transfer and better intermolecular interactions between ZnO nanoparticles and polymer matrices[16-19].

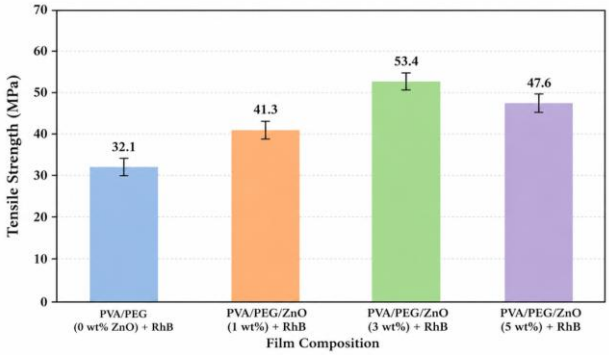


Figure 6. Comparative tensile strength behavior of PVA/PEG/ZnO/RhB nanocomposite films with varying ZnO concentrations.

However, elongation at break decreased slightly with increasing concentrations of ZnO due to the decrease in polymer chain mobility when the nanoparticles were added.

Table 4. Mechanical properties of PVA/PEG/ZnO/RhB nanocomposite films.

Sample	Tensile Strength (MPa)	Elongation at Break (%)
S0	28.5 ± 1.1	102 ± 3
S1	31.2 ± 1.4	97 ± 2
S3	35.6 ± 1.2	91 ± 4
S5	37.1 ± 1.6	85 ± 3

Accelerated Aging Performance

The UV-aging resistance of coated and uncoated paper samples was accelerated to test the protective performance of the fabricated nanocomposite coatings. The data on the whiteness retention behaviour under UV exposure is given in **Figure 7** and the data on the UV exposure aging behaviour is summarized in **Table 5**.

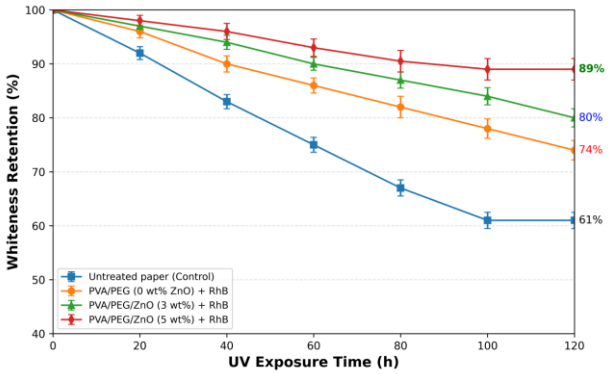


Figure 7. Whiteness retention behavior of coated and uncoated paper samples during accelerated UV aging exposure.

It is observed from **Figure 7** and **Table 5**, that the paper samples without any treatment (blank) showed significant degradation and lost 61% of the whiteness rating after 120 h

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of UV exposure. The ZnO containing coatings, on the other hand, showed a much better aging resistance, where around 89% of the initial whiteness was retained even after aging for the S5 sample[17, 18, 21].

Table 5. Accelerated aging resistance performance of coated and uncoated paper samples under UV exposure.

Sample	Initial Whiteness (%)	Final Whiteness After UV Exposure (%)	Retention (%)
Untreated Paper	100	61	61
S0	100	74	74
S1	100	80	80
S3	100	87	87
S5	100	89	89

The improved preservation behavior is primarily due to the high UV-absorption properties of the ZnO nanoparticles that successfully prevented the degradation of cellulose and UV-induced yellowing. This kind of protection has been reported earlier for protective coatings of ZnO[1, 15].

Fluorescence Sensing Behavior

The fluorescence sensing behavior of Rhodamine B (RhB) incorporated into the PVA/PEG/ZnO nanocomposite films were evaluated during accelerated UV aging. The fluorescence emission spectra are shown in **Figure 8**, and the intensity values in **Table 6**.

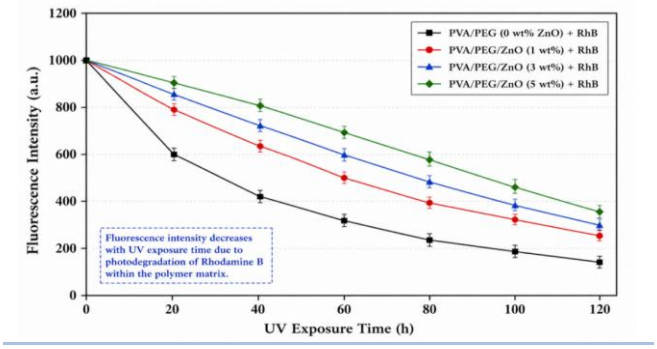


Figure 8. Variation in (RhB) fluorescence intensity of nanocomposite films during accelerated UV exposure.

As can be seen in **Figure 8** and **Table 6**, the fluorescence intensity decreased as function of the UV exposure time and after 120 h of UV exposure, the intensity reached 65 a.u. which is less than before aging. This is a sign of degradation of the polymeric matrix and changes in the local environment of the (RhB) molecules[19, 21].

(RhB) molecules can be subjected to oxidative reactions and bond cleavage in the polymer matrix as a result of extended UV exposure, which causes the microenvironment of the dye molecules to change, and hence reduces the fluorescence intensity. These structural changes create more pathways that dissipate energy in a non-radiative manner leading to the quenching of the emission and decreasing in the intensity of the emission[22, 23] [22-25].

Table 6. Fluorescence intensity response of (RhB) during accelerated UV aging tests.

Exposure Time (h)	Fluorescence Intensity (a.u.)
0	100
24	95
48	89
72	82
96	74
120	65

The fluorescence response observed shows that RhB is a potentially good optical indicator of material degradation. Thus, the resulting nanocomposite coating has protective characteristics and also will provide a practical fluorescence-based sensing mechanism for early detection and monitoring of degradation in artifacts of cultural heritage paper.

Overall, the obtained results demonstrate that the developed PVA/PEG/ZnO/RhB nanocomposite coating provides a multifunctional approach for cultural heritage conservation. Previous conservation studies have focused primarily on strengthening paper substrates using cellulose nanomaterials, silica nanoparticles, or UV-absorbing metal oxides. In contrast, the present coating combines UV shielding, mechanical reinforcement, and optical sensing capabilities within a single system. The observed fluorescence response introduces a smart monitoring function that extends beyond the passive protection offered by conventional conservation coatings, highlighting the potential of the proposed material for next-generation heritage preservation technologies[20-23].

To highlight the novelty and multifunctional performance of the developed coating, a comparison was made between the proposed PVA/PEG/ZnO/RhB nanocomposite and previously reported conservation coating systems. As shown in **Table 7**, most conventional coatings primarily provide protective functions such as mechanical reinforcement or UV shielding, whereas the developed nanocomposite integrates protection and fluorescence-based degradation monitoring within a single platform. This combination offers a significant advantage for cultural heritage conservation by enabling both preservation and real-time condition assessment of paper artifacts[18, 20-24].

Table 7. Fluorescence intensity response of (RhB) during accelerated UV aging tests.

Coating System	UV Protection	Mechanical Reinforcement	Real-Time Monitoring	Transparency	Main Application
PVA-based coatings	Moderate	Moderate	No	High	Paper consolidation
Cellulose nanomaterial coatings	Low–Moderate	High	No	High	Mechanical strengthening of paper
SiO_2 nanoparticle coatings	Moderate	Moderate	No	High	Surface protection and consolidation
ZnO/polymer nanocomposite coatings	High	High	No	Moderate–High	UV shielding and preservation
Smart fluorescent polymer coatings	Moderate	Moderate	Yes	High	Degradation monitoring
Proposed PVA/PEG/ZnO/RhB nanocomposite coating	High	High	Yes	High	Simultaneous protection and fluorescence-based monitoring of paper artifacts

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Developed Practical Applications

The fabricated smart nanocomposite cover can be utilized in archives and museums for the protection of priceless manuscripts and delicate objects made of paper. It is also expected to have great potential for the preservation of ancient Iraqi and Mesopotamian documents due to its non-destructive and protective properties.

The coating can also be applied to the design of smart packaging solutions produced for the sake of archival materials that should be preserved for a long time and be resistant to the environment. The fluorescent property of the nanocomposite allows for its use in optical monitoring systems for long-term monitoring of the condition and ongoing conservation assessment. In addition, the material can be used in the research of non-destructive diagnostic techniques for cultural heritage conservation and restoration.

Conclusion

The multifunctional PVA/PEG/ZnO nanocomposite films doped with Rhodamine B dye for optical tracking and preserving of cultural paper relics were successfully developed. ZnO nanoparticles significantly enhanced mechanical strength and UV-blocking ability while Rhodamine B provided fluorescence-based degradation monitoring. In accelerated aging tests, the developed coating reduced the degradation caused by UV rays and helped to preserve the appearance of paper.

The fluorescence response of Rhodamine B was used to demonstrate the ability to coat as a smart optical sensor for early detection of degradation. The developed nanocomposite system is a promising approach for adopting photonic materials, smart sensing and nanotechnology in cultural heritage protection applications.

Limitations and Recommendations

There are a few limitations in the present study. Thermal characterization (TGA and DSC) was not carried out and thus the thermal stability and thermal degradation behavior of the prepared PVA/PEG/ZnO/RhB nanocomposite films could not be analysed. Furthermore, accelerated aging was restricted to UV aging in the laboratory. More complete thermal analysis, longer aging times, and combined environmental stresses (such as cycles of temperature and humidity, visible light, and atmospheric pollutants) should be examined in future studies to replicate, as closely as possible, the conditions in museums and archives, and to confirm the durability of the coating in the long term. The resulting nanocomposite coating possessed a high transparency, high UV shielding and high fluorescence monitoring, showing good potential for protecting paper-based cultural heritage. Additional testing of real paper historical samples and a combination with portable non-destructive optical monitoring is suggested to facilitate practical conservation applications.

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Statement on Data Availability

The data for this study is available on request from the corresponding author. This published article includes all data generated or analyzed during this study.

Declaration of Competing Interests

The author has no financial, commercial, or personal relationships that could be construed as causing a conflict of interest. The author has no competing interests to declare.

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