



2D Photocatalyst System of Mn_3O_4 : Ag Nanocomposite to the Degradation of Methylene Blue Under Solar Light

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Abstract

Constructing two-dimensional (2D) composites using layered materials is considered a practical approach to achieving high-efficiency photocatalysts. This study aims to prepare manganese oxide nanoparticles (NPs) from the aqueous extract of thyme leaves, characterize them by spectroscopic methods, and bridge the gap by developing an environmentally friendly, photostable coating and evaluating its efficiency in degrading the methylene blue (MB) dye under sunlight, suitable for practical water treatment applications. In this work, a 2D nano-photocatalytic coating of Mn_3O_4 :Ag was developed for the efficient removal of organics and pollutants from water under sunlight. The method involves synthesizing a photocatalytic nanomaterial via green synthesis of a Mn_3O_4 /Ag nanocomposite using thyme leaf extract. The products were characterized using X-ray diffraction, Fourier transform infrared spectroscopy, and scanning electron microscopy. The image of the Mn_3O_4 : Ag nanocomposite showed spherical NPs with extensive aggregation. The average crystal size was calculated to be 23.7 nm using the Debye-Scherrer equation, and the crystals were incorporated into a white, waterproof coating to produce a homogeneous nano-coating. The final product is applied to solid surfaces and dried at room temperature. The resulting layer demonstrates high efficiency in degrading the MB dye, a model pollutant. The system is eco-friendly, cost-effective, and suitable for water treatment applications. The advantage of this work is the use of green synthesis to produce Mn_3O_4 :Ag nanocomposite, which is incorporated into a coating that serves as a photocatalytic layer under sunlight, making it suitable for environmental remediation applications.

Keywords: Mn_3O_4 :Ag Nanocomposite, Photocatalysis, Two-dimensional, Water treatment.

1. Introduction

Water contamination from industrial discharges, including dyes such as MB, continues to threaten ecosystems and human health. Effective treatment and reuse of wastewater are essential for ensuring environmental protection and sustainable development¹. Industrial expansion, especially in textiles, has led to the continuous discharge of hazardous substances into aquatic systems. These pollutants pose significant risks due to their persistence and toxicity². Conventional treatment methods, such as chemical precipitation and biological degradation, often prove ineffective at entirely removing complex organic pollutants³. Various treatment strategies have been adopted to address pollution-related issues. Recent studies have increasingly focused on advanced oxidation processes (AOPs). Among these, photocatalysis stands out for its efficiency, low cost, and eco-friendliness, making it one of the most widely used AOP techniques in water treatment. Semiconductor photocatalysts have attracted significant attention for innovative

physicochemical methods due to their low energy requirements and simplicity^{4,5}. This process relies on the excitation of electrons from the semiconductor's valence band to the conduction band, leaving behind holes. These electron-hole pairs initiate redox reactions that generate reactive oxygen species (ROS), which then degrade organic pollutants^{6,7}. Among various semiconductors, manganese oxide (Mn_3O_4) has received considerable interest due to its favorable redox behavior, mixed valence states (Mn^{2+} : Mn^{3+}), spinel structure, and efficient catalytic activity under light⁸⁻¹⁰. Mn_3O_4 -based photocatalysts encounter a substantial constraint stemming from the swift recombination of photogenerated electron-hole pairs, which markedly diminishes their photocatalytic effectiveness. To resolve this issue, researchers have investigated the incorporation of noble metals, such as silver (Ag), with Mn_3O_4 to enhance charge separation and extend the longevity of photogenerated carriers¹¹⁻¹³. However, using suspended photocatalysts presents practical limitations, including difficulty in recovery, limited reuse, and secondary pollution. To address this, researchers have focused on immobilizing nanocatalysts onto solid substrates via coatings or films, enabling fixed-bed photocatalytic systems¹⁴. Nanostructured coatings offer several advantages, including mechanical stability, chemical resistance, improved durability, and the ability to provide additional functional properties, such as UV protection, antimicrobial effects, and self-cleaning behavior. These features make them ideal for environmental, electronic, and industrial applications¹⁵. Industrial coatings based on organic-inorganic hybrids, particularly those synthesized via sol-gel processes, have emerged as advanced solutions due to their customizable chemical and mechanical properties. These coatings combine hardness, durability, and flexibility¹⁶. Various synthesis routes have been developed to fabricate such nanostructured coatings, ranging from traditional vapor deposition to modern methods such as sol-gel and laser cladding^{17,18}. Despite these advancements, the green synthesis and immobilization of Mn_3O_4 :Ag nanocomposites in water-repellent coatings remain underexplored. This study aims to prepare manganese oxide nanoparticles (NPs) from the aqueous extract of thyme leaves and characterize them by spectroscopic methods. The study also aimed to bridge the gap by developing an environmentally friendly, photostable coating and evaluating its efficiency in degrading the methylene blue (MB) dye under sunlight, suitable for practical water treatment applications.

2. Materials and Methods

Silver nitrate (AgNO_3) was procured from Merck (Germany), manganese (II) sulfate monohydrate ($\text{MnSO}_4 \cdot \text{H}_2\text{O}$) from Qualikems (India), deionized water (DI water), and *Thymus vulgaris* leaves. The rubber coating was commercially obtained from a local supplier. A plastic container with dimensions of (20 x 20) cm was used as the coating bath.

2.1. Preparation of the photocatalytic coating

The Mn_3O_4 :Ag nano composite used in this study was synthesized by mixing two individually prepared silver NPs (Ag NPs) and manganese oxide NPs. Both NPs were green-synthesized using plant extract. The selected weight ratio of Mn_3O_4 to Ag was 1:0.5, ensuring uniform dispersion and optimal integration of both components. Each NP was dispersed in deionized water, and the resulting suspensions were combined and ultrasonicated. The green synthesis of Mn_3O_4 :Ag nanocoating for photocatalytic applications is shown in **Figure 1**. The mixture was subjected to thermal condensation and then dried at 80°C. To prepare the photocatalytic coating, different amounts of the three (1%, 2%, and 4% w/w) were manually blended with a liquid white dye to form a solid-in-liquid dispersion. The mixture was stirred by hand, and the resulting formulations were applied directly to the inner surface of a clean plastic basin using a brush, forming a thin, even film.

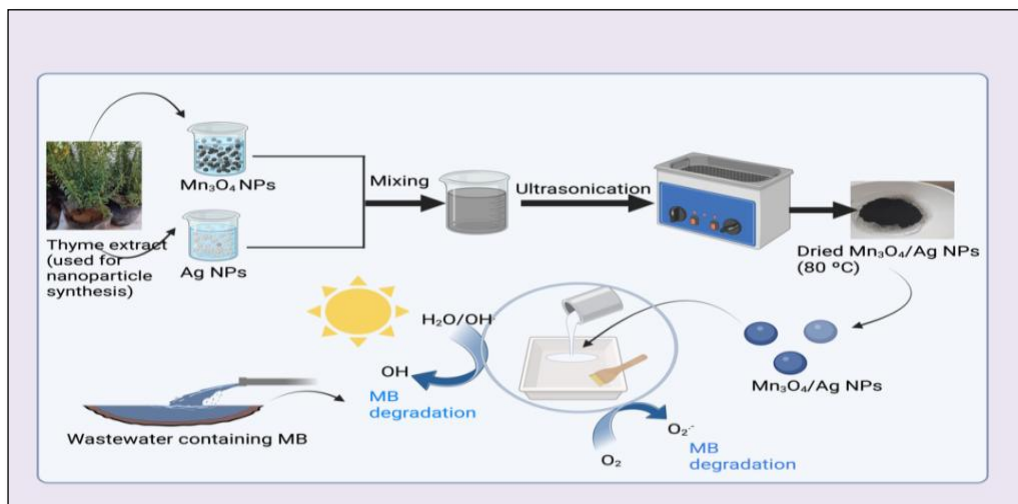


Figure 1. Green synthesis of $\text{Mn}_3\text{O}_4\text{:Ag}$ nanocoating for photocatalytic application.

After coating, it was left to dry at room temperature overnight to allow complete evaporation of the liquid phase. The thoroughly dried coating was then used for photocatalytic degradation experiments under sunlight¹⁹.

2.1.1. Photocatalytic degradation procedure

The photocatalytic efficiency of the coated plastic basin was evaluated by the degradation of a white dye under natural sunlight. A stock solution of MB was prepared at 1000 ppm by dissolving the required amount of dye powder in deionized water. From this stock, a 200 ppm solution was prepared by diluting 40 mL of the stock with 160 mL of deionized water to a final volume of 200 mL. The nanocomposite was incorporated into a waterproof, white, rubber-based dye, which was applied to the inner surface of the plastic basin using a brush. The coating was left to dry overnight at room temperature, forming a water-resistant photocatalytic film. The system was exposed to direct sunlight for 72 hours. Aliquots were withdrawn and analyzed using a UV-Vis spectrophotometer. The maximum absorption wavelength (λ_{max}) of MB was determined to be 664 nm, and all measurements were performed at this wavelength to monitor degradation²⁰. The degradation efficiency (R%) was calculated using the following **Equation 1**:

$$R\% = (c_0 - c_t / c_0) \times 100 \quad (1)$$

Where: C_0 is the initial dye concentration, and C_t is the dye concentration at time

Based on the Beer–Lambert Law, which assumes a linear correlation between absorbance and concentration, C_t values were estimated from absorbance readings as **Equation 2**:

$$R\% = (A_0 - A_t / A_0) \times 100 \quad (2)$$

Where A_0 and A_t are the absorbance values at the initial time and time t , respectively, the average solar irradiance recorded during the exposure period was 900 W/m^2 .

2.1.2. Product characterization

The compounds were synthesized and characterized utilizing diverse spectroscopic and microscopic methodologies and apparatus, including a magnetic stirrer, a PC21-A electric oven, a Batec, and a PLC (80-1) electric centrifuge. A sensitive electronic balance (AS 220C1) was utilized for accurate measurements. X-ray diffraction (XRD) investigation was conducted utilizing a Phillips PW1730 from the Netherlands. The FT-IR spectra were acquired using a Shimadzu 1800 (Japan) over $400\text{--}4000\text{ cm}^{-1}$, and field-emission scanning electron microscopy (FESEM) was performed using a MIRA II (Czech Republic). The colors of the treated and untreated samples were determined using a ColorFlex spectrophotometer in accordance with ASTM E313-10. The samples with thick plaques (2 mm) and flattened surfaces were placed in the specimen measuring port, and L^* was monitored.

3. Results

3.1. FTIR spectrum characterization

The FTIR spectrum that is shown in **Figure 2** exhibited Mn–O stretching bands at (500, 700, and 915) cm^{-1} , indicative of Mn–O stretching vibrations. The spectrum appeared bands at (3441, 3194, 2936, 1734, and 1652) cm^{-1} .

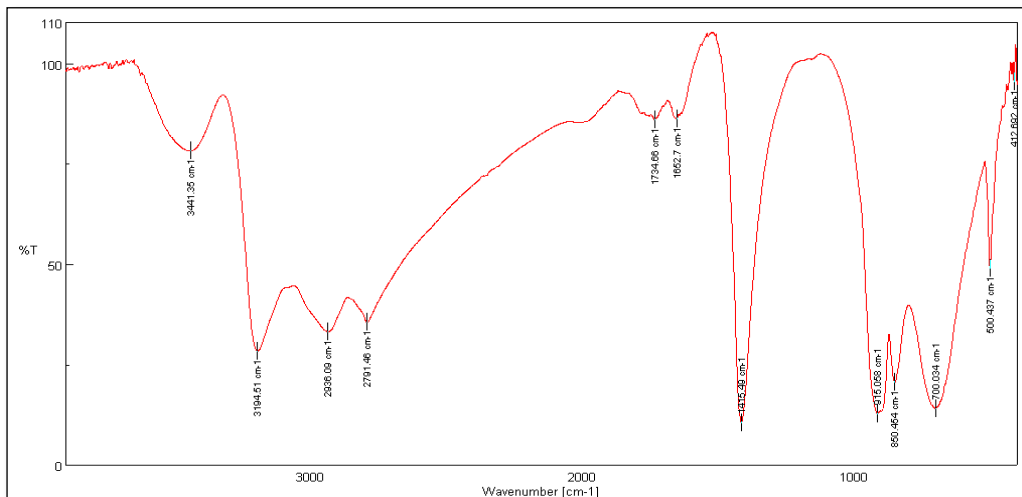


Figure 2. FTIR spectrum of the $\text{Mn}_3\text{O}_4:\text{Ag}$ nanocomposite

3.2. XRD pattern of the produced $\text{Mn}_3\text{O}_4/\text{Ag}$ nanocomposite

The XRD pattern of the produced $\text{Mn}_3\text{O}_4:\text{Ag}$ nanocomposite is presented in **Figure 3** and **Table 1**. The diffractogram shows distinct peaks at $2\theta = 33.4^\circ, 38.6^\circ, 44.7^\circ, 55.6^\circ, 64.9^\circ,$ and 77.8° .

Table 1. XRD data of the $\text{Mn}_3\text{O}_4:\text{Ag}$ nanocomposite.

No.	$2\theta^\circ$	hkl	FWHM (degree)	$2\theta^\circ$ (Rad.)	FWHM (Rad)	D (nm)
1	33.4	320	0.2362	0.291558	0.004	35.108
2	38.6	111	0.3306	0.336437	0.006	25.445
3	44.7	200	0.6612	0.390448	0.012	12.987
4	55.6	051	0.3306	0.48505	0.006	27.151
5	64.9	220	0.4723	0.565935	0.008	19.919
6	77.8	220	0.4723	0.678907	0.008	21.604

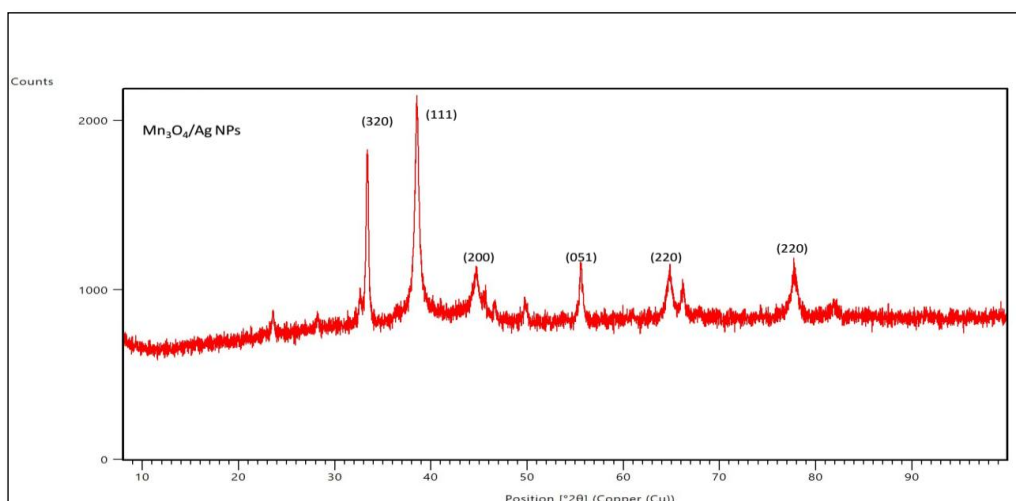


Figure 3. XRD picture of the $\text{Mn}_3\text{O}_4:\text{Ag}$ nanocomposite

3.3. FESEM of the $Mn_3O_4:Ag$

FESEM picture of the $Mn_3O_4:Ag$ nanocomposite, **Figure 4** illustrating quasi-spherical NPs with significant aggregation. The particle sizes ranged from 30 to 80 nm, confirming the material's nanoscale nature.

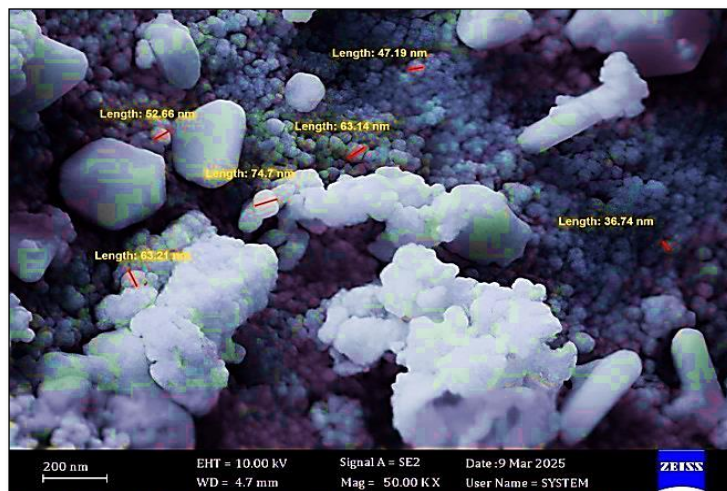


Figure 4. FESEM pictures of the $Mn_3O_4:Ag$ nanocomposite

3.4. Photocatalytic degradation of MB

Photocatalytic degradation behavior of MB under sunlight in the presence of $Mn_3O_4:Ag$ -based coatings with different catalyst loadings (1%, 2%, and 4% w/w), as shown in **Figure 5**.

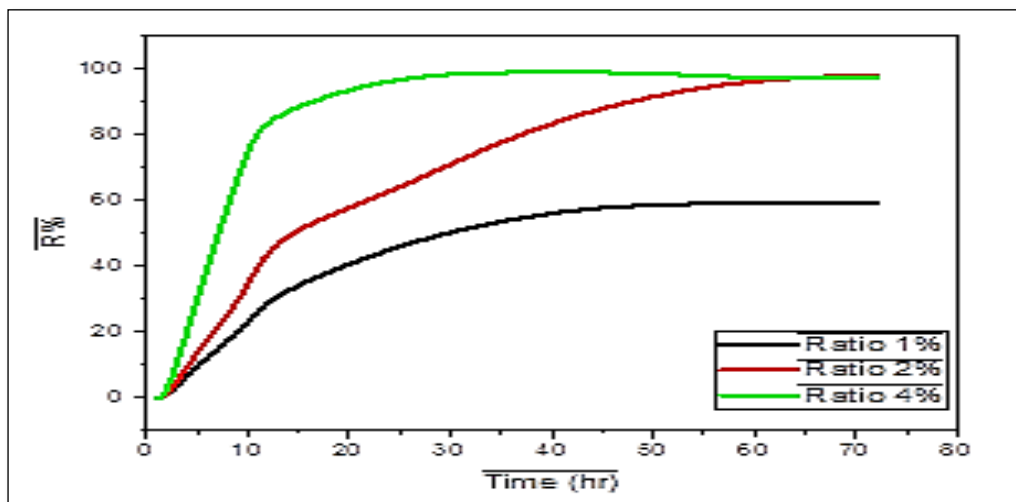


Figure 5. The R% of MB using coated films with $Mn_3O_4:Ag$ -coated films (1, 2, and 4)%

The absorbance of the dye solution gradually decreased with increasing exposure time, indicating continuous degradation. The 2% $Mn_3O_4:Ag$ coating demonstrated the most efficient dye removal, with the absorbance curve showing a sharp decline and stabilizing after 72 hours. The 4% coating presented a high performance, although slightly lower than the 2%, while the 1% formulation exhibited the slowest and least effective degradation.

3.5. Colorimetric evaluation

Colorimetric analysis was conducted to evaluate the lightness (L^*) of the $Mn_3O_4:Ag$ -based coatings before sunlight exposure. As shown by the measurements, the 1% formulation exhibited the highest L^* value (85.49), indicating a closer match to the original white dye. Increasing the catalyst concentration led to darker initial coatings, with L^* values of 73.58 and 70.74 for 2% and 4% catalyst loadings, respectively. These results suggest that higher nanocomposite content

reduces the coating's optical brightness at the outset and present the complete colorimetric data for the coated samples before sunlight exposure. While various parameters were measured (such as a^* , b^* , and ΔE), the current analysis focuses solely on L^* , which indicates the surface's visual lightness. The corresponding visual color distributions are shown in **Figure 6**, highlighting differences in sample appearance across loadings.

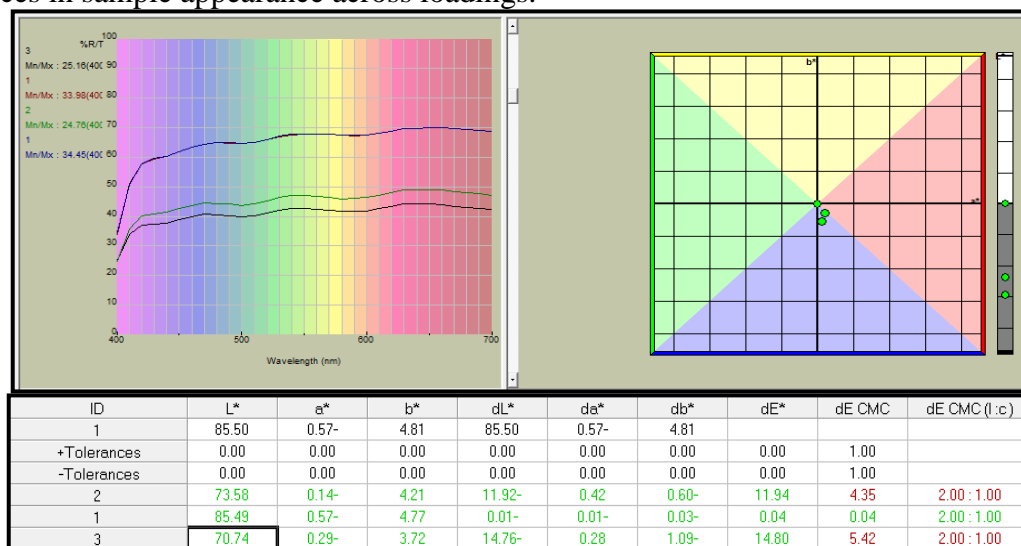


Figure 6. Color space representation and spectral reflectance curve of $Mn_3O_4:Ag$ -coated samples

4. Discussion

4.1. FTIR spectrum

The FTIR spectrum in **Figure 2** shows bands at 500, 700, and 915 cm^{-1} , indicative of Mn–O stretching vibrations, confirming the formation of Mn_3O_4 . The other bands, which appear at (3441, 3194, 2936, 1734, and 1652) cm^{-1} , refer to stretching vibration of O–H, N–H, C–H aliphatic groups, C=O, and C=C, respectively, originating from residual phytochemicals present in plant extract used during green synthesis²⁰⁻²².

4.2. XRD pattern

The observed peaks in **Figure 3** and **Table 1** correspond to the crystallographic planes (hkl) of Mn_3O_4 and Ag phases, consistent with JCPDS card no. 01-086-2337 and JCPDS no. 01-087-0719, respectively. The pronounced peak at 38.6°, associated with the (111) plane, verifies the existence of face-centered cubic silver NPs²³. The crystallite size was determined using the Debye–Scherrer equation ($D = K\lambda/\beta\cos\theta$). The average crystallite size measured 23.7 nm, affirming the nanoscale architecture of the produced material. The absence of any additional distinct peaks indicates that the synthesized $Mn_3O_4:Ag$ nanocomposite is highly crystalline, confirming the structural purity of the material. These characteristic peaks validate the successful synthesis of the nanocomposite²⁰.

4.3. FESEM

This partial agglomeration is frequently reported in green-synthesized nanomaterials. This agglomeration is due to two factors, primarily attributed to the high surface energy of the nanoscale particles, which drives them to coalesce to minimize surface tension. Additionally, the absence of a chemical stabilizer in the green synthesis method reduces electrostatic repulsion between particles, further promoting aggregation. Nevertheless, this structural clustering does not compromise the crystalline purity of the nanocomposite, as confirmed by the XRD and FTIR analyses. The particle surfaces were not completely smooth; instead, they exhibited nanoscale imperfections and tiny granular features, as shown in **Figure 4**. Surface asperities can increase the effective surface area and facilitate interactions with surrounding media, which is advantageous in surface-dependent applications, including photocatalysis²².

4.4. Photocatalytic degradation of MB

The results suggest that increasing the catalyst loading improves the photocatalytic activity up to an optimal point. The 2% loading achieved the highest degradation efficiency (98% within 72 hours), indicating sufficient surface area and active sites without excessive particle agglomeration. In contrast, the 1% catalyst may have had insufficient active centers, while the 4% catalyst may have led to excess coverage or light scattering, slightly reducing performance. Therefore, 2% catalyst loading provides the most balanced and effective degradation behavior under sunlight irradiation²², as shown in **Figure 5**.

4.5. Colorimetric evaluation of Mn₃O₄:Ag NPs

The observed decrease in L* values with increasing catalyst concentration suggests that the Mn₃O₄:Ag nanocomposite layer becomes optically darker at higher loadings. This may be due to greater surface coverage, higher particle density, or increased light absorption caused by the dense nanostructure. A lower L* value at 2% and 4% loading could also indicate enhanced light interaction, which may benefit photocatalytic efficiency. Therefore, the colorimetric differences reflect structural and optical changes in the coating as a function of catalyst concentration. Optimizing catalyst loading is essential for attaining a functioning photocatalytic coating that provides both high degrading efficiency and satisfactory visual characteristics. The 1% Mn₃O₄:Ag coating demonstrated the highest lightness value (L* = 85.49), signifying negligible visible disruption to the original white dye. Nonetheless, it yielded the lowest photocatalytic efficiency (59%). The 2% formulation demonstrated exceptional photocatalytic performance (98%) and sustained a relatively high L* value (73.58), indicating a favorable equilibrium between catalytic efficacy and surface aesthetics. In contrast, the 4% loading attained a similar deterioration (97%) but exhibited increased darkening (L* = 70.74), potentially diminishing its visual appeal. The 2% catalyst loading was identified as the best composition, yielding effective dye degradation and satisfactory visual stability, thereby positioning it as a viable contender for practical applications²³.

5. Conclusion

This study demonstrated that Mn₃O₄:Ag-based photocatalytic coatings prepared with a rubber-based white dye were effective in degrading MB under sunlight. Including the tested formulations, the 2% catalyst loading achieved the highest degradation efficiency (98%) within 72 hours while also maintaining acceptable visual properties (L* = 73.58). Although the 1% composition exhibited better lightness, it showed limited photocatalytic performance. Therefore, the 2% formulation was identified as the optimal balance between functionality and appearance, making it a promising candidate for practical photocatalytic applications involving surface coatings. A distinctive green-synthesized Mn₃O₄/Ag nanocomposite is presented for solar-driven degradation of organic dyes, addressing a direction that remains underexplored in the current literature.

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Conflict of Interest

The authors declare that they have no conflicts of interest.

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