

# **Photocatalysis of Ag-loaded MgTiO<sub>3</sub> for degradation of Fuchsin dye and dyes present in textile wastewater under sunlight**

*Dissertation submitted in partial fulfillment of  
the requirements for the award of degree of*

**Master of Science  
in Chemistry**

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## DECLARATION

I hereby declare that the dissertation, “**Photocatalysis of Ag-loaded MgTiO<sub>3</sub> for degradation of Fuchsin dye and dyes present in textile wastewater under sunlight,**” submitted to the Department of Chemistry and Biochemistry, Thapar Institute of Engineering and Technology, Patiala, in partial fulfillment of the requirements for the award of a Master of Science in Chemistry, is a record of my work that I conducted from January 2024 to July 2024 under the supervision of Dr. Bonamali Pal and Dr. Satnam Singh. The results contained in this dissertation have not been submitted in part or full to any other university or institute for the award of any other degree or diploma.

Date: 30-7-2024

Place: Patiala

  
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## CERTIFICATE

This is to certify that the dissertation entitled “**Photocatalysis of Ag-loaded MgTiO<sub>3</sub> for degradation of Fuchsin dye and dyes present in textile wastewater under sunlight**” being submitted by **Vishal Tiwari** to the **Department of Chemistry and Biochemistry, Thapar Institute of Engineering and Technology, Patiala**, in partial fulfillment of the requirements for the award of degree of the **Master of Science in Chemistry**, is an authentic record of the work carried out by the candidate under our guidance and supervision. He has fulfilled the requirements for the submission of this dissertation, which, to our knowledge, has reached the requisite standard.

The results embodied in the dissertation have not been submitted in part or full to any other university or institute for the award of any other degree or diploma.

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## ABSTRACT

Our research focuses on the fabrication of MgTiO<sub>3</sub> by a sonochemical approach and improving its photocatalytic characteristics by loading it with Ag metal. This change allows the photocatalyst to efficiently absorb broad-spectrum solar light while also effectively separating charges, which are critical for photocatalysis. Both MgTiO<sub>3</sub> and Ag-MgTiO<sub>3</sub> were discovered to exhibit spherical shapes, with Ag showing as smaller spheres and MgTiO<sub>3</sub> as bigger ones. The Ag-MgTiO<sub>3</sub> photocatalyst effectively degraded Fuchsin dye (Fuc) under solar light exposure, achieving a rate constant of 0.0314 min<sup>-1</sup> over 50 minutes. Comparable tests with visible and UV light produced lower Fuc breakdown rates of 87% and 62.3%, respectively, highlighting direct sunlight as our catalyst's most effective light source (99.2%). Even at low concentrations (0.3 g/L), Ag-MgTiO<sub>3</sub> demonstrated excellent efficiency. Its photocatalytic activity substantially outperformed typical market options for TiO<sub>2</sub>-P25 powder, which only achieved 42% removal of fuc dye. The catalyst had a zero charge surface pH of 4.46 and achieved maximum Fuc decomposition efficiency at pH 7. Reusability experiments showed the catalyst's high stability, with an 81.8% Fuc degradation efficiency after five repetitions. Scavenger studies emphasized the crucial role of O<sub>2</sub><sup>•-</sup> active species in the photodecomposition process, including DMSO, a marked alteration lowering Fuc degradation effectiveness to 37.5 percent. Additionally, Ag-MgTiO<sub>3</sub> showed a remarkable 67.8% reduction in total organic carbon (TOC) for Fuc Dye. HRMS demonstrated the formation of simpler intermediates during contaminant degradation. Ag-MgTiO<sub>3</sub>, with its simple synthesis method and outstanding performance, is a promising alternative for eliminating persistent pollutants in textile wastewater systems.

**Keywords:** Ag loaded MgTiO<sub>3</sub> photocatalyst; Photocatalytic activity of Ag-MgTiO<sub>3</sub>; Photodegradation of Fuchsin dye; Textile dye degradation; Visible/Sunlight irradiation

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## LIST OF ABBREVIATIONS

**Fuc:** Fuchsin dye

**DI:** distilled water

**wt%:** weight percentage

**NCs:** nanocomposites

**VB:** valence band

**CB:** conduction band

**Eg:** band gap

**LSPR:** Localized surface plasmon resonance

**XRD:** X-ray diffraction

**FE-SEM:** Field Emission scanning electron microscope

**EDS:** Energy-dispersive X-ray spectroscopy

**DRS:** UV-visible diffuse reflectance spectroscopy

**PL:** photo-luminescence

**XPS:** X-ray photoelectron spectroscopy

**TOC:** Total Organic Carbon

**HR-TEM:** High-resolution Transmission Electron Microscopy micrographs

## LIST OF SYMBOLS

**°C:** degree Celsius

**%:** percentage

**k:** rate constant

**a.u.:** arbitrary unit

**rpm:** revolution per minute

**eV:** electron volt

**mg:** milligram

# 1. INTRODUCTION AND LITERATURE REVIEW

## 1.1 Introduction

The expanding demands on the world's energy supply and the damaging consequences of environmental contamination have emerged as humanity's top concerns.[1,2] As industrialization progresses, pollutants like pesticides, heavy metals, organic compounds, synthetic dyes, pharmaceutical residues, and other dangerous chemicals are being released at an unprecedented rate, seriously contaminating water supplies.[3–5] An astounding 43,000 tons of organic colorant are fabricated annually, and unfortunately, over 10% of this quantity ends up in our priceless water bodies [6]. Synthetic dyes, which we frequently use daily, significantly contribute to environmental contamination. Azo dyes are among the most dangerous types of artificial colors. Its discharge through textile industry wastewater must be taken severely because of its rich chemical composition, which includes amino groups, aromatic rings, and azoic linkages. As a result, they are potent pollutants in the aquatic environment. Specifically, the synthetic dyes released into the effluent exhibit a high degree of resistance to the usual water remediation methods, including chemical remediation, adsorption, membrane filtration, electrolytic removal, ozone processing, and other methods [7,8].

Semiconductor-based photocatalysis is one of the most enticing modern oxidation methods for cleaning wastewater. Its advantages include its simplicity, effectiveness, and environmental friendliness. In essence, energy-rich photons are absorbed, equivalent to or greater than the semiconductor's band gap, to create electron-hole pairs, which subsequently combine with hydroxyl and oxygen species to produce active ions capable of swiftly and efficiently breaking down the pollutants in the water [9,10]. Numerous semiconductor technologies and their composite materials have been designed and explored in great detail thus far in this area. Perovskite oxides in the form of  $ABO_3$  are among the many catalytic materials that have been studied to date, and they are attracting much interest due to their enormous potential in various applications [11,12]. Perovskite oxides have been the subject of extensive research owing to their exceptional physical-chemical and optoelectronic features, encompassing great heat-related and photo-corrosion durability, electron conductivity, economical manufacturing expense, and structural flexibility [13].

Under sunlight irradiation, photocatalysis breaks down organic contaminants into simpler substances like water ( $H_2O$ ) and carbon dioxide ( $CO_2$ ) [14]. Numerous semiconductor materials have been investigated for their potential as a viable photocatalyst for eliminating ecological contaminants in

the presence of sunlight. Among them, one effective photocatalyst is  $\text{MgTiO}_3$ , a perovskite metal oxide. The layered structure of  $\text{MgTiO}_3$  allows for improved charge separation, and it also has strong chemical and thermal stability, producing a high level of photocatalysis [15]. Apart from its photocatalytic function, this substance has wide-ranging uses in communication antennas and global position systems operating on microwave radiation [16,17].

To synthesize  $\text{MgTiO}_3$  nanoparticles, several techniques have been used, including sol-gel, co-precipitation, metal-organic, solid-state processes, and hydrothermal mechano-chemical complexation approaches [18–20]. In general, two phases make up  $\text{MgTiO}_3$ :  $\text{MgTi}_2\text{O}_5$  and  $\text{Mg}_2\text{TiO}_4$ . The current investigation utilized the sonochemical approach to successfully manufacture  $\text{MgTiO}_3$  nanoparticles without needing these two phases. The use of ultrasound in research has grown recently due to the peculiar qualities of the product created by the sonochemical process, which set it apart from other methods. This is because tiny particles with a large surface area are created using ultrasound. In addition, this process produces nanoparticles and may react quickly with high purity and adjustable reaction conditions [21,22].

The effectiveness of the synthesized nanocomposite was next evaluated concerning the degradation of a colored pollutant (fuchsin dye) and wastewater that we took from a textile dyer in Model Town, Patiala, using solar light. A thorough series of tests were carried out, including the refinement of metal-loaded  $\text{MgTiO}_3$  concentration, reaction kinetic analysis, pH influence, recyclability assessment, and active species detection tests.

Plasmonic metal nanoparticles (Ag, Au, Cu, Pt) can potentially improve the photocatalytic efficiency of SCs. Plasmonic metal nanoparticles (NPs) have exceptional localized surface plasmon resonance (LSPR) properties that enhance the spectral behavior of the visible region by creating excited electrons, producing more radicals, and increasing the separation of charge carriers [23]. Furthermore, due to a Schottky barrier at the M-SC junction, these metal nanoparticles can operate as electron trap sites, catching the high-energy electrons flowing from the  $\text{MgTiO}_3$  Conduction Band. This significantly speeds up the separation of electron-hole pairs, contributing to the enhancement [24]. A. Shawky et al. produced Pt-decorated  $\text{CaTiO}_3$  nanocrystals with different Pt loadings and tested their photoreduction of nitrobenzene to aniline. Pt deposition boosted the conversion rate significantly [25]. Zhen et al. fabricated  $\text{Ag}/\alpha\text{-MoO}_3$  heterojunctions that facilitate the removal of thiophene by nearly 2.5 times compared to bare  $\alpha\text{-MoO}_3$  [26]. Similarly,  $\text{Au}/\text{ZnTiO}_3$  nanocomposites demonstrated an incredible increase in the  $\text{H}_2$  evolution rate under UV and visible light. Silver has been identified as a leading candidate for producing plasmonic photocatalytic materials because of

its minimal cost, excellent electrical conductance, and significant Localized surface plasmon resonance effect [27].

Numerous observations in the literature indicate that metal deposition significantly enhances the photocatalytic activity of the material when exposed to visible illumination. Thus, decorating MgTiO<sub>3</sub> surfaces with metals enhances their photocatalytic activity.

The report examines the photocatalytic performance of Ag-modified MgTiO<sub>3</sub> nanocomposites (containing 1 wt% Ag). The sonochemical method was used to produce the photocatalyst MgTiO<sub>3</sub>, and subsequently, Ag was deposited on it using the photo-deposition process. Due to its availability, low cost, and acceptable redox potential (Ag = 0.799 V) to the conduction band of MgTiO<sub>3</sub>, The synthesized sample was then characterized using various approaches and assessed for photocatalytic performance by removing fuchsin dye under sunlight. Individual studies examined the impact of Ag loading on light absorption and photocatalytic activity.

## 1.2 Objectives

- 1) Preparation and characterization of MgTiO<sub>3</sub> and the photo deposition of noble metal Ag on it.
- 2) To study the various physiochemical properties of Ag-modified MgTiO<sub>3</sub> nanocomposites.
- 3) To study the photocatalytic degradation of fuchsine dye and dyes in textile wastewater by Ag-MgTiO<sub>3</sub> nanocomposites under visible illumination and solar light.

## 2. EXPERIMENTAL SECTION

### 2.1. Apparatus

Measuring cylinders, Beakers, Conical flask, volumetric flask, Magnetic Stirrer, Glass Vials, Droppers, Petri dishes, Spatula, Magnetic beads, Test tubes, thermometer, crucible, weighing balance, centrifugation tubes, and pH paper were used.

### 2.2 Chemicals and Reagents

Potassium Hydroxide Pellets (85%), Nitric Acid (69%), isopropyl alcohol (98%), Magnesium nitrate hexahydrate (98%), ethanol (C<sub>2</sub>H<sub>5</sub>OH, 99.9%) were purchased from Loba Chemie, india. Titanium dioxide (P25-TiO<sub>2</sub>) was obtained from Degussa Corporation. Silver nitrate (AgNO<sub>3</sub>) was purchased from Sigma-Aldrich (India), and Fuchsin dye (Pulver) was bought from MERCK DARMSTADT, Germany. Double distilled water from Organo Biotech Laboratories Pvt. Ltd.

## 2.3 Synthesis of MgTiO<sub>3</sub> nanocomposite

**Scheme 1** depicts the flowchart that illustrates the experimental procedure of the preparation method. After being dissolved in 60 ml of 1M nitric acid, 1g of TiO<sub>2</sub> was subjected to a 15-minute sonication. Concurrently, a Mg(NO<sub>3</sub>)<sub>2</sub> solution was made, which involved dissolving 2.5 g of Mg(NO<sub>3</sub>)<sub>2</sub>· 6H<sub>2</sub>O in 10 mL of double-deionized water, mixing it with the sonicated solution and then sonicating it for an additional 15 minutes. Following sonication, a further two hours of sonication were performed after adding 3M KOH solution using a dropper to get the pH down to 12. Following the reaction's conclusion, the precipitate was recovered using centrifugation, repeatedly cleaned with double-deionized water to remove contaminants, and rinsed with ethanol. The final product was obtained by drying the precipitate at 100 °C for 4 hours and calcining it for 2 hours at 700 °C after washing.

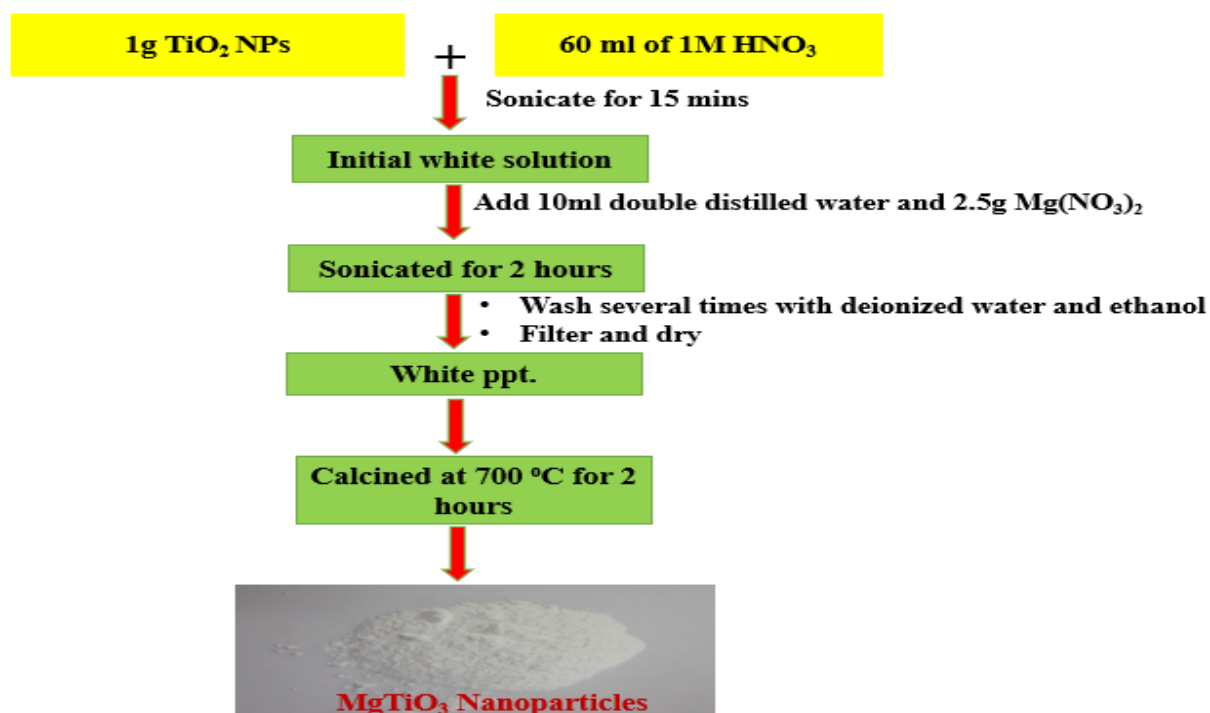
## 2.4 Synthesis of Ag-modified MgTiO<sub>3</sub>

**Scheme 2** shows the technique used to carry out the photo deposition of Ag on the MgTiO<sub>3</sub> surface. In separate test tubes with deionized water and isopropyl alcohol as a hole absorber (5 ml each), 100 mg of the produced MgTiO<sub>3</sub> powder was suspended. The necessary quantity of metal salt AgNO<sub>3</sub> (0.01M; 928-4636μL) corresponding to the required weight percentage (1 wt%) was added to these suspensions. Test tubes were securely sealed with a rubber septum following a 20-minute argon (Ar) gas purge to produce an inert atmosphere. The test tube contents were continuously stirred by magnetic means in a photochemical reactor for three hours while being revealed to UV illumination (125 W, 10.3 mW/cm<sup>2</sup>). The metal-deposited solutions were then subjected to centrifugation at 8500 revolutions per minute, Frequently cleaned with ethanol and deionized water, followed by drying for two hours at 70 °C in an oven. The obtained Ag-deposited nanocomposites have the following names: 1, 2, and 3 weight percent Ag-MgTiO<sub>3</sub>.

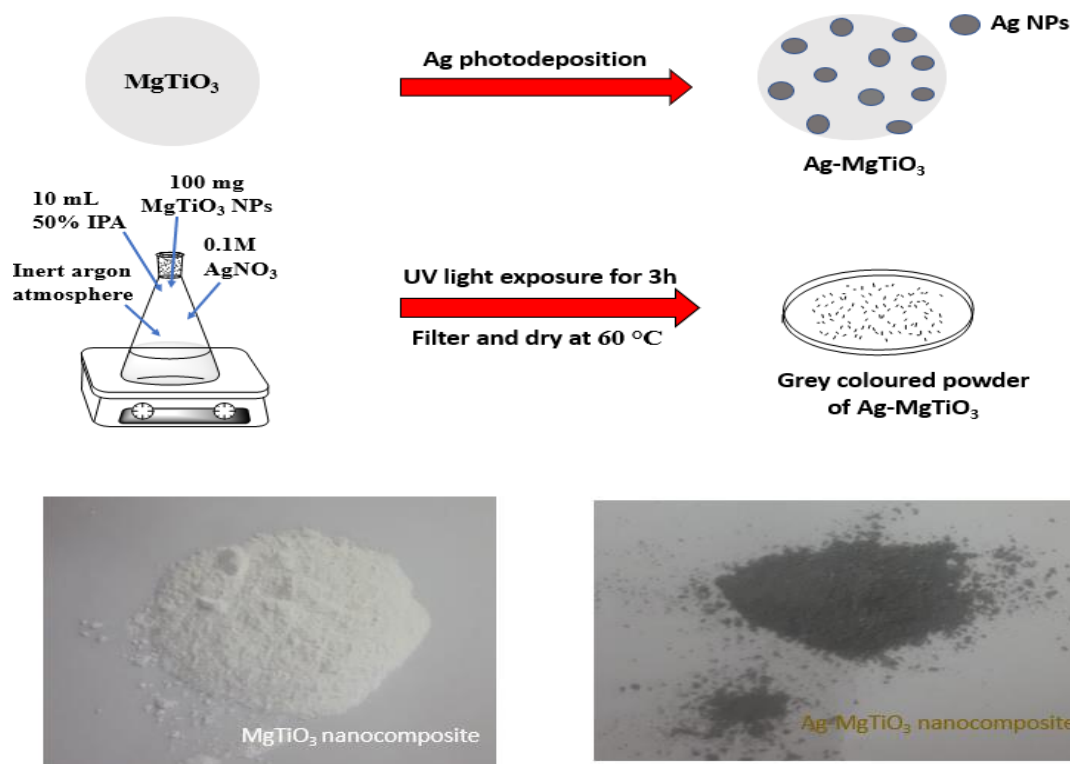
## 2.5. Characterization techniques

The produced samples were subjected to crystallographic examinations using an X-ray powder diffraction Panalytical-Xpert-PRO machine that was outfitted with a Cu-K $\alpha$  radiation source (1.54 Å) that operated at 45 kV and a diffraction angle (2 $\theta$ ) that ranged from 10° to 80° at a 5°/min scan rate. Using a spectrofluorometer (Perkin-Elmer LS55), the photoluminescence (PL) emission spectrum was measured to investigate the separation of photogenerated electron-hole pairs. Both high-resolution transmission electron microscopy (HRTEM TALOS F200S G2 model running at 200 kV voltage) and scanning electron microscopy (SEM; JEOL JSM-7600F operated at 30 kV) were

used to analyze the materials' size and shape. The elemental composition ratio was analyzed using energy-dispersive spectroscopy (SEM-EDS, JEOL JSM-7600 F) at 30 kV.



**Scheme-1.** Flow chart of synthesis of MgTiO<sub>3</sub> nanoparticles



**Scheme 2:** Experimental procedure for photo-deposition of Ag nanoparticles onto MgTiO<sub>3</sub>.

The catalyst's oxidation status was investigated using X-ray photoelectron spectroscopy (XPS, KRATOS axis 165 Shimadzu) with Mg-K $\alpha$  radiations at 75 W. The optical absorption properties of powder solid samples were explored using an Avantes DRS instrument and a UV-vis spectrophotometer (JASCO, V-750), over a spectral range of 200-800 nm, with BaSO<sub>4</sub> as a reference standard.

## 2.6. Photocatalytic degradation of organic waste

The photocatalytic efficiency of the produced materials (MgTiO<sub>3</sub> and silver-modified MgTiO<sub>3</sub>) was tested using Fuchsin (Fuc) dye Deterioration. The photocatalytic process was carried out under direct sunlight. 3 mg of photocatalyst was dispersed into 10 mL of Fuc solution (10 ppm) at a 0.3 g/L concentration. Before irradiation, the suspensions were agitated in the absence of light for 40 minutes to establish an Equilibrium of adsorption and desorption processes between the dye molecules and the catalyst's surface. To initiate deterioration, the solution was stirred for 110 minutes with the vessel horizontal and exposed to direct sunlight. After removing the MgTiO<sub>3</sub> catalyst, roughly 5–6 mL of the solution was taken out and examined with a UV-visible spectrophotometer at intervals. The photocatalysis studies occurred at the Thapar University in Patiala, India, in a subtropical region. The study spanned from April 17 to May 15, 2024. Using a LICOR pyranometer, the median sunlight exposure intensity was determined to be 845 W/m<sup>2</sup>. The solution was exposed to visible illumination from a 45 W CFL lamp (Philips) at a brightness of approximately 100 W/m<sup>2</sup>. UV light was applied to the solution using a 100 W Hg lamp at 365 nm with a flux density of 64–66 W/m<sup>2</sup>. Each experiment was performed four times, and graphical representations were created with error margins representing a 4% data source error. The absorbance spectrum of Fuc was measured at frequent intervals, specifically at  $\lambda_{\text{max}} = 547$  nm. The photodegradation efficiency was computed using the formula below:

$$\text{Deterioration effectiveness (\%)} = \{(C_0 - C_t)/C_0\} \times 100 \quad (1)$$

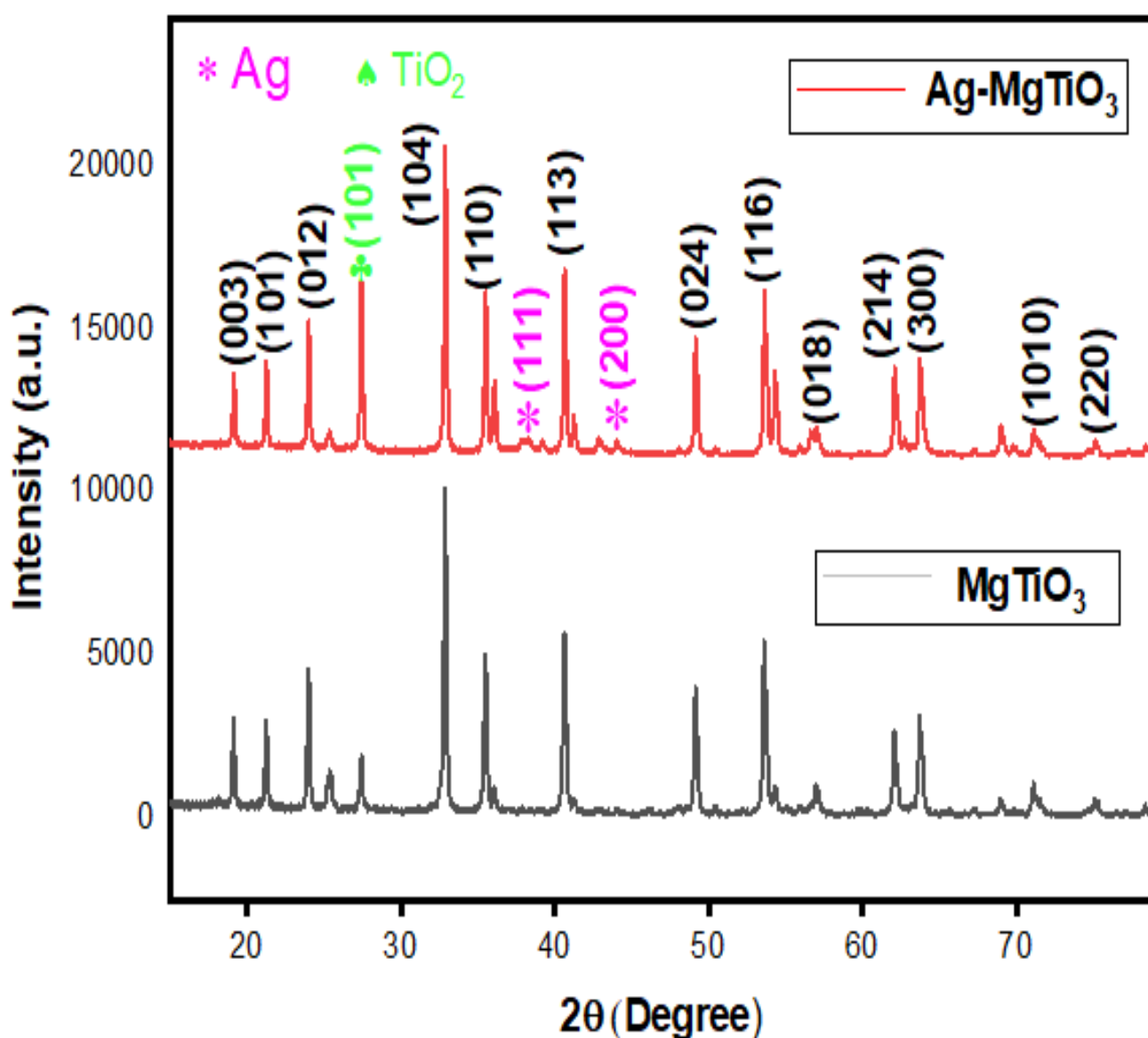
Here,  $C_0$  denotes the initial concentration of Fuc prior to light exposure at time '0', and  $C_t$  is the concentration of Fuc following light exposure at the time 't.'

## 3: RESULTS AND DISCUSSION

### 3.1 Surface structural studies:

**XRD (X-ray diffraction) evaluation:** XRD analysis was employed to forecast the prepared samples' crystal structure and phase composition. Figure 1 depicts the XRD profiles of MgTiO<sub>3</sub> and Silver-

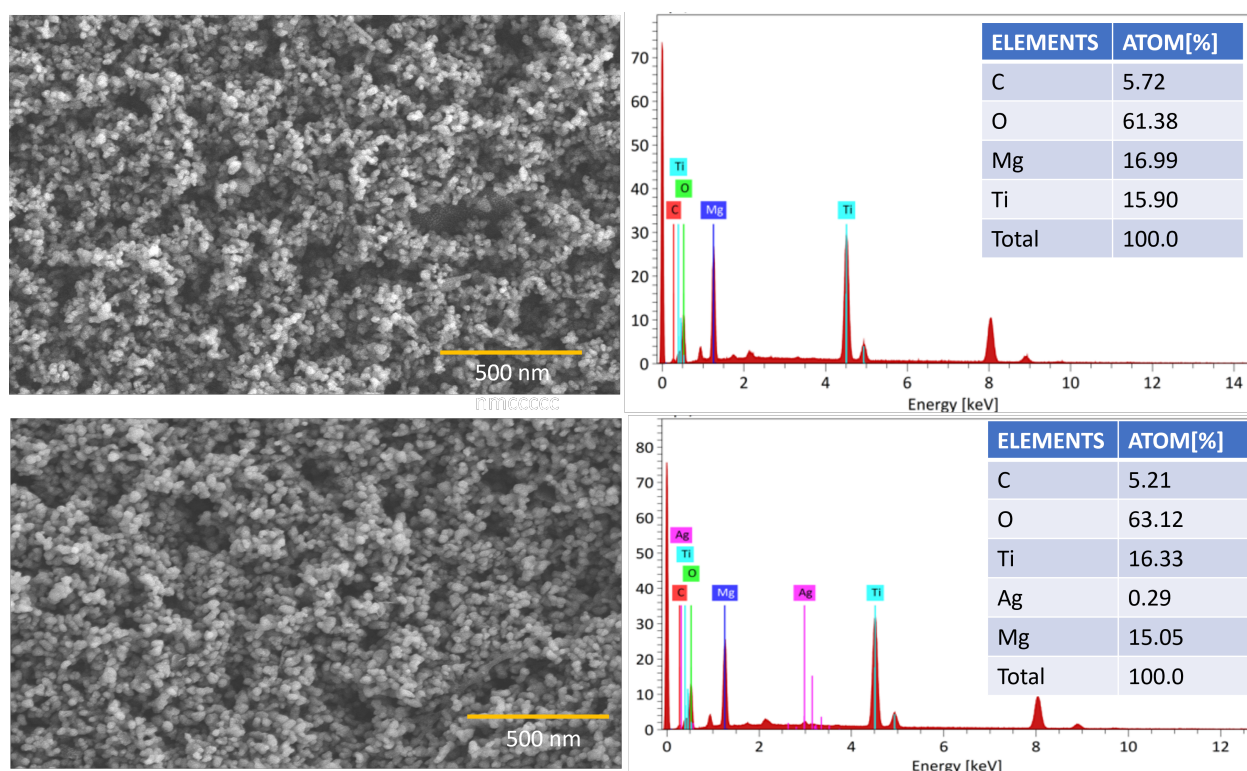
deposited MgTiO<sub>3</sub> samples at optimal 1 wt%. The MgTiO<sub>3</sub> structure has twelve main reflection peaks at the diffraction angle (2θ) readings of 19.25°, 21.28°, 24.03°, 32.79°, 35.53°, 40.64°, 49.135°, 57.10°, 62.08°, 63.78°, 71.22°, and 75.28°. These correspond to the (003), (101), (012), (104), (110), (113), (024), (116), (018), (214), (300), and (220) crystal planes, respectively (Reference pattern 01–079-0831) [28]. In addition to the diffraction peaks, the minor secondary phase TiO<sub>2</sub> peak at (2θ) 27.38 is indicated by “♣” asterisks on the graph. The XRD pattern of Ag-MgTiO<sub>3</sub> has two low-intensity peaks at 2θ values of 38.2° and 44.1, which relate to the (111) and (200) planes of metallic Ag, respectively. These peaks are denoted with “\*” asterisks in the graph. The fact that the silver Nanoparticles are well-loaded or disseminated on the surface instead of being embedded into the MgTiO<sub>3</sub> lattice is further supported by the fact that the MgTiO<sub>3</sub> peak location did not alter.



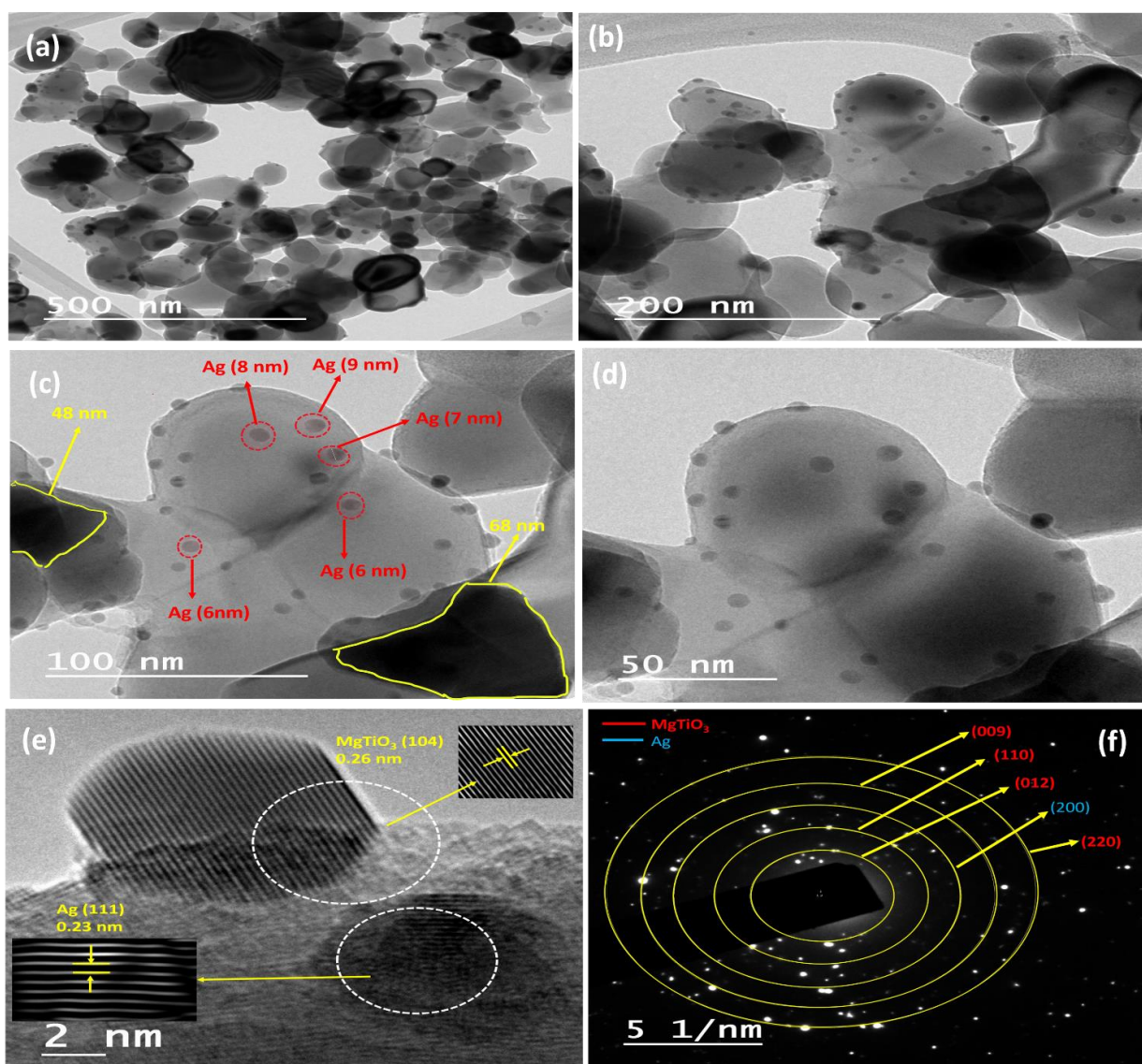
**Fig. 1:** X-ray diffraction profiles of MgTiO<sub>3</sub> and Ag-MgTiO<sub>3</sub> nanocomposites.

### 3.2 Morphological study:

The surface pattern of the catalysts was characterized by employing FE-SEM and HR-TEM analyses. Fig. 2(a)–(b) shows FE-SEM images of MgTiO<sub>3</sub> and optimal 1wt% Ag-MgTiO<sub>3</sub> composites. All samples showed predominantly spherical clumps or clusters. The formation of clumps may be caused by a high calcination temperature of 700 °C. FE-SEM was unable to distinguish between the deposited silver nanoparticles and pure MgTiO<sub>3</sub> due to the low weight % of Ag deposited. However, the elemental composition data supports their presence on the MgTiO<sub>3</sub> surface. EDS analysis was utilized to determine the amount of elements found in the composite materials. Figures 2 (a)–(b) show separate EDS spectra and tables revealing elemental content data. The anticipated loading amount of Ag was 1wt%, whereas the observed value was 0.29%. The reduced Ag concentration could be attributed to selected area selection for EDX, mass loss during photo deposition, or washing.



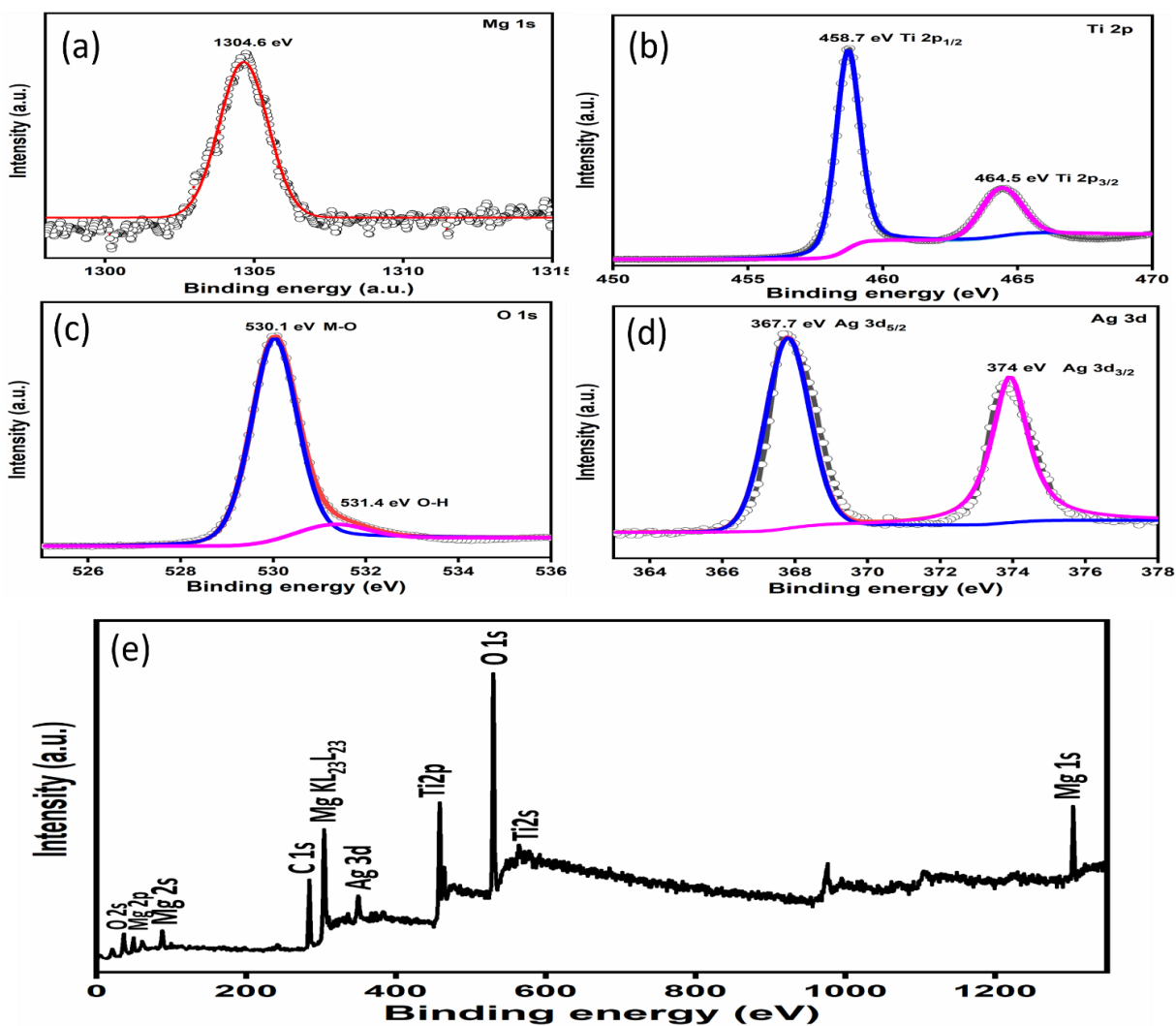
**Fig.2:** Field Emission Scanning Electron Microscopy images of (a) MgTiO<sub>3</sub>, (b) 1 wt% silver-modified MgTiO<sub>3</sub> and their corresponding EDX profiles.



**Fig. 3:** High-resolution TEM micrographs of (a-d) Ag-MgTiO<sub>3</sub> photocatalyst (e) associated lattice fringes and (f) SAED pattern for Ag-MgTiO<sub>3</sub> composite.

### 3.3. Oxidation state analysis by XPS

XPS was used to analyze each element's valence states and chemical composition in the Ag-MgTiO<sub>3</sub> nanocomposite. The outcomes are depicted in **Figure 4**. The survey spectrum shows clear peaks for Mg, Ti, O, and Ag (Fig. 4(e)). The detailed XPS spectra of Mg (1s) (Fig. 4(a)) centered at 1304.6 eV match up with the Mg<sup>2+</sup>. Figure 4(b) shows two distinct binding energy peaks at 458.7 eV and 464.5 eV, inferring that the oxidation state of Ti is +4. Fig. 4(c) depicts the deconvoluted spectra of O 1s, segmented into two separate peaks.



**Fig. 4:** XPS spectra of Ag-MgTiO<sub>3</sub> nanocomposite (a) Mg 1s, (b) Ti 2p, (c) O 1s, (d) Ag 3d, (e) survey scan.

One at a (Binding Energy) of 530.01 eV is assigned to oxygen in the MgTiO<sub>3</sub> crystal lattice, and the latter peak at 531.4 eV is attributed to the oxygen atom of O-H group bound to the surface of MgTiO<sub>3</sub> in the nanocomposite. The spectra of silver 3d (Fig. 4(d)) contain two distinct peaks, Ag 3d<sub>5/2</sub> (367.7 eV) and Ag 3d<sub>3/2</sub> (374.0 eV).

### 3.4 Porosity and Surface Area Evaluation

The BET-specific surface area graph of the Ag-MgTiO<sub>3</sub> nanocomposite and bare MgTiO<sub>3</sub> nanocomposite is depicted in Fig. 5. The nitrogen adsorption and desorption isotherms of both samples, classified by the IUPAC, exhibited type-IV Langmuir isotherm profiles with characteristic H<sub>3</sub>-structured hysteresis loops, suggesting their multi-layered design and mesoporous nature. Both samples' major pore sizes are between 3 and 21 nm, which supports the idea that the catalysts are made of mesoporous material. The Ag-MgTiO<sub>3</sub> composite (16.67 m<sup>2</sup>/g) has a lower BET-specific

surface area than pristine MgTiO<sub>3</sub> (20.247 m<sup>2</sup>/g). This modest drop in surface area could be attributed to Ag NPs populating the pores of mesoporous MgTiO<sub>3</sub>. Despite a minor decrease in surface area, this may not have diminished photocatalytic efficiency, which is also reliant on other parameters such as material band gap, electron transport between plasmonic Ag and MgTiO<sub>3</sub>, and suppression of electron-h<sup>+</sup> recombination, as Table illustrates in Fig. 5. Furthermore, the pore volume and size range of the Ag-MgTiO<sub>3</sub> nanocomposite was found to be slightly increased. Loading metallic silver nanoparticles into the MgTiO<sub>3</sub> surface may be responsible for the expansion in the different BET parameters. This would provide many active zones for contaminant adsorption and speed up the movement of reactants and products, both of which are necessary to achieve high photocatalytic activity.

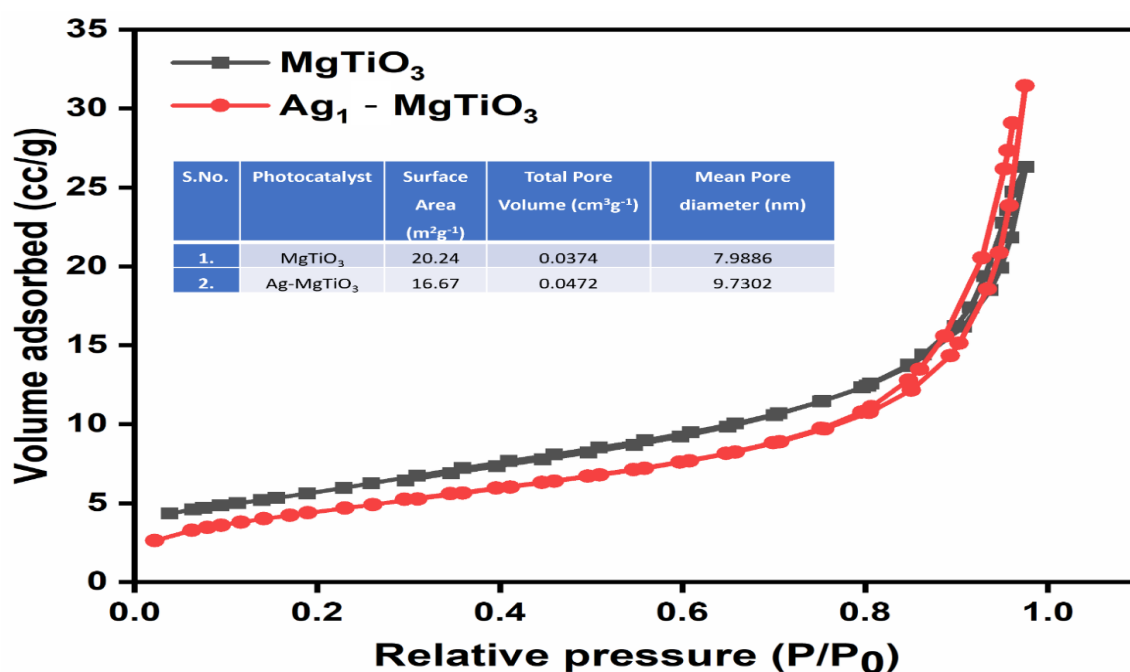
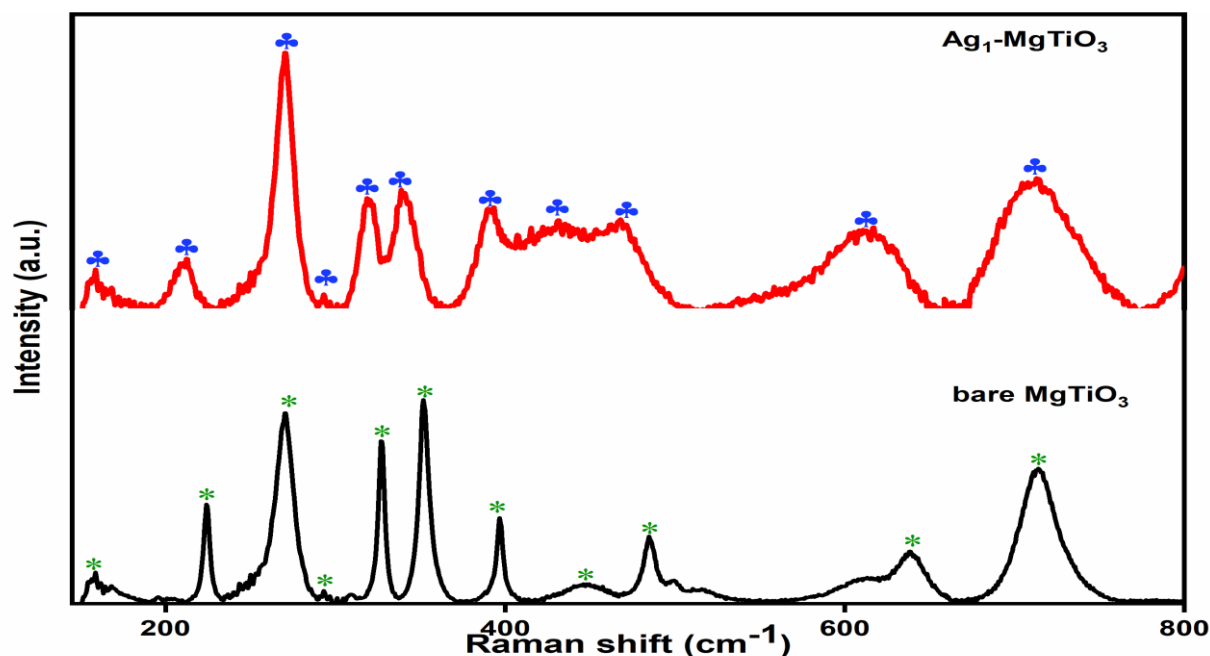


Fig. 5: Nitrogen adsorption isotherms curves of MgTiO<sub>3</sub> and Ag-MgTiO<sub>3</sub> nanocomposite.

### 3.5 Vibration mode analysis by RAMAN spectra

Figure 6. displays the Raman spectrum comparison of the Ag-MgTiO<sub>3</sub> and MgTiO<sub>3</sub> samples. The Raman spectrum of pure magnesium titanate (MgTiO<sub>3</sub>) has multiple characteristic peaks at 225, 271, 291, 318, 351, 397, 432, 483, 632, and 716 cm<sup>-1</sup>, which correspond to Ag, Eg, Ag, Eg, Eg, Ag, Eg, Ag, Eg, and Ag symmetry. Additional peaks at 162 cm<sup>-1</sup> may be due to unreacted starting precursors[30,31]. The peak at 225 cm<sup>-1</sup> is primarily due to vibrations between Mg and O atoms. The broad, strong signals at 318 and 352 cm<sup>-1</sup> are due to the O-Ti-O bending vibration within the TiO<sub>6</sub> group of MgTiO<sub>3</sub>. The higher frequency peak at 716 cm<sup>-1</sup> indicates symmetric Ti-O stretching

vibrations. After Ag-decoration, all modes showed a slight peak broadening, which might be attributed to scattering contributions outside the Brillouin zone [32]. The  $E_g$  peak at 318 and 351  $\text{cm}^{-1}$  shows a significant drop in intensity, demonstrating the fall in crystallinity of  $\text{MgTiO}_3$  nanostructures with Ag loading [33].



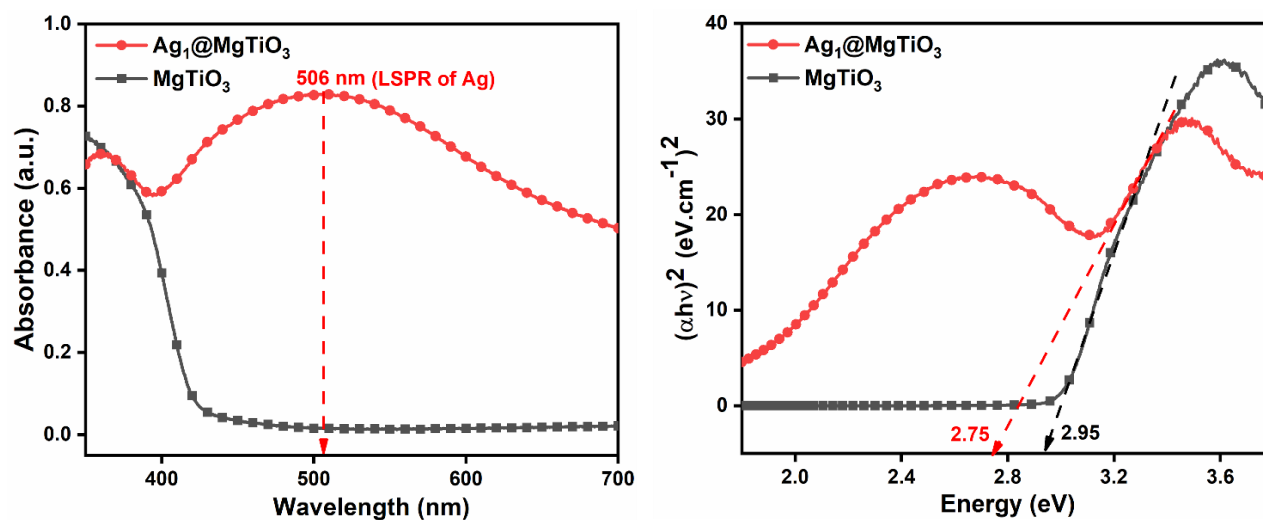
**Fig 6.** Raman spectra of  $\text{MgTiO}_3$  and the  $\text{Ag-MgTiO}_3$  nanocomposite.

### 3.6. Optical and charge transfer specifications

#### UV-Vis Diffuse reflection measurements

**Figure 7.** Depicts the U-V spectra of bare and 1 wt%  $\text{Ag-MgTiO}_3$  composites.  $\text{MgTiO}_3$  exhibited a peak in the UV region, possibly indicating a transition of electrons from the valence to the conduction band. After the deposition of silver, the composite range of absorption moved dramatically toward visible light. LSPR bands for silver in composites were observed at 506 nm.  $\text{Ag-MgTiO}_3$  composites exhibit UV and visible absorbance maxima, indicating their high photoactivity and ability to respond in the full-range spectrum. Depositing Ag metal on the  $\text{MgTiO}_3$  surface resulted in a color change from white ( $\text{MgTiO}_3$ ) to grey ( $\text{Ag-MgTiO}_3$ ) (see Scheme 2). This demonstrates that the deposited metal alters the light absorption capabilities of  $\text{MgTiO}_3$ . Additionally, the band gap energies of the samples were determined through the Tauc relation, given by:  $\alpha h\nu = A (h\nu - E_g)^n$  where  $\alpha$  = absorption coefficient,  $h\nu$  = photon energy,  $A$  = constant,  $E_g$  = the band gap of the material and  $n$  is the exponent coefficient ( $n=2$  for direct band gap). The exact band gap value was computed by plotting the graph between  $(\alpha h\nu)^2$  versus  $h\nu$ . The  $E_g$  values of the  $\text{MgTiO}_3$  and  $\text{Ag-MgTiO}_3$  samples were determined to be 2.95 eV and 2.75 eV.

The band gap value decreases notably from 2.95 to 2.75 eV upon the deposition of silver onto MgTiO<sub>3</sub>. The Ag-MgTiO<sub>3</sub> catalyst has a narrower band gap than the MgTiO<sub>3</sub> catalyst. Based on DRS spectra findings, it could be concluded that enhancing the visible-illumination absorption effectiveness here would favor the generation of numerous charge carriers and thus elevate the photocatalytic efficiency of the metal-loaded composite.

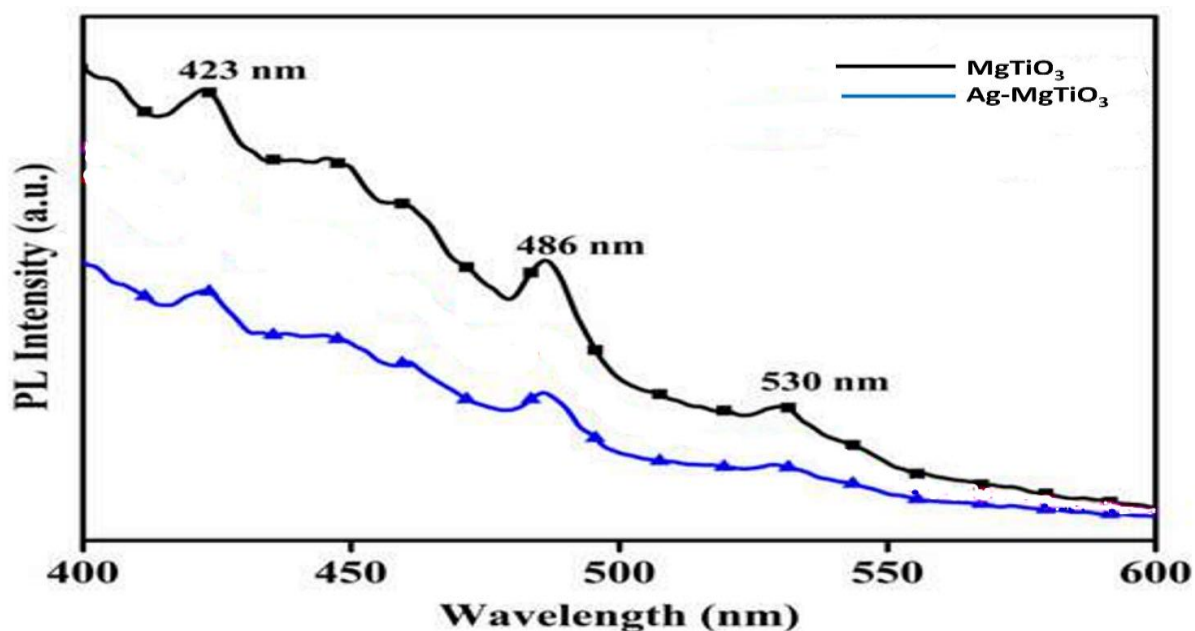


**Figure 7.** UV-Vis diffuse reflectance spectra and (b) a graph of  $(\alpha h\nu)^2$  against  $h\nu$  (eV) to determine the band gap of MgTiO<sub>3</sub> and Ag-MgTiO<sub>3</sub> nano-sized particles.

### Photoluminescence studies

PL spectroscopy was employed to evaluate the separation of charge carrier efficiency across several samples. PL strength is generally recognized as directly related to the rate of electron-hole pair recombination: higher recombination rates result in stronger PL signals, whereas lower rates result in weaker signals [34]. Figure 8. depicts the PL spectra recorded at room temperature for both MgTiO<sub>3</sub> and Ag-MgTiO<sub>3</sub> nanocomposites excited at 335 nm. Both samples show peaks at 423, 486, and 530 nm. The 423 nm peak relate to near-band edge emission, whereas the 486 and 530 nm peaks correspond to deep-level emissions. The near-band edge peak in the UV range is caused by the reformation of charge carriers generated by stimulation with energy equal to or surpassing the bandgap. DLE peaks in the visible area are associated with inherent defects such as oxygen vacancies (VO), titanium vacancies (VTi), and imperfections on the surface. The addition of Ag did not significantly affect the band edge positions, but it did cause a considerable decrease in PL intensity when compared to bare MgTiO<sub>3</sub>. This reduction indicates a decrease in the reformation rate of photoinduced electron-hole pairs in the silver-modified MgTiO<sub>3</sub> composites. This effect is most likely owing to Ag's capability to trap electrons, which improves charge transfer from the MgTiO<sub>3</sub> surface

to the silver particles, minimizing reformation and increasing the lifetime of charge carriers. The highest PL quenching occurs in 1 wt% Ag-MgTiO<sub>3</sub> nanocomposites, indicating that this Ag concentration is ideal for effective electron transport. Enhancing the silver content beyond 1 wt% leads to an increase in PL intensity, likely due to overwhelming metal loading that covers the MgTiO<sub>3</sub> surface. This excess metal can increase the rate of electron-hole reformation and decrease photocatalytic effectiveness. Among the two samples, the 1 wt% Ag-loaded MgTiO<sub>3</sub> had the lowest PL signal.



**Fig. 8:** Photoluminescence ( $\lambda_{\text{max}} = 335 \text{ nm}$ ) spectra of bare MgTiO<sub>3</sub> and Ag- MgTiO<sub>3</sub> nanocomposites.

### 3.7. Photocatalytic activity

#### 3.7.1. Photocatalytic degradation

The light-driven catalytic efficiency of the Ag-MgTiO<sub>3</sub> nanocomposite was initially assessed by investigating the UV light, Visible light, and sunlight-induced breakdown of the cationic dye fuschin blue (Fuc) (Fig. 9 (a-b)). To provide a comparison, Fuc was removed from the bare MgTiO<sub>3</sub> and Ag-MgTiO<sub>3</sub> composites. Along with photocatalysis, assessments on photolysis and adsorption were carried out, and the findings are shown in Figure 9(a). In the nonexistence of a photocatalyst, just 12% of Fuc is destroyed after 50 minutes of direct sunlight exposure, suggesting its remarkable steadiness.

Following 30 minutes of dark adsorption, the adsorption effectiveness of several composites was computed: MgTiO<sub>3</sub>: 13.16%; Ag-MgTiO<sub>3</sub>: 18.21%. Ag-MgTiO<sub>3</sub> exhibits higher adsorption towards Fuc than unmodified MgTiO<sub>3</sub>, and when Ag is integrated onto the surface of MgTiO<sub>3</sub>, its adsorption capacity increases, which is attributed to the increased BJH pore volume, size, and adsorption sites. Furthermore, natural sunlight was irradiated before the photocatalytic process to improve removal efficiency. After 50 minutes of light exposure, the nanocomposites advance according to the outlined progression: Ag-MgTiO<sub>3</sub> (99.2%) > MgTiO<sub>3</sub> (55.6%) {Fig. 9(c-d)}. Thus, the Ag-MgTiO<sub>3</sub> exhibits dynamic Fuc elimination, which is consistent with previous characterization findings. Furthermore, utilizing pseudo-first-order rate kinetics, the related reaction rate constants (k) were Determined through numerical analysis,

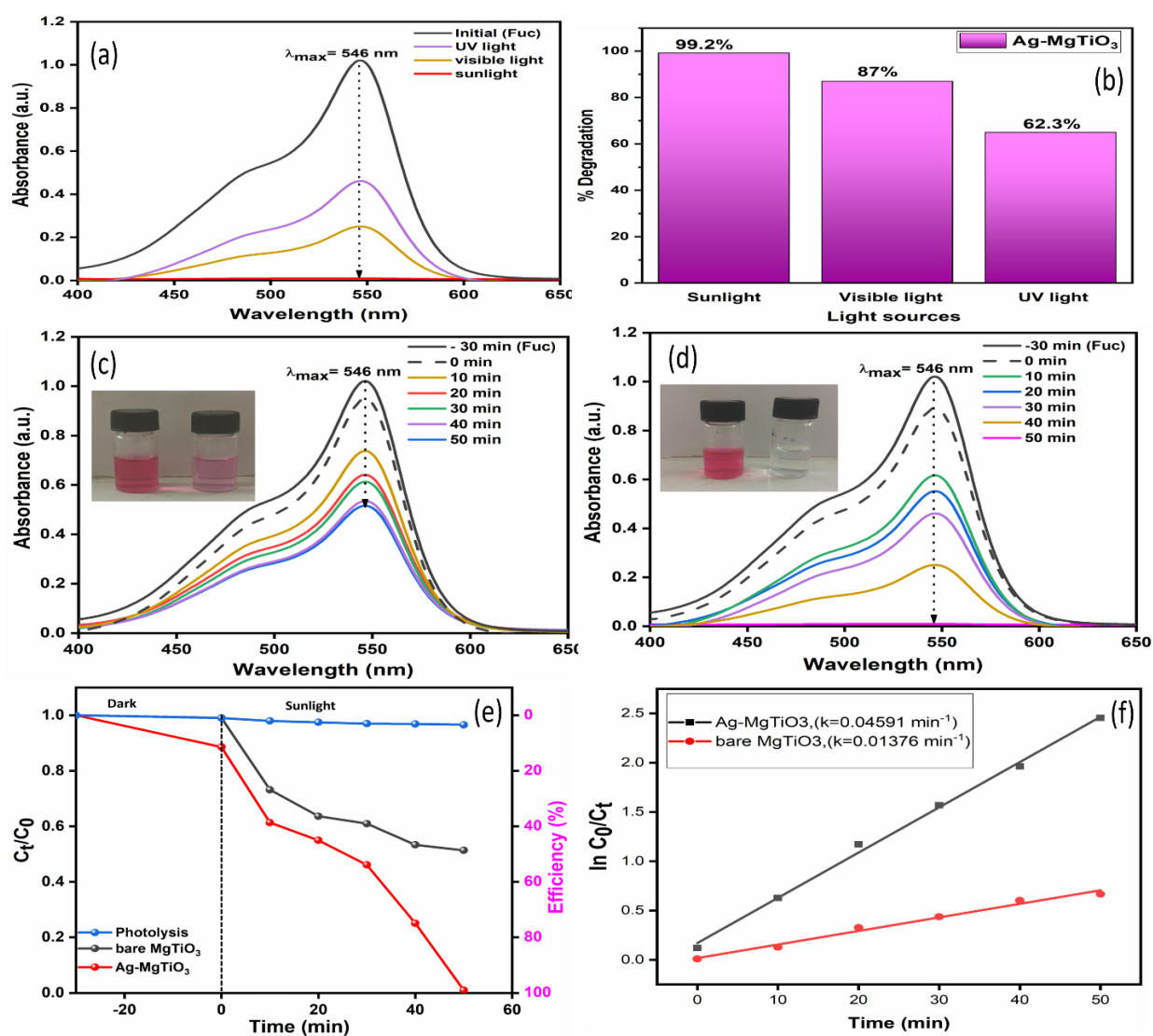
$$\{\ln(C_t/C_0) = -kt\} \quad (2)$$

Here C<sub>0</sub> is the initial concentration of C<sub>v</sub>, C<sub>t</sub> is its concentration at the time (t), and k denotes the rate constant (min<sup>-1</sup>). Figure. 9(f) compares the ln(C<sub>t</sub>/C<sub>0</sub>) readings for the various samples based on the extent of their exposure to light. The Ag-MgTiO<sub>3</sub> photocatalyst had a high Fuc degrading rate constant (0.04591 min<sup>-1</sup>), which is nearly 3.336 times greater than that of pristine MgTiO<sub>3</sub> NRs. It is apparent that the intensity of the maximum absorption peak of the characteristic feature increases as illumination time lengthens during deterioration, Fuc dye at 546 nm is observed to diminish progressively by both catalysts. The Ag-MgTiO<sub>3</sub> nanocomposite showed a markedly greater decline after 50 minutes of sunlight irradiation. The comprehensive reducing pattern seen in the Fuc spectral analysis demonstrates the superior photocatalytic efficiency of Ag-MgTiO<sub>3</sub> suggesting that the Ag loading helps enhance the photocatalytic activity of MgTiO<sub>3</sub>. The experimental results show that adding Ag on the surface of MgTiO<sub>3</sub> considerably improves its degrading performance. These metallic coatings operate as electron accumulation sites, catching photo-excited electrons from the MgTiO<sub>3</sub> surface and transferring them to oxygen molecules. This process enhances the production of highly reactive electrons and superoxide radical anions (·O<sub>2</sub><sup>-</sup>), which are essential for successfully decomposing Fuc. To get deeper insights into the improved photoactivity of MgTiO<sub>3</sub>, the valence band and conductance band potentials of MgTiO<sub>3</sub> relative to the Normal Hydrogen Electrode were calculated based on its band gap using Equations. **(3)-(4):**

$$E_{CB} = X - E_e - 0.5E_g \quad (3)$$

$$E_{VB} = X - E_e + 0.5E_g \quad (4)$$

In this equation, X represents the absolute electronegativity of  $\text{MgTiO}_3$  (5.12 eV),  $E_e$  is the energy of free electrons on the hydrogen scale (4.5 eV), and  $E_g$  is  $\text{MgTiO}_3$ 's band gap (2.75 eV). The computed Conduction and Valence potentials were found to be -0.755 V and 1.995 V vs NHE.



**Fig. 9:** (a-b) Shows degradation efficiency of Fuchsin dye using  $\text{Ag-MgTiO}_3$  photocatalyst in various light sources, (c-d) demonstrates the variations in the UV-vis absorption spectrum of the Fuchsin dye solution over both catalysts ( $\text{MgTiO}_3$  and the  $\text{Ag-MgTiO}_3$  sample) across different exposure durations, (e) Kinetic plots over time and (f) the associated variations in apparent reaction rate constants ( $k$ ) for the deterioration of Fuchsin dye under the influence of distinct photocatalysts.

**3.7.2 Effect of visible/sunlight illumination:** Under ideal conditions, relative studies assessed photocatalytic activity utilizing various light sources, including UV, visible, and natural sunlight. As shown in Fig. 9(a-b), pollutants deteriorated at an efficiency of 62.3% under UV irradiation and 87% under visible irradiation. Notably, using the  $\text{Ag-MgTiO}_3$  nanocomposite under natural sunlight resulted in the best removal efficiency of 99.2%. These findings highlight the superior

effectiveness of natural sunlight in pollutant degradation in conjunction with the proposed photocatalyst. Thus, natural sunlight has a significant advantage over non-natural light sources for Contaminant breakdown.

**3.7.3 Influence of amount of catalyst:** The correct amount of catalyst utilized considerably impacts the photocatalytic process' efficiency. Using the appropriate catalyst quantity is critical to avoid overuse, Potentially causing particle clustering. Furthermore, utilizing the appropriate amount ensures maximum photon absorption, which improves photodecomposition efficiency. Experiments Were performed to determine the impact of catalyst concentrations ranging from 0.1 to 0.5 g/L on dye photocatalytic degradation. It was observed that raising the photocatalyst dose from 0.1 to 0.3 g/L significantly improved degradation efficiency, reaching around 99.2% at 0.3 g/L. This increase is due to the increased number of active reaction sites caused by the larger catalyst quantity. However, as the concentration increased to 0.4 g/L, degradation efficiency dropped. This drop can be due to the solution becoming opaque, resulting in light dispersion and decreased effectiveness in the solution. Furthermore, at greater concentrations, the dye fully coats the catalyst surface due to full adsorption, making further catalyst embedding useless. This saturation causes the deactivation of active sites, reducing their efficiency. Consequently, the optimal concentration for future studies was 0.3 g/L of photocatalyst[35].

### **3.8 Mineralization of dye by TOC measurement**

As mineralization is the end goal of pollutant treatment, Total Organic Carbon (TOC) testing was employed to determine how effectively contaminant molecules were mineralized during photocatalysis. The TOC value generally represents the quantity of organic carbon dissolved in the aqueous solution, and a decline in the concentration of TOC provides insight into the level of mineralization of the organic substance upon completion of the reaction. TOC was analyzed at the beginning and end of the photodegradation process to determine the amount of Fuc dye mineralization over the fabricated Ag-MgTiO<sub>3</sub> photocatalyst. The TOC removal efficiency of the Ag-MgTiO<sub>3</sub> photocatalyst for removing Fuc dye was examined using a dichromate-based chemical approach. The findings demonstrate that the TOC values of Fuc dye decreased from 27.7 to 8.9 mg/l after 50 minutes of direct sunlight exposure, representing a 67.8% TOC reduction. The decreased TOC values indicate that the Ag-MgTiO<sub>3</sub> photocatalyst will mineralize Fuc pollutants. Furthermore, degradation efficiencies obtained from the UV-Vis spectrophotometer were higher than the mineralization activity. The slow rate of both photodegradation and mineralization is evident from the partial mineralization of pollutants. This is primarily due to the creation of intermediate products as studied

by HRMS studies. Complete mineralization of the remaining intermediates is possible, but it will likely need additional sunlight exposure.

### 3.9 Scavenger studies

Radical capture studies were carried out to understand better how active species in Ag-MgTiO<sub>3</sub> nanocomposite engaged in photocatalytic removal of Fuc as well as propose a mechanism. Various scavengers (K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub>, benzoquinone, IPA, and methanol) were assigned as quenchers of e<sup>-</sup>, O<sub>2</sub><sup>•-</sup>, <sup>•</sup>OH, and h<sup>+</sup> radicals. As shown in Fig. 10, Under identical conditions as degradation experiments, revealed that the photo removal effectiveness of Fuc was decreased in the presence of quenchers listed according to their significance in the following sequence Methanol > IPA > K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> > Benzoquinone, indicating that O<sub>2</sub><sup>•-</sup> and e<sup>-</sup> radicals are the primary contributors, with <sup>•</sup>OH playing a minor role. These results demonstrate that e<sup>-</sup> and O<sub>2</sub><sup>•-</sup> are important reactive species in Fuc photocatalytic degradation.

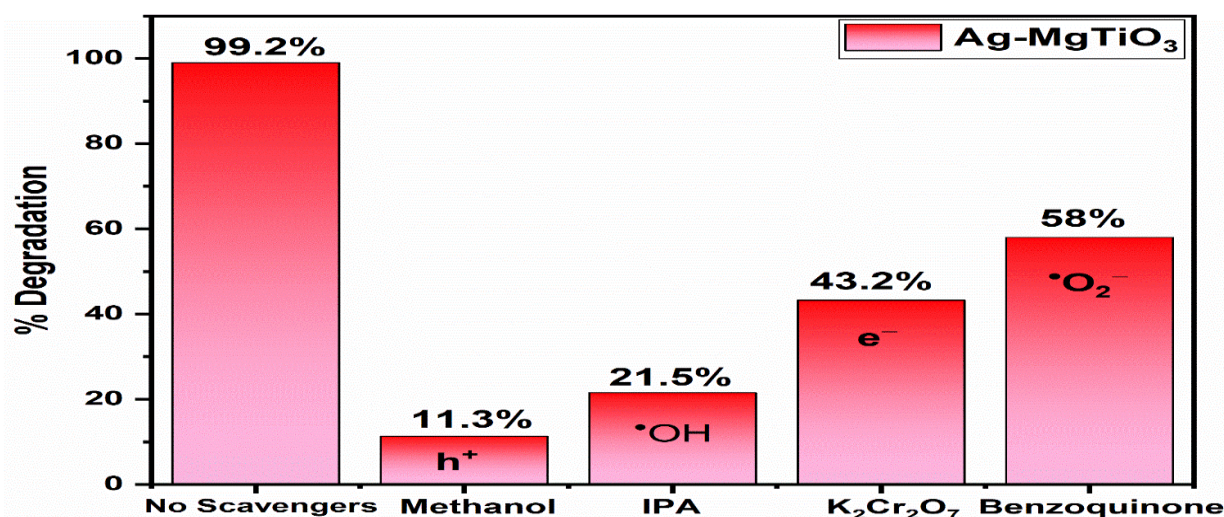
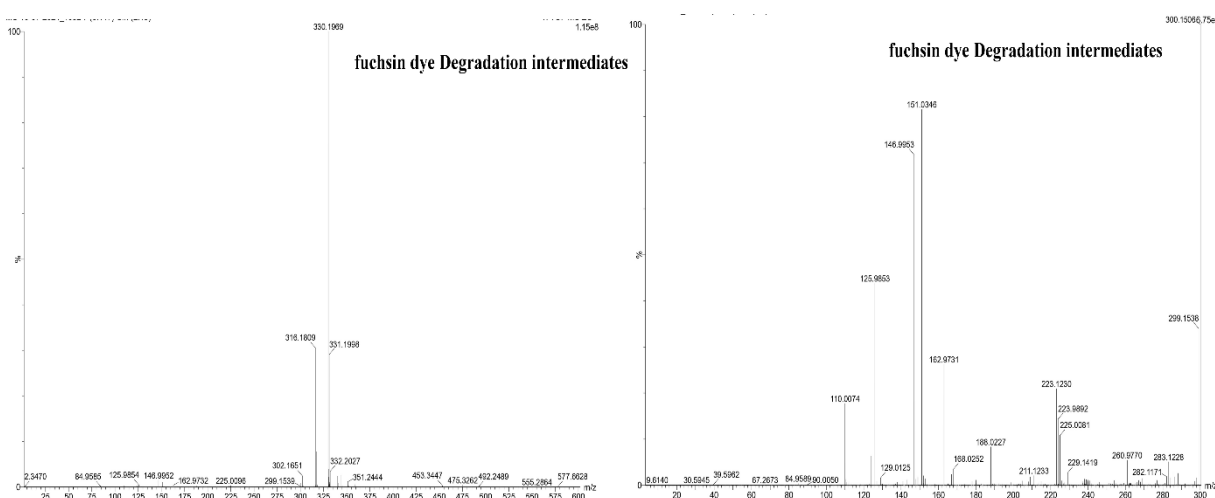


Fig 10: Impact of a set of scavengers on Fuchsin dye degradation by Ag-MgTiO<sub>3</sub> nanocomposite.

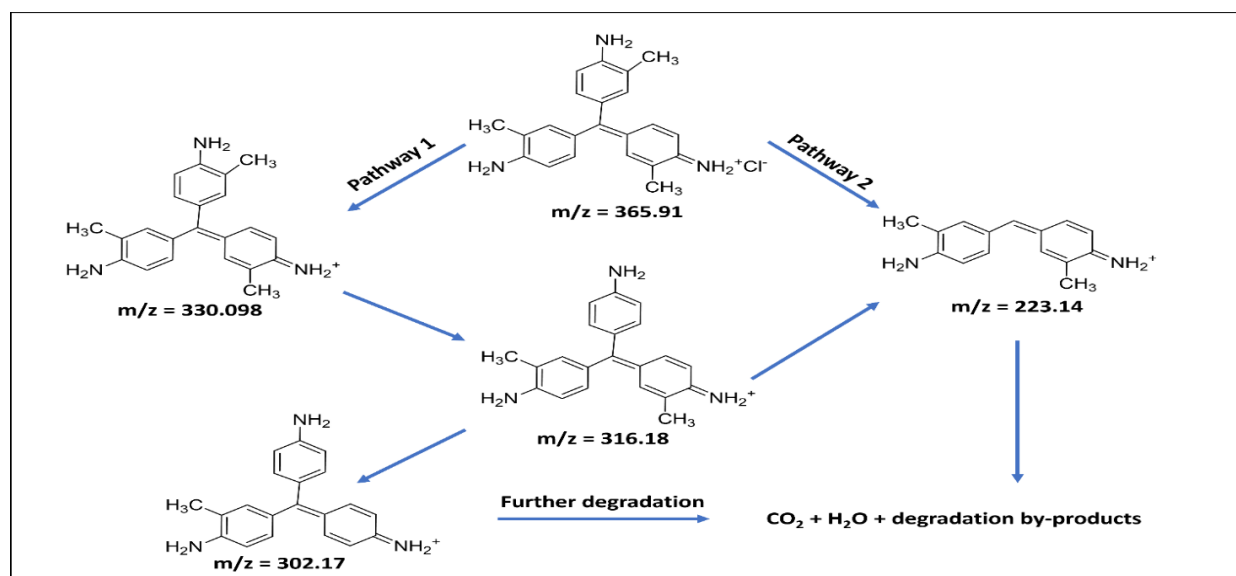
### 3.10 Photocatalytic degradation pathways

Based on the HRMS data, two degradation routes of fuchsin dye are suggested and provided in Fig. 12. Several fragments of smaller molecular masses than fuchsin dye are observed in the HRMS chromatogram, out of which the structures of compounds with peaks at m/z = 316, 302, 223 have been identified. In pathway 1, the fuchsin dye (m/z = 365) undergoes the loss of a methyl (-CH<sub>3</sub>) group to give the fragment with m/z = 316, which further loses a -CH<sub>3</sub> group, forming the fragment with m/z = 302. In pathway 2, the fuchsin dye can undergo the loss of a 2-methylaniline ring, thus

forming the intermediate with  $m/z = 223$ . Further, prolonged solar or UV/visible irradiation may be required for these fragments of fuchsin dye to fully mineralize into  $\text{CO}_2$ ,  $\text{H}_2\text{O}$ , and other simple molecules compared to what has been investigated here [36].



**Fig 11.** The mass spectra of Fuchsin dye after photodegradation using  $\text{Ag-MgTiO}_3$  catalyst



**Fig 12:** Degradation pathway of Fuchsin dye using  $\text{Ag-MgTiO}_3$  photocatalyst under sunlight.

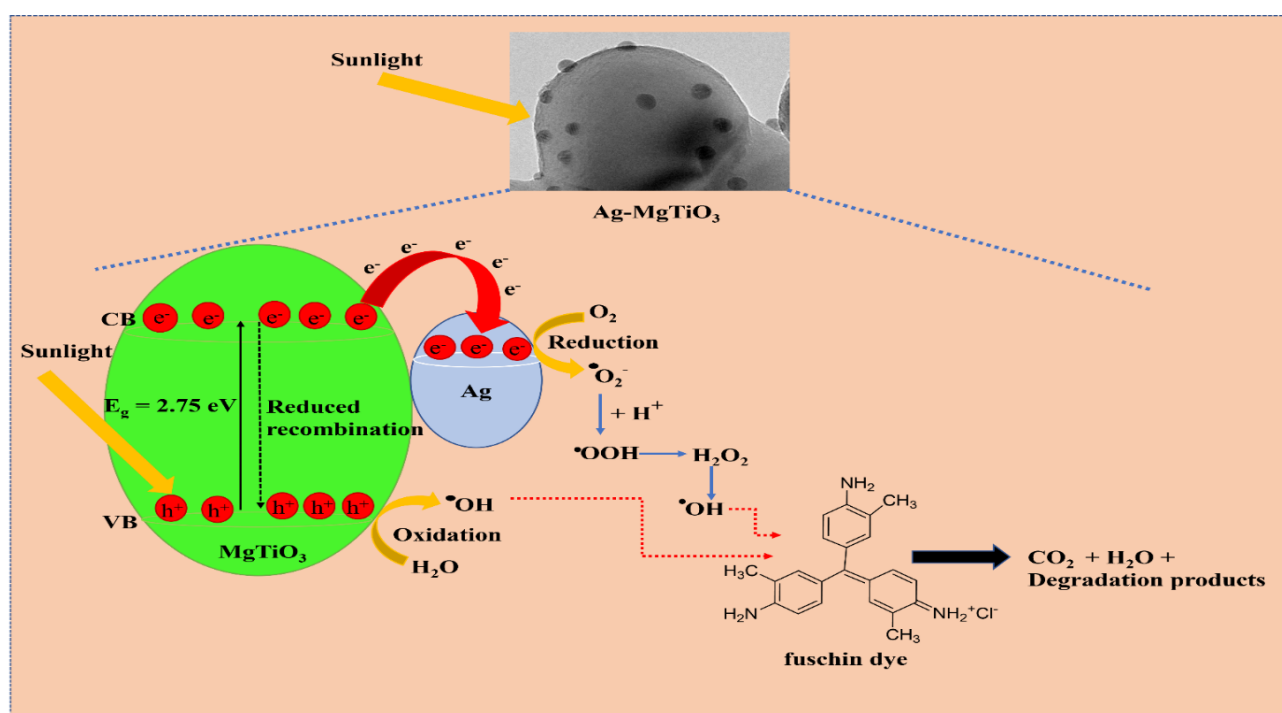
### 3.11 Possible degradation mechanism

Scheme 3. depicts the mechanism responsible for the degradation of Fuc under sunlight using the as-prepared catalysts. When exposed to light, electrons in  $\text{MgTiO}_3$  are excited from the valence band to the conduction band, forming holes ( $\text{h}^+$ ) in the Valence Band. These photoexcited electrons and holes subsequently interact with adsorbed oxygen and water molecules, resulting in reactive oxygen species including superoxide radical anions ( $\cdot\text{O}_2^-$ ) and hydroxyl radicals ( $\cdot\text{OH}$ ). These highly oxidative species work together to effectively degrade or mineralize Fuc. In contrast, Ag has a lower redox

potential (+0.799 V vs. NHE) than MgTiO<sub>3</sub>'s CB potential (-0.75 V), which allows photoexcited electrons to flow quickly from MgTiO<sub>3</sub> to the Ag surface. This speeds up the separation of electron-hole pairs within MgTiO<sub>3</sub>. The deposited Ag functions as an electron sink or trap, preventing the electron-hole reformation process. The overall reactions during photocatalysis under sunlight, with Ag-deposited (MgTiO<sub>3</sub>) photocatalyst are depicted below:



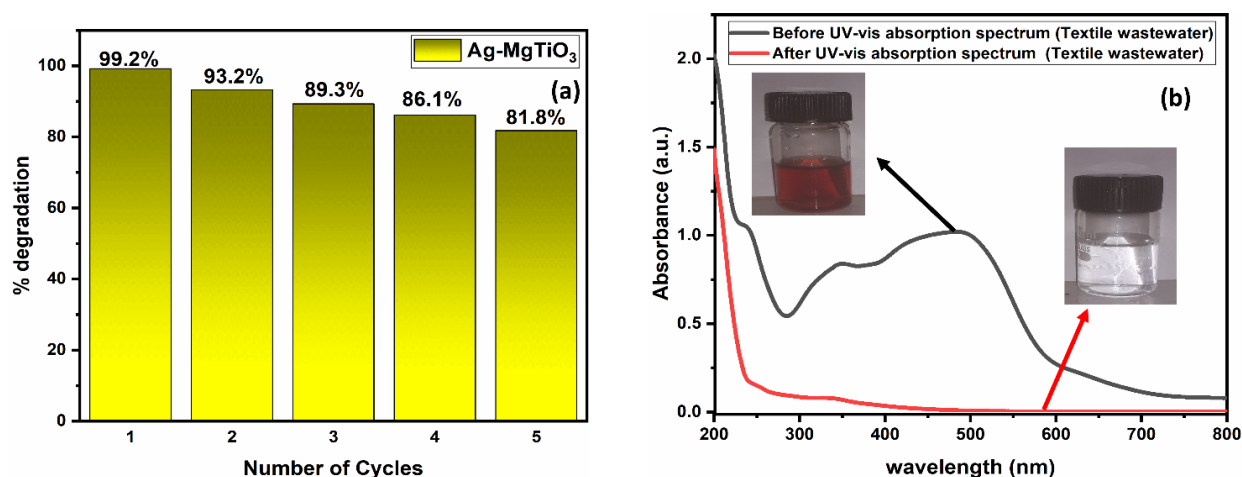
This transport mechanism in the metal-loaded photocatalyst is anticipated to reduce charge carrier recombination, greatly enhancing photocatalytic performance.



**Scheme 3:** Plausible mechanism of Fuchsin dye degradation under sunlight irradiation.

### 3.12 Photodegradation of textile dye in wastewater And Recyclability Studies

In practical applications, the reusability and stability of photocatalysts are essential for their effectiveness. To evaluate these aspects, a series of recycling tests were performed using an Ag-MgTiO<sub>3</sub> composite to degrade Fuc dye. This involved running five sequential cycles, each with a 50-minute reaction duration. For each cycle after the initial one, the photocatalyst was retrieved via centrifugation, cleaned, and dried for subsequent use. According to Figure 13(a), the degradation efficiency of the Fuc dye dropped by just 17.4% from the first to the fifth cycle, demonstrating the high reusability of the Ag-MgTiO<sub>3</sub> catalyst. The reduction in performance from 99.2% to 81.8% is likely due to some catalyst loss during the separation and recycling phases. In summary, the Ag-MgTiO<sub>3</sub> composite shows excellent degradation performance, good renewability, and robust photostability, making it a highly effective material for photocatalytic applications. Fig 13(b) shows the photocatalytic efficiency of the produced materials Ag-MgTiO<sub>3</sub>, which was tested using textile wastewater that we took from a textile dyer in Model Town, Degradation. The photocatalytic process was carried out under direct sunlight. 5 mg of photocatalyst was dispersed into 10 mL of textile wastewater at a 0.5 g/L concentration. Before irradiation, the suspensions were agitated in the absence of light for 30 minutes to establish an Equilibrium of adsorption and desorption processes between the dye molecules and the catalyst's surface. To initiate deterioration, the solution was stirred for 110 minutes with the vessel horizontal and exposed to direct sunlight. After removing the MgTiO<sub>3</sub> catalyst, roughly 5–6 mL of the solution was taken out and examined with a UV-visible spectrophotometer. The Ag-MgTiO<sub>3</sub> nanocomposite showed a markedly greater decline after 110 minutes of sunlight irradiation(99.4%).



**Fig.13:(a)** Reusability studies,(b) degradation efficiency of the textile wastewater solution over Ag-MgTiO<sub>3</sub> photocatalyst when exposed to direct sunlight.

## 4. CONCLUSION

The sonochemical method was used to synthesize MgTiO<sub>3</sub> and enhance its photocatalytic properties by loading it with Ag metal. The effective fabrication of the samples was confirmed using various analytical techniques, including XPS, DRS, FE-SEM, EDS, HR-TEM, XRD, PL, and BET analyses. FE-SEM and HR-TEM images revealed that Ag was effectively deposited onto the MgTiO<sub>3</sub> surface in the form of small spheres. The photocatalytic efficiency of the synthesized samples was evaluated by examining the deterioration of Fuc dye under natural sunlight. Ag-MgTiO<sub>3</sub> showed the highest photocatalytic efficiency and rate constant among the photocatalysts produced. PL, BET, and UV-visible DRS analyses showed that the composite effectively decomposed contaminants under natural sunlight thanks to its lower reformation rate, large surface area, and suitable band gap. The study also explored various factors affecting photocatalytic degradation, including catalyst concentration, pH levels, reaction kinetics, light sources, scavenger analysis, and the reusability of the catalyst. Trapping experiments identified h<sup>+</sup> as the main species driving pollutant degradation. The composite exhibited excellent recyclability over five cycles, with a high total organic carbon (TOC) removal rate, highlighting its efficiency and superiority over conventional physicochemical methods. HRMS study was conducted to identify the products and intermediates formed during the photodegradation. In conclusion, the Ag-MgTiO<sub>3</sub> catalyst revealed effective organic pollutant deterioration with sparse usage in natural sunlight, making it an attractive option for large-scale industrial wastewater treatment due to its cost-effectiveness, simplicity, and environmental benefits.

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