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IRON OXIDE THIN FILMS ON FTO FOR EFFICIENT PHOTOCATALYTIC APPLICATIONS: A NANO-ENHANCED APPROACH

Rajesh Kumar
Institute of Chemistry, Shah Abdul Latif University, Khairpur, Sindh, Pakistan
Email: rajeshkumar@salu.edu.pk

Bhajan Lal
Institute of Chemistry, Shah Abdul Latif University, Khairpur, Sindh, Pakistan

Muhammad Bachal Korai*
Institute of Chemistry, Shah Abdul Latif University, Khairpur, Sindh, Pakistan
ORCID iD: 0000-0002-0446-3876

Muhammad Aslam Korai
Institute of Chemistry, Shah Abdul Latif University, Khairpur, Sindh, Pakistan

Sagar Kumar
Institute of Chemistry, Shah Abdul Latif University, Khairpur - 66022, Sindh, Pakistan

ABSTRACT

In this study, we explored the use of ferrocene-based precursors for the fabrication of iron oxide nanoparticles and thin films on fluorine-doped tin oxide (FTO) substrates (dimensions: 40 × 40 × 2 mm) via a simplified precursor pyrolysis method. Utilizing the solid single-source precursor 4-ferrocenylaniline, two samples—designated S-1 and S-2—were synthesized to ensure uniform dispersion of iron oxide with well-defined morphology and controlled particle size distribution, effectively enhancing the surface area on the FTO substrates. Synthesis involved precise masses of 4-ferrocenylaniline (0.1–0.125 g), thermally decomposed at 550 °C for 30 minutes in an electric furnace. Utilizing Scanning Electron Microscopy (SEM), surface morphology of thin films was comprehensively analyzed, which revealed a uniform distribution of nanostructures. Elemental composition was analyzed using Energy Dispersive X-ray Spectroscopy (EDS), which verified existence of iron (Fe) and oxygen (O) as primary constituents, indicating high purity and successful deposition of iron oxide onto the FTO substrate. The photocatalytic activity of the films (S-1 and S-2) was further evaluated through degradation of Methylene Blue (MB) dye under natural sunlight, demonstrating their potential for environmental remediation applications. Sample S-2 demonstrated superior photocatalytic activity, achieving 60% degradation efficiency. Additionally, S-2 displayed excellent stability across multiple degradation cycles,

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highlighting its potential as a robust and reliable photo catalyst.

Keywords: Iron Oxide; 4-Ferrocenylaniline; Photo catalyst

Introduction

Nanotechnology marks a pivotal advancement in both scientific research and industrial innovation, offering unprecedented control and manipulation of matter at atomic and molecular dimensions (Manjunatha, Biradar, & Aladakatti, 2016). The term "Nano," derived from the Greek nanos meaning "dwarf" and the Latin nanus referring to something "extremely small," signifies a scale of measurement equivalent to 1 x 10^{-9} meters, thereby reflecting the minute dimensional focus of this discipline (Boholm, 2016; Madkour, 2019). In 2004, the Royal Society of Chemistry formally defined nanoscience as the systematic study and controlled manipulation of matter at atomic and molecular dimensions, whereas nanotechnology spread out this understanding to involve the conceptual design, engineering, and practical deployment of materials and devices with nanometer-scale features (Maynard, 2007). The roots of nanoscience can be traced back to 1902, when Richard Zsigmondy used an ultra-microscope to observe nanoparticles, laying the foundation for what would become a transformative scientific discipline (Gudimalla, Mishra, & Arora, 2022; Mukherjee et al., 2014; Sur et al., 2019). Today, semiconductor nanomaterials are at the forefront of nanoscience and nanotechnology research. These materials—existing as crystalline solids, amorphous forms, or even liquids—serve as the fundamental building blocks of modern nanotechnological applications (Talapin & Shevchenko, 2016). They are now crucial parts of fields ranging from optoelectronics and power conversion to environmental treatment and healthcare engineering because of their adjustable electric, optical, and structural characteristics (Tanaka & Shimakawa, 2011; Tauc, 2012).

The ease of production and broad variety of uses of metallic oxide nanoparticles make them one of the most adaptable substances for semiconductors (Campbell, Bateni, Volk, Abdel-Latif, & Abolhasani, 2020). They may be used in many production processes due to their exceptional chemical and thermal endurance, which also contributes to their growing importance in fields including physics, chemistry, and materials science (Camacho-Flores et al., 2015; Maduraiveeran, Sasidharan, & Jin, 2019; Nunes et al., 2019; J. Xu et al., 2018). These nanoparticles exhibit useful optical, electrical, and catalytic properties that are essential for a number of current technology-based applications (Chavali & Nikolova, 2019; Ishak, Kamarudin, & Timmiati, 2019). A wide variety of devices and systems, including photovoltaic cells, sensors that detect chemicals, catalytic agents, and environmental treatment methods, use metallic oxide nanoparticles (Falcaro et al., 2016; Khan et al., 2015; Li, Zhang, Niu, & Chen, 2012; Mallakpour & Madani, 2015). Because of their wide range of applications, it is essential to understand their construction methods, structural characteristics, and operating performance before using them in practical settings. They are very coveted for scientific research and industry expansion due to their capacity to function as magnetic, ocular, power, and catalytic components as well as their chemical endurance (Carenco, Portehault, Boissiere, Mezailles, & Sanchez, 2013).

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They can be used in a variety of fields, from medicine research and cosmetic preparations to electricity transmission and catalysis, thanks to their small particle size, enlarged interfaces, and increased chemical reactivity (Clavero, 2014; Melagraki & Afantitis, 2015). Ferric oxide nanostructures are particularly noteworthy among all of them. These are known for their super paramagnetic properties, high surface area in relation to size, and recyclability, which allow for usage in catalysis, medicine, and the environment. Furthermore, they are a popular choice for continued research and technological advancement due to their affordable and simple fabrication processes (A. Ali et al., 2016; Assa et al., 2016; Dulińska-Litewka et al., 2019; P. Xu et al., 2017).

Iron oxides nanostructures can be created using a variety of methods, such as precipitation by chemical means and sol-gel processes, which take controlled changes in particle size and shape into account (Campos, Pinto, Oliveira, Mattos, & Dutra, 2015; Sun et al., 2014). One-dimensional nanoparticles such as nanotubes and nanorods, which have unique properties which render them suitable for applications associated with water purifying and catalytic processes, are receiving more and more attention in contemporary scientific research (G. Zhang et al., 2017). Because of having semi-conductive character and highly paramagnetic properties, nanoparticles of iron oxide have drawn a lot of interest, which has prompted further research and advancement in this field. Their larger external surface, anisotropy charging transportation, and catalytic efficiency, which open up possibilities for practical applications of cutting-edge nanotechnology, are the primary factors driving the move toward one-dimensional structures (Zhao et al., 2010). Among such things, heterogeneity photocatalysis—which uses photocatalysts to boost photon-driven chemical reactions—is a key strategy for restoring the environment (Ibhadon & Fitzpatrick, 2013). Metallic oxides and transition- metal sulfide compounds are examples of semiconductor materials that have demonstrated a significant capacity to increase photoactivity and remove environmental pollutants from sources of water and air (F. Zhang et al., 2019). The present study used 4-ferrocenylaniline as the sole iron precursor to create iron oxide nanostructures in the form of nanoparticles and thin films on FTO substrates. Coatings with uniform morphology and a regulated particle size distribution across a wide interface were expected to be produced using this method. These findings confirm potential uses in a variety of nanotechnology and environmental applications and lend credence to growing scientific knowledge on the production of iron oxide nanomaterials.

Material and Methods

Materials

Investigational stuffs employed comprised 4-ferrocenylaniline, iron chloride, ethylene glycol, ammonium hydroxide, ethyl alcohol, caustic soda, sodium chlorate, hydrogen chloride, sodium nitrate, ammonium chloride, and deionized water. Each substance was of laboratory-standard and employed with no additional refinement.

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Method

Entire research was performed consuming water with highest pureness and substances of laboratory-standards with no more improvement. Entire glassware were washed through distilled water and oven-dried at 95°C. Fabrication of carbon-coated iron oxide was performed via pyrolysis of ferrocene in a temperature controlled furnace, with temperatures sustained minimum four hundred degree Celsius.

Creation of 4-ferrocenylaniline

The preparation of 4-ferrocenylaniline was carried out through a 2 step synthetic route starting with ferrocene. At first, a diazonium intermediate was generated from 4-nitroaniline, which subsequently underwent electrophilic substitution onto one of the cyclopentadienyl rings of ferrocene to yield a nitro-substituted derivative. This intermediate was then subjected to a catalytic reduction employing 10% palladium on carbon (Pd-C) in ethanol, ultimately affording the target amine compound (S. Ali, Badshah, Ataf, Lal, & Khan, 2013).

1st Step

In initial phase, 4-nitroaniline was dissolved in concentrated hydrochloric acid (HCl) and cooled to 0-5°C for 30 minutes. A sodium nitrite solution was then added dropwise, maintaining temperature lower than 5 °C for 30 minutes to form the diazonium salt. This freshly prepared diazonium solution was then introduced dropwise into a ferrocene solution while continuously stirring. After stirring for four hours at ambient temperature, reaction fusion was concentrated using rotary evaporation, and resultant mixture was filtered to remove the salts.

2nd Step

In the second step, the product from Step I was dissolved in ethanol and mixed using 80% hydrazine monohydrate along with 10% Pd-C catalyst in reaction vessel. The mixture was refluxed for four hours, after which the palladium catalyst was separated via filtration process. Final yield, identified as pale orange crystals, was isolated via rotary evaporation. Purity was assessed using TLC plates.

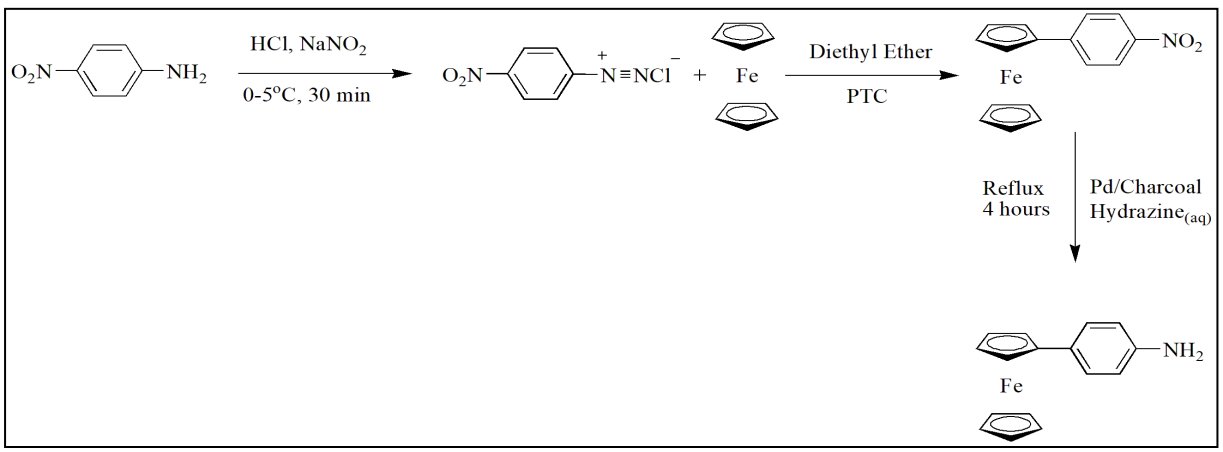

Figure 1 Creation of 4-ferrocenylaniline

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Fabrication of Iron oxide nanofilms over FTO

Fluorine-doped tin oxide (FTO) glasses were first cleaned with alcohol and dried. They were then placed in a porcelain dish containing varying amounts of 4-ferrocenylaniline (0.1 g and 0.125 g). The sample was sealed with an FTO glass cover and thermally treated at 500°C for half an hour. This treatment led to the formation of reddish brown iron nanofilms onto the FTO substrates (see Table 1 and Fig. 2).

Table 1. Iron Oxide Thin Film Formation Using 4-Ferrocenylaniline as a Precursor

Composites	Specimen codes	Original slides
Formation of Iron-oxide thin film by decomposing 0.1g of $C_{17}H_{16}FeN$	S-1	
Formation of Iron-oxide thin film by decomposing 0.125g of $C_{17}H_{16}FeN$	S-2	

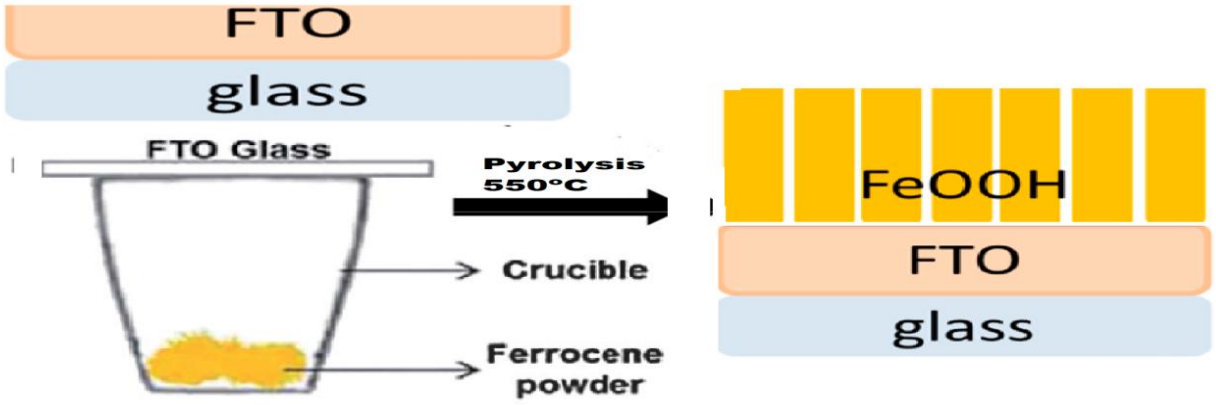

Figure 2 Formation of iron nanofilms onto the FTO substrates

Dye Degradation

To evaluate the degradation efficiency of the iron oxide thin films, commercially available Methylene Blue was used as a representative dye pollutant. The film was positioned at the base of a beaker filled with the dye solution, which was continuously stirred using a magnetic stirrer. The degradation progress was tracked by measuring the UV-Vis absorbance of Methylene Blue at 665 nm at regular intervals. The percentage of dye degraded was determined by comparing the absorbance values before and after treatment.

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Characterization

The stability of the iron oxide thin films was validated using UV-visible absorption spectroscopy. Spectral analysis in the 4000–400 cm^{-1} range was conducted using Fourier Transform Infrared (FTIR) spectroscopy (Thermo Nicolet-5700). To analyze the elemental makeup, Energy Dispersive X-ray (EDX) spectroscopy was performed, while morphology of surface of specimens was studied using SEM.

Results and Discussion

Characterizations of Fabricated Thin Films

Investigation of Functional Groups

FTIR spectroscopy was employed to investigate functional groups present in 4-ferrocenylaniline. A distinct absorption band at 492 cm^{-1}, attributed to the Fe–Cp (cyclopentadienyl) bond, confirmed the stability of the organometallic complex and reflected the basic character of the aniline moiety. The NH_2 group exhibited characteristic asymmetric and symmetric stretching vibrations at 3435cm^{-1} and 3372cm^{-1}, correspondingly. Additionally, noticeable difference in H-C enlarging frequencies was detected amongst nitro-substituted compound (2924cm^{-1}) and amino-functionalized analogue (3036cm^{-1}), indicating electron delocalization effects toward nitro functionality.

Surface Morphology

The surface morphology of synthesized iron oxide thin films was characterized using SEM, as surface features are critical to catalytic performance. SEM analysis revealed a bar-like and platelet-based morphology, as illustrated in **Figure 3**.

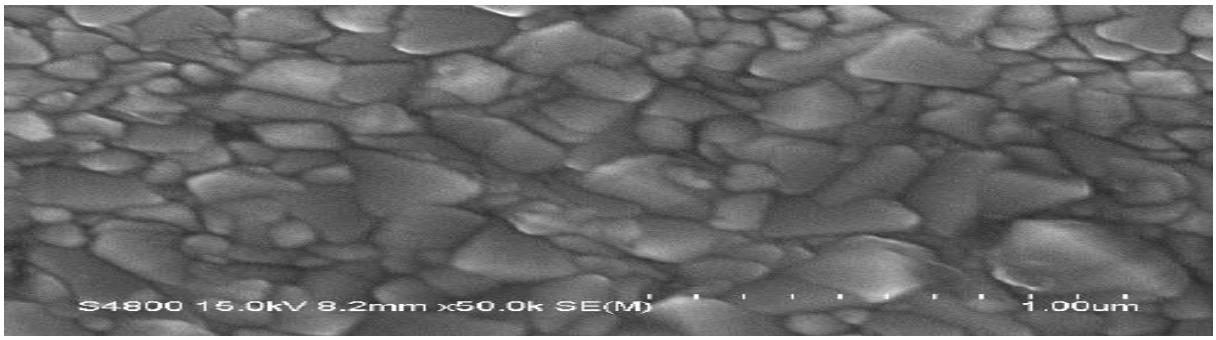

Figure 3 SEM image of naked FTO

Fig. 4 presents SEM images of iron-oxide thin films fabricated from 0.1 g of 4-ferrocenylaniline, deposited on FTO substrates. Images captured at low (500 μm) and high (1 μm) magnifications highlight the porous structure of the thin film uniformly covering the FTO surface. The micrographs reveal nanoscale platelet clusters dispersed across the substrate, forming aggregated heaps that define the film's texture and increase surface area.

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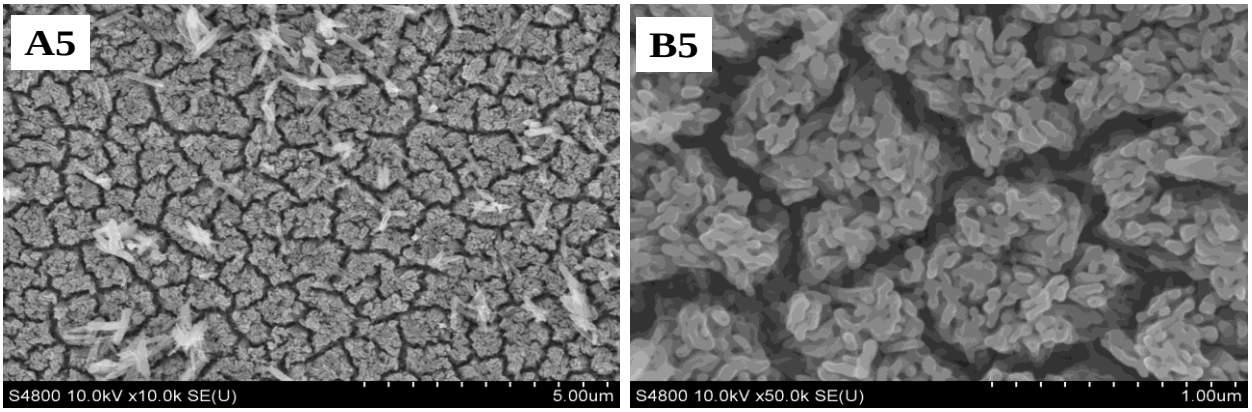

Figure 4 Sample S-1 SEM Images-(A5) Low, (B5) High Resolution

Figure 5 further shows SEM images of similar films at intermediate (5 μm) and high (1 μm) magnifications. These micrographs confirm the presence of densely packed nanoscale platelets, consistently distributed across the substrate. The porous and interconnected platelet structures suggest an increased active surface area, which is favorable for photocatalytic reactions.

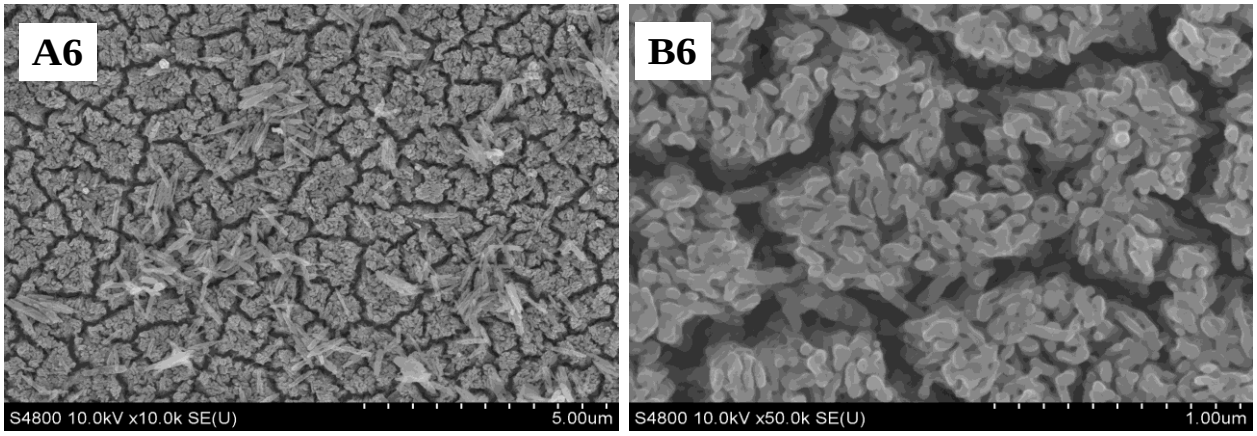

Figure 5 S-2 SEM Images-(A6) Low, (B6) High Resolution

Elemental Examination

Elemental analysis confirmed high purity of synthesized nanofilms, as demonstrated in Figure 6. The composition was determined using EDX, which revealed elemental profile of iron oxide layers set down on FTO substrates. EDX results validate successful incorporation of iron and oxygen elements while affirming the absence of significant impurities, indicating the effectiveness of the synthesis approach. These findings highlight the reliability and precision of the fabrication technique and are critical in supporting the potential of these nanostructured films for catalytic applications. In processes where elemental purity and precise stoichiometry are essential, such as in photocatalysis, the demonstrated material quality significantly

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enhances performance and reproducibility.

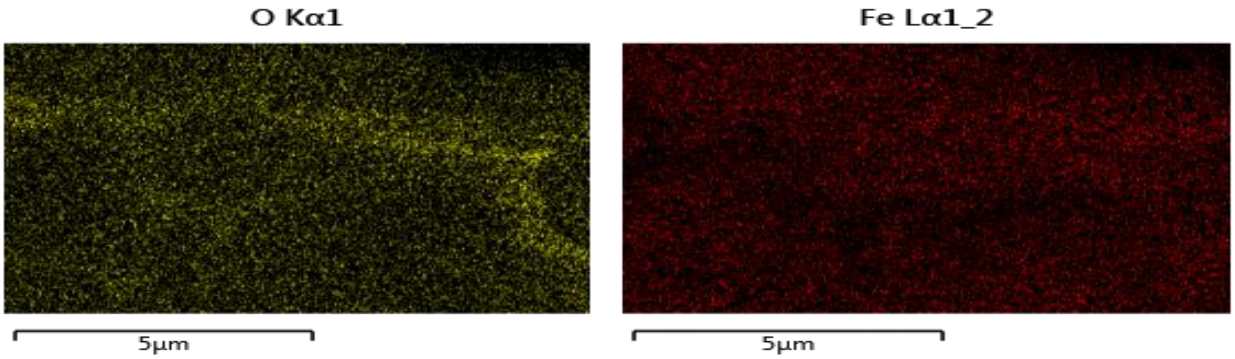

Figure 6 Elemental Profiling of Thin Film Catalysts Using EDX

Assessment of the Catalytic Efficiency of Prepared Thin Films

Photocatalytic efficacy of the iron oxide-coated fluorine-doped tin oxide (FTO) thin films was systematically investigated through the degradation of MB dye under natural sunlight radiation. The progression of the photodegradation process was meticulously monitored using UV/Vis spectrophotometry, wherein the temporal decline in the dye's characteristic absorption peak at 665 nm served as a quantitative indicator of catalytic activity. As the reaction progressed, a gradual fading of the dye's color was observed, indicating effective degradation in existence of iron-oxide covered films. Notable differences in degradation efficacy were recorded over the 1–30 minute sunlight exposure period (Figure 7). Thin films exhibiting a smooth surface and uniform distribution of iron oxide demonstrated enhanced catalytic performance compared to those with less uniform coatings.

Interestingly, the catalytic activity varied with the type and amount of precursor used. Among the samples derived from 4-ferrocenylaniline, those with thinner iron oxide coatings achieved higher degradation rates. This difference is likely due to increased thickness and density of iron oxide layer in other samples, which may have limited active surface area or light penetration.

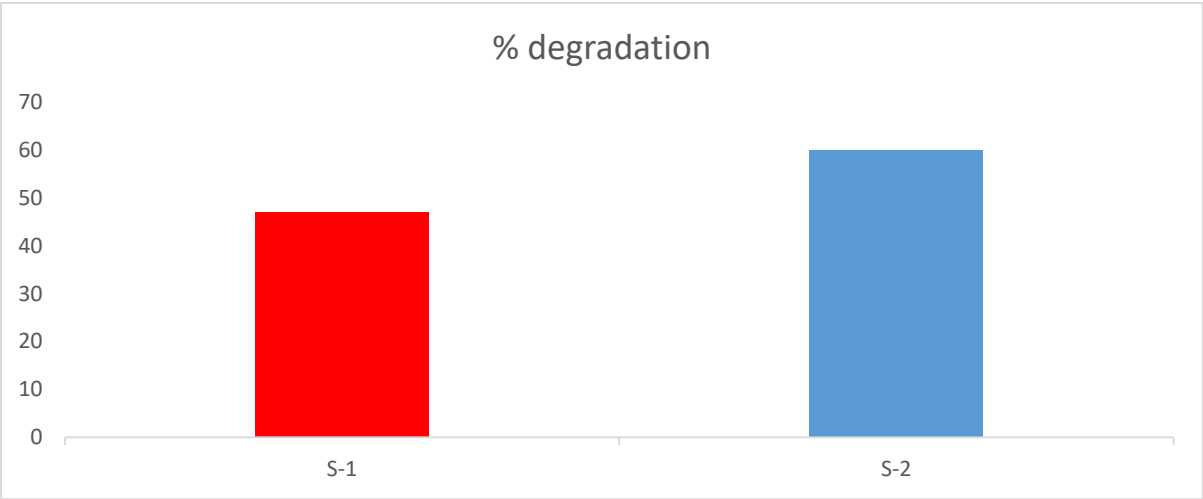

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Figure 7 Photo degradation of Methylene Blue: 30-Minute Sunlight Exposure

Conclusion

Heterogeneous catalysts have attracted considerable interest over their homogeneous counterparts due to their recyclability and associated socioeconomic advantages. In this study, iron oxide thin films were produced on FTO substrate via a pyrolysis-based approach. Comprehensive characterization using EDX, SEM, and UV/Vis spectroscopy verified fruitful fabrication of the thin films. These tailored iron oxide films were then employed as photocatalysts for degradation of Methylene Blue dye, demonstrating significant catalytic efficiency under controlled conditions.

Two distinct iron oxide nanofilms, designated S-1 and S-2, were fabricated from 4-ferrocenylaniline, a precursor known to decompose at temperatures above 400 °C. Upon pyrolysis at 500 °C, FeOOH-based nanostructured films formed on the FTO substrates, as confirmed by SEM analysis. Photocatalytic degradation tests conducted under sunlight irradiation for 30 minutes revealed varying levels of efficiency, with sample S-2 exhibiting the highest degradation percentage. These findings highlight the role of iron oxide thin films as cost-effective and efficient materials for environmental pollutant degradation

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