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Morphological Effect of Iron Oxide Thin Films and its Photocatalytic Potential on the Methylene Blue Dye Degradation Efficiency

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ABSTRACT

In this research, we are going to examine the fabrication and photocatalytic behaviours of iron oxide thin films synthesized by pyrolytic decomposition of ferrocene at varied loadings of precursor solution 0.1 g and 0.125 g, on fluorine-doped tin oxide (FTO) glass substrate. The main purpose of this was to study the morphological changes that occurred in the produced nanofilms and how they related to the photocatalytic degradation rate of methylene blue (MB) dye in the presence of sunlight. The thin films produced were analyzed using the UV/Visible spectroscopy, Fourier- transform infrared spectroscopy (FTIR), energy dispersive X-ray (EDX) and scanning electron microscopy (SEM) techniques. Morphological analysis showed agglomerated forms of nanoparticles which were well dispersed and almost spherical. The 0.125 g ferrocene sample had a smoother surface texture and uniformity with a higher surface area which gave enhanced photocatalytic degradation efficiency than that of the 0.1 g sample. The results indicate the importance of film morphology towards the improvement of photocatalytic ability of iron oxide thin films in degrading dyes and in environment cleanup programs.

Key words: Iron Oxide Thin Films, Active Photo Catalyst, Mb Dye Degradation, Organometallic Precursor, FTO Substrate, Pyrolysis, SEM, FTIR, EDX.

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Introduction:

The field of nanotechnology has become essential and allows tailoring materials at best, at worst, at the atomic, and molecular scale to obtain new physical, chemical, and electronic properties (Manjunatha et al., 2016; Madkour, 2019). Nanomaterials in the shape of metal oxide have become particularly well-known among other nanostructures thanks to their peculiarities of optical and magnetic properties as well as catalytic activity (Chavali and Nikolova, 2019; Ishak et al., 2019). Another example of such material is the iron oxide, because it is inexpensive, non-toxic and has amazing photocatalytic properties to use in the environment (Ali et al., 2016; Duliński-Litewka et al., 2019).

Iron oxide nanomaterials can be synthesized in large numbers using a variety of physical and chemical methods, including sol-gel and co-precipitation, as well as pyrolysis (Campos et al., 2015; Sun et al., 2014). Pyrolysis, in any case, is a cost-efficient and continuous operation, which makes it possible to regulate the formation of particles and deposition of films on the materials (Bang and Suslick, 2010). The resulting thin films may have various morphologies based on the composition and reaction conditions of precursors and these two parameters have a powerful impact on the photocatalytic efficiency (Lee et al., 2011; Nanda, 2015).

Most recently, there has been a great curiosity in photocatalytic degradation of organic dyes including methylene blue (MB), as a green method of wastewater treatment (Fujishima and Honda, 1972; Khakpour et al., 2018). The catalysts are semiconductor materials like TiO_2, ZnO, and $Fe_2 O_3$, which possess a small band gap and are very stable to chemical synthesis (Sivula and Gratzel, 2013). Specifically, iron oxide thin films exhibit great sunlight absorption and effective electron-hole separation on conductive FTO substrates (Aroutiounian et al., 2007; Ramimoghadam et al., 2014).

The current article examines the morphological effect of iron oxide thin films prepared using two distinct ferrocene loadings 0.1 g and 0.125 g through pyrolysis process and their photocatalytic efficacy in dye decomposition of MBs under natural sunlight. The research creates structure activity correlation of the morphology of the surfaces and performance.

Materials:

Chemicals were of an analytical grade and were not further purified. The materials used in the research were, Ferrocene ($C_{10}H_{10}Fe$) as a source of iron, FTO glass substrates, Methylene blue dye as the model pollutant, Ethanol (C_2H_5OH) and distilled water to clean. Prior to filling, all glassware had been immersed in distilled water which had been heated at 95 ^oC.

Method

Direct pyrolysis of ferrocene was done as thin films of iron oxide in a controlled atmosphere furnace. Morphological variation was examined using two different masses of precursors 0.1 g and 0.125 g. The required weight of ferrocene was put into a small crucible and the FTO substrate was placed on the ferrocene conductive side, in downward direction. The assembly was put in an electric furnace and heated at 500 ^oC for 30 minutes. On cooling a coherent film of iron oxide of uniform thickness of

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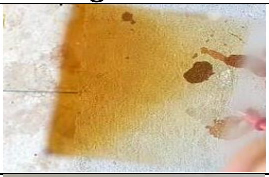
reddish-brown color was formed on the surface of substrate.
The ready samples were marked as S1 and S2.

Composite	Sample code	Original slide
Iron oxide thin film from 0.1g ferrocene	S1	
Iron oxide thin film from 0.125g ferrocene	S2	

Work scheme of the current research.

Step 1	Precursor loadings	0.1 g and o.126 g ferrocene in small crucible
Step 2	Film deposition	Pyrolysis at 550 °C for 30 minutes
Step 3	Cooling	Deposited layers were allowed to cool in air
Step 4	Annealing	Films annealed at 750 °C to improve crystallinity
Step 5	Characterization	Uv/Visible, FTIR, SEM and EDX
Step 6	Application	Photocatalytic degradation of

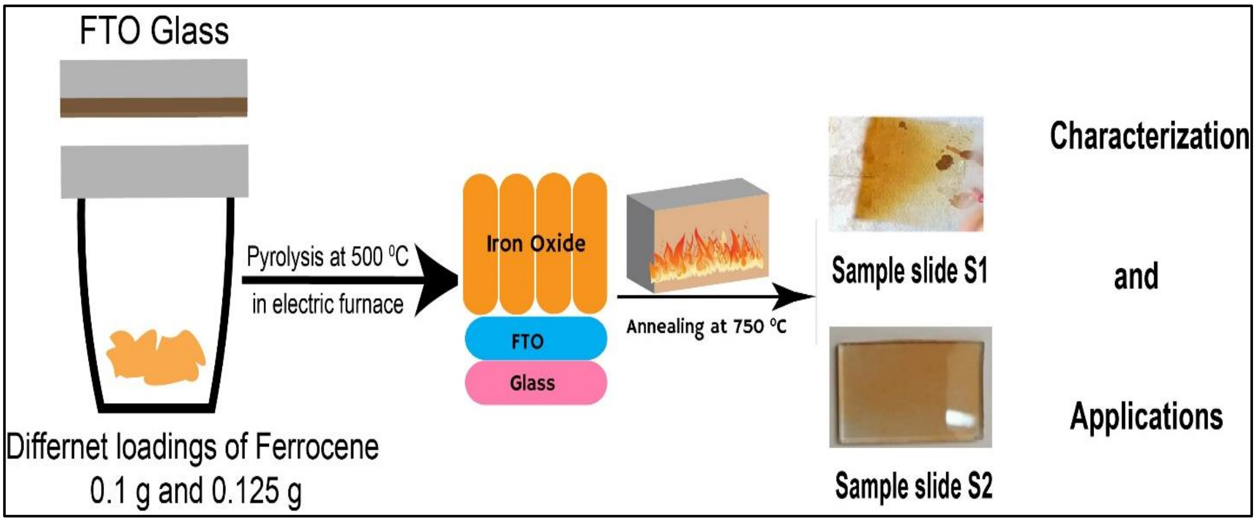


Figure 1 Scheme of synthesis of iron oxide thin-film on FTO substrates

Dye degradation catalytic capacity

In a sunlight driven test, FTO-supported iron-oxide films were used to degrade methylene blue (10 ppm). The film prepared with 0.125 g of ferrocene (S2) degraded about 75 % of the dye within 30 min, whereas the 0.1 g film (S1) achieved roughly 65 %. The superior performance of S2 is attributed to its more homogeneous surface, increased area and enhanced light absorption.

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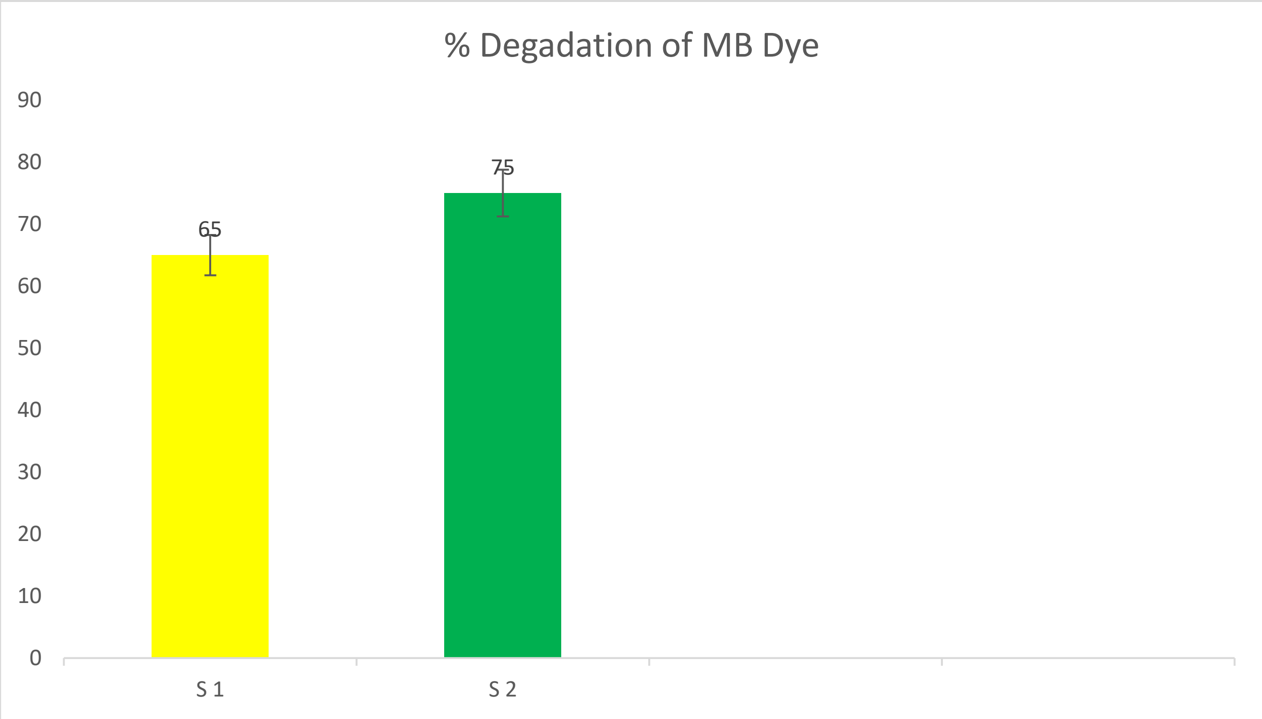

Characterization

The characterization was performed with UV/Visible to monitor kinetics of Methylene Blue degradation and iron oxide optical response. FTIR Spectroscopy, confirmed Fe-O and Fe-OH bonding and metal-ligand transformation. EDX Analysis, Tested elemental composition with dominant elements of Fe and O mapping. SEM, analysis, gave detailed morphological visualization of nanostructure on FTO.

Characterization of iron oxide by FTIR

The FTIR spectrum of the iron oxide thin film displays prominent absorption features at approximately 3422 cm^{-1} and 542 cm^{-1}. The broad band in the 3600–3000 cm^{-1} range is characteristic of -OH groups, indicating both asymmetric and symmetric O–H stretching vibrations. The sharper line near 542 cm^{-1} is attributable to the Fe–O stretching mode. Together, these absorptions reveal the key bonding interactions and structural characteristics of the synthesized iron-oxide film.

Morphological Analysis

The figure 1 shows Scanning Electron Microscope (SEM) bare FTO, glass substrate at 1 □m magnification, prior to depositing an iron-oxide thin film. The SEM micrographs reveal that the FTO glass is composed of a network of interlinked crystalline grains, forming a polycrystalline fluorine-doped tin oxide structure with a uniformly distributed constituent composition.

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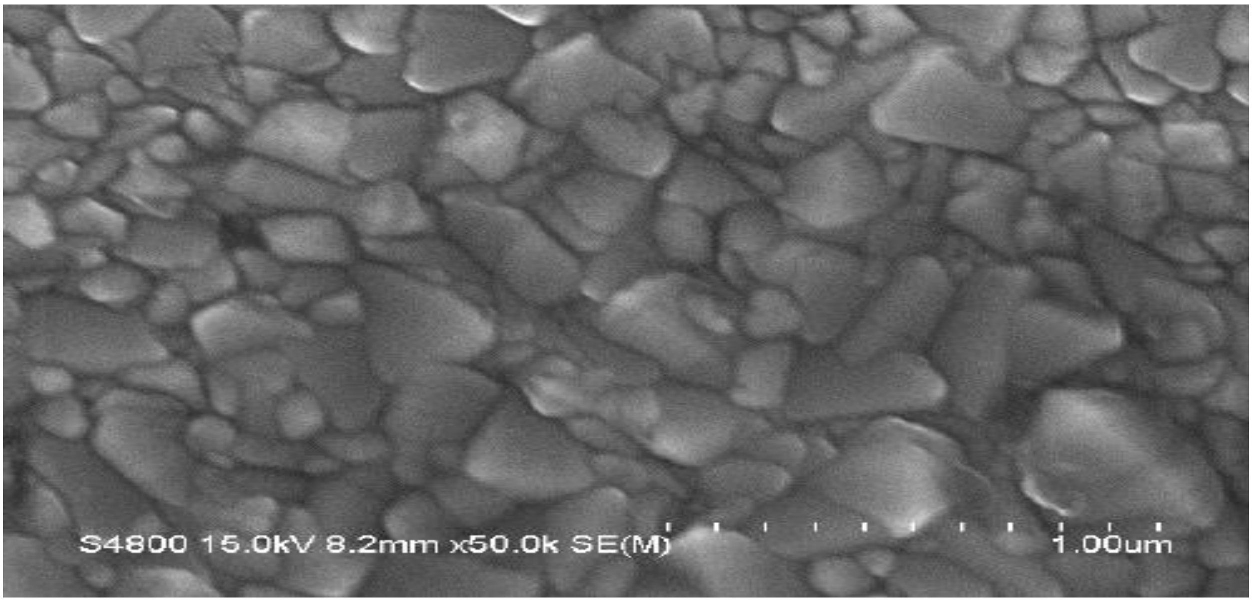

Figure 2: SEM of bare FTO

Sample Slide S1 from 0.1 g Ferrocene

SEM photographs (A1 and A2) captured at different low and high magnifications, indicated homogenous, agglomerated and almost spherical, nanoparticles that were distributed uniformly. The grains were made up of a few nanocrystals aggregated together, which meant that nanostructured iron oxide was made successfully. The surface roughness and particle coverage in the film was moderate.

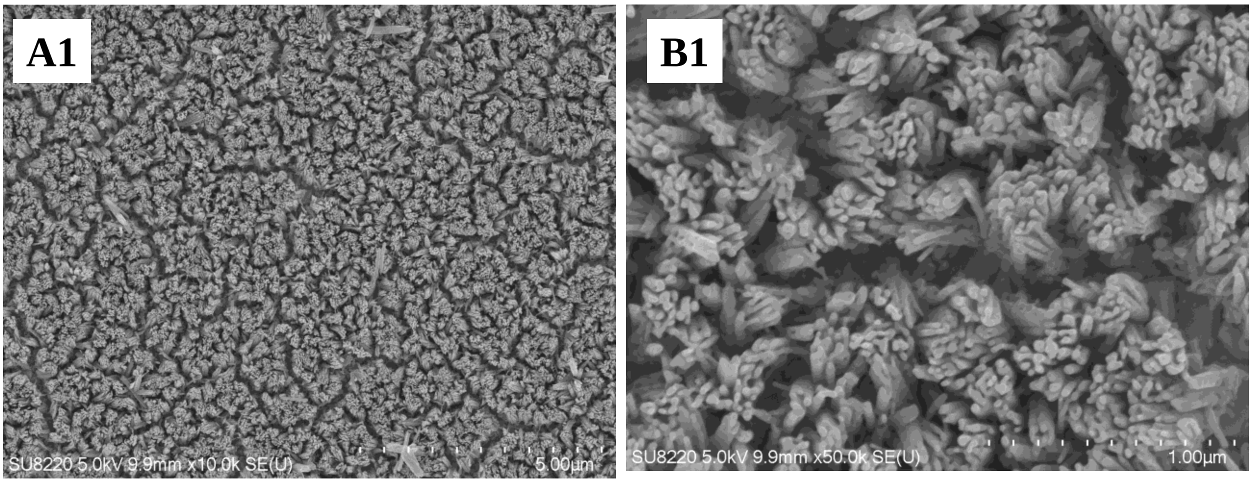

Figure 3 SEM images of 01 g ferrocene loaded Iron Oxide thin film on FTO

Sample Slide S2 from 0.125 g Ferrocene

SEM photographs (A2 and B2) captured at low and high magnifications with higher loading of a precursor produced a denser, smoother feeling using interconnected nanograins in the film. Improved nucleation generated a continuous surface architecture resulting in the increase of active surface sites to generate photocatalysis.

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This architectural enhancement was directly proportional to the rate of the better MB degradation rate in S2 than in S1.

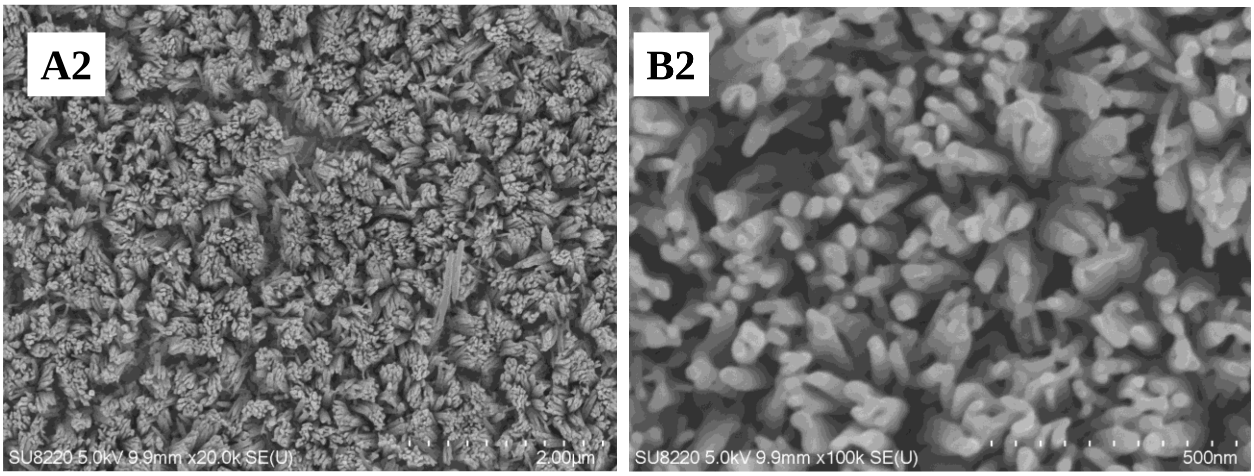
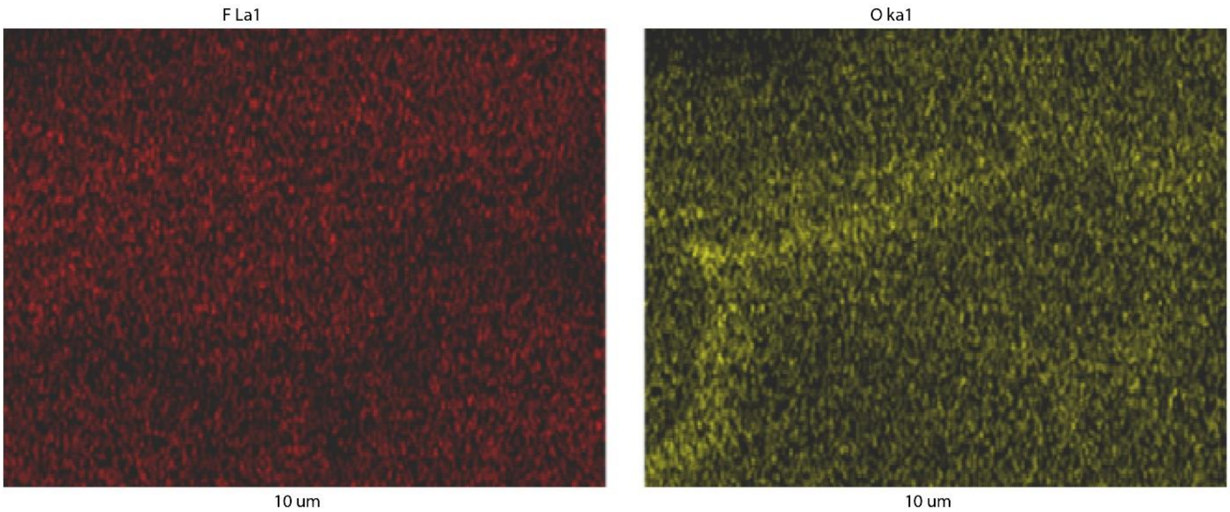


Figure 4: SEM images of 0.125 g ferrocene loaded Iron Oxide thin film on FTO

Elemental Analysis

Figures 5 and 6 display the Fe (Lα1) and O (Kα1) EDS maps of the thin film. The red Fe signal shows a smooth, even spread of iron across the surface, while the green O map indicates a similarly uniform oxygen distribution. Together, these mappings confirm that iron oxide has formed successfully on the FTO substrate, with no detectable regions of depletion or agglomeration.



Figures 5 and 6: Elemental an Analysis of Thin Film Catalysts Using EDX

Conclusion

It is established in the study that it is possible to produce ferrocene based iron oxide thin films on FTO substrate through a simple pyrolysis process. The difference in the amount of the precursors had a great impact on the film morphology and accordingly

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the photocatalytic activity. The film initially coagulated with 0.125 g ferrocene had much of a smoother morphology, increased surface uniformity and increased photocatalytic degradation pace of methylene blue under sunlight. These results indicate that surface morphology is a crucial factor in establishing catalytic activity and make iron oxide thin films one of the potential materials to be used in the cleanup of wastewater in the future.

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