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The Pyrolysis of Ferrocene to Iron Oxide, Pyrolytic Engineering of Photocatalytic Nano films on Conductive Glass

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
Email: bhajan.lal@salu.edu.pk Oric ID 0000-0002-0781-0992

Ajeeb Ali Mandan
Institute of Chemistry, Shah Abdul Latif University, Khairpur, Sindh, Pakistan
Email: ajeebalimandan@gmail.com

Permeshri
Dr MA KAZI Institute of Chemistry, University of Sindh, Jamshoro
Email: permeshri95@gmail.com

Kavita Krishna Morti
Liaqat University of Medical and Health Sciences, Jamshoro
Email: doctor.kavita85@gmail.com

ABSTRACT
The conversion of ferrocene to iron oxide (Fe_2O_3) nano film on the substrate of fluorine doped tin oxide (FTO) was carried through a simple and low cost pyrolysis route. Ferrocene residues were decomposed by heating in air up to 500 ^oC to produce iron oxide thin films. The films prepared were characterized by UV/Visible absorption, FT-IR, EDX and SEM. Images of SEM demonstrate that well-adherent, spherical (Fe_2O_3) nanostructures are homogeneous and gathered into a nest-like network. To determine photocatalytic performance, degradation of methylene blue (MB) in the presence of natural sunlight was observed. Of all the samples, the film produced with 0.125 g of ferrocene (S2) was the most active, reaching 75 percent MB degradation in 30 minutes. These findings indicate that ferrocene pyrolysis is scalable and a process that is related to the environment and can be used to formulate (Fe_2O_3) nanostructured films that can be easily utilized in photocatalytic and environmental remediation.

Introduction
Nanotechnology has emerged as a radium of revolution in the world of materials science because it has made it possible to precisely manipulate matter sizes between 1-100 nm thereby giving systems optical, electronic, and catalytic properties with specifications (Kumar et, al., 2025). The new transition metal oxides are cheap, Earth abundant semiconductors with moderate band gaps and chemistries on their surfaces that are highly adaptable to many different applications in energy conversions, electronics, catalysis, medicine and environment remediation (Manjunatha et al., 2016; Ngo and Van de Voorde, 2014). Iron (III) oxide (Fe_2O_3) is particular due to its non-toxicity, its chemical durability and band gap of approximately 2.0 eV that raise its potential to be

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used in photocatalysis, sensing and solar or chemical energy transformations (Aroutiounian et al., 2007; Ali et al., 2016; Assa et al., 2016).

The biggest problem with utilizing (Fe_2O_3) into a device architecture has been the ability to deposit uniform, pixelated, highly crystalline numbers of films on a conducting medium, like fluorine-doped tin oxide (FTO) glass. Traditional thin-film technologies, such as sputtering, sol-gel processing and pulsed-laser deposition, typically require the use of costly equipment and in the presence of high-vacuums (Hufschmid et al., 2015; Wickman et al., 2017). Pyrolytic deposition, by comparison, is a scalable, low cost option, where oxide formation on the substrate is done by deposition of organometallic precursors that decompose thermally, no vacuum or complicated equipment is required. Ferrocene and its derivatives are particularly attractive single-source precursors. Their well-defined Fe–C bonds allow for controlled thermal decomposition and the formation of homogeneous Fe_2O_3 films (Bang & Suslick, et al., 2010; and Carbajal-Franco et al., 2013).

In this study, we synthesize Fe_2O_3 nanostructured thin films by pyrolytic deposition of ferrocene, onto FTO conducting glass. We investigate how the functional groups on the ferrocene precursors influence (i) decomposition pathways, (ii) film morphology and (iii) photocatalytic performance. The films were characterized by FTIR, SEM, EDX, and UV/Visible spectroscopy and their photocatalytic activity is evaluated through the photodegradation of methylene blue under natural sunlight. This work demonstrates a facile route to tailor Fe_2O_3 photocatalysts via precursor engineering, paving the way for scalable production of high performance nanostructured films on conductive substrates.

Working Scheme

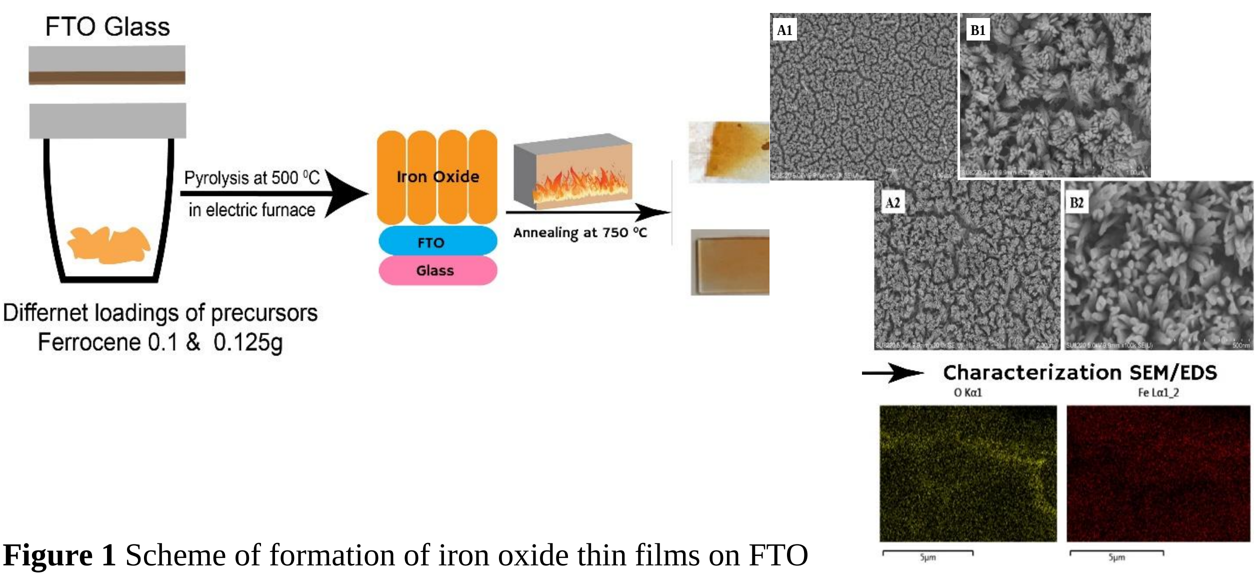

Figure 1 Scheme of formation of iron oxide thin films on FTO substrates

Materials and Method

All the reagents were of analytical grade. Ferrocene ($C_{10}H_{10}Fe$) was arranged from (Sigma Aldrich), while ethanol (C_2H_5OH), ethylene glycol ($C_2H_6O_2$), ammonium hydroxide (NH_4OH) and hydrochloric acid (HCl) were purchased. A transparent conductive substrate was Fluorine-doped tin oxide (FTO) glass.

Before the fabrication, the FTO substrates were ethanol washed and sonication cleaned

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with distilled water and dried at 95 °C. the exact weighed quantities (0.1 g and 0.125 g) of precursor were put in a small crucible and covered by the FTO substrate with its bottom up facing. The pyrolysis was performed at 500 °C in 30 minutes which formed reddish-brown thin films of Fe_2O_3. Then then films were cooled and annealed at 750 °C for 10 minutes.

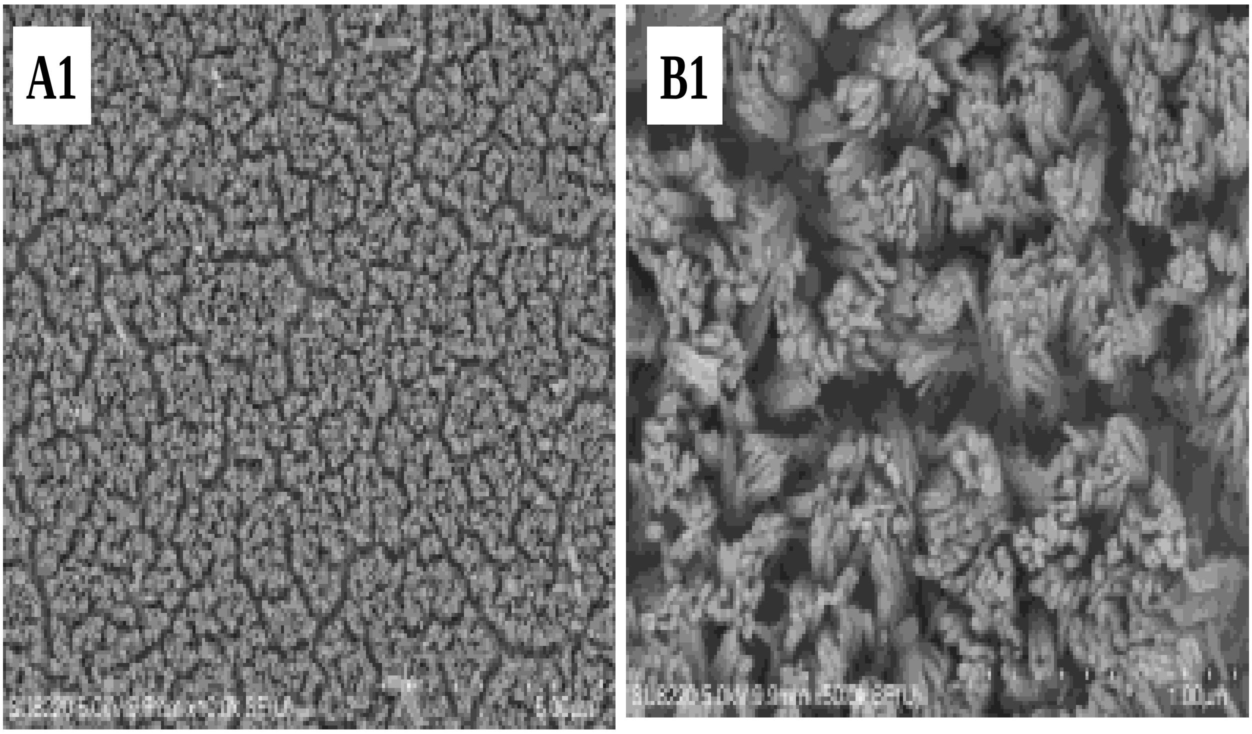
Characterization

The FTIR analysis showed that every organometallic precursor had completely decomposed, establishing Fe–O linkages. Characteristic Fe–O stretching vibrations appeared between 472 and 492 cm^{-1}. In the starting materials, the nitro groups displayed asymmetric and symmetric absorptions at 1549 and 1323 cm^{-1}, respectively; these bands were absent in the final films, confirming that the nitro groups had been completely decomposed.

Whereas UV/Visible spectroscopy was used to gain information for the photocatlytic degradation of methylene blue dye Elemental composition was confirmed using the Energy Dispersive X-ray (EDX) and Scanning Electron Microscopy (SEM) was used to identify the morphology of the Fe_2O_3 films on the surface.

Morphology

The analysis of SEM images at different magnifications indicated that the pyrolytically prepared films of Fe_2O_3 had a homogeneous coverage on the surface and a strong attachment with the substrate. Ferrocene based samples (S1 and S2) were characterized by well-dispersed almost spherical grains. When the precursor mass increased three times 0.1 g to 0.125 g, the topography became smoother and denser which also improved charge movement and photocatalytic properties of the films.



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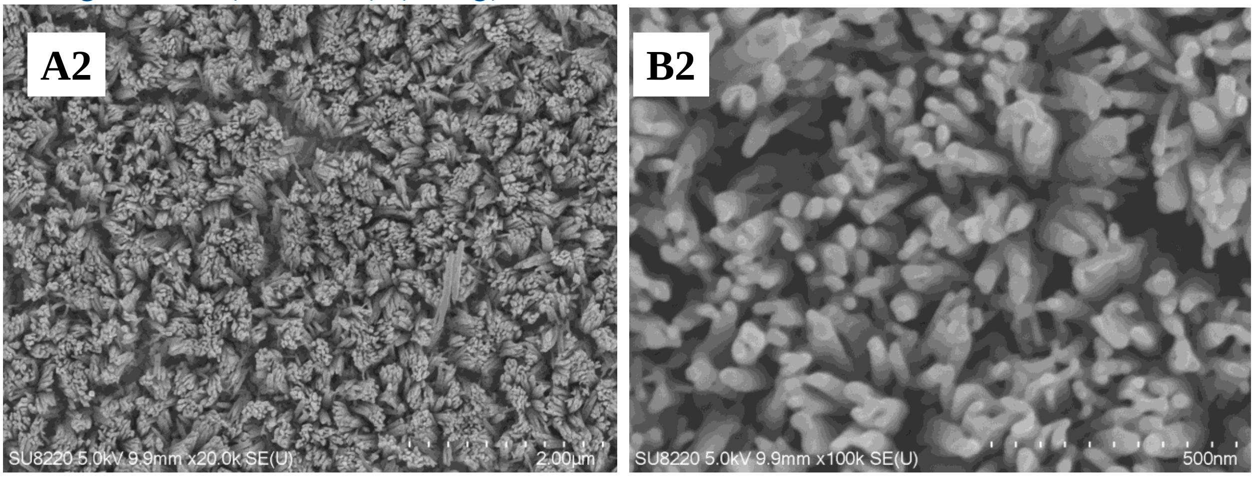

Figure 2: Surface morphology of S1 and S2 at different magnifications

The Energy Dispersive X-ray Spectroscopy (EDX) technique is applicable for analyzing the sample to examine the atomic composition of the product. Spectra EDX proved Fe and O to be the dominant elements.

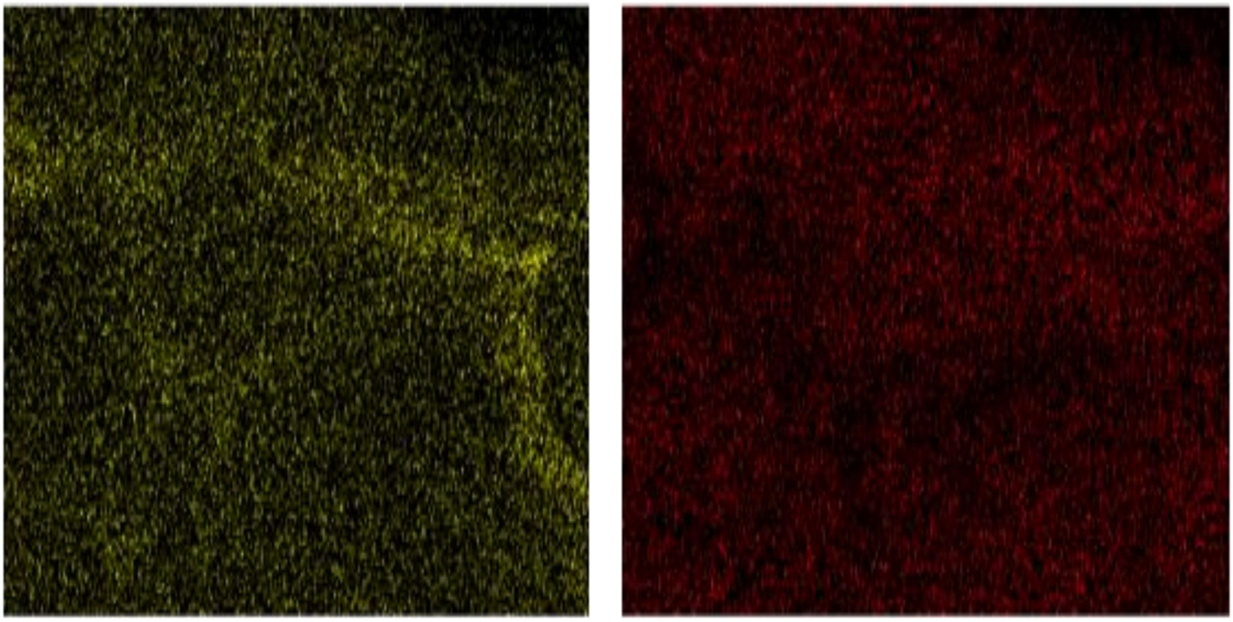

Figure 3: Elemental mapping of O and Fe

The photocatalytic ability of S1 and S2 films

The photocatalytic ability of the FeO_3/FTO films was evaluated with methylene blue (MB) as a model dye, light exposed to the sunlight. Absorbance spectra were taken at different time intervals at 665 nm. Progressive reduction in the absorbance intensity showed that the amount of dye was also degraded. S2 is the most efficient in photocatalyss (mainly reaching 75% in 30 min) due to its uniform nanostructure and ideal film thickness, compared to S1.

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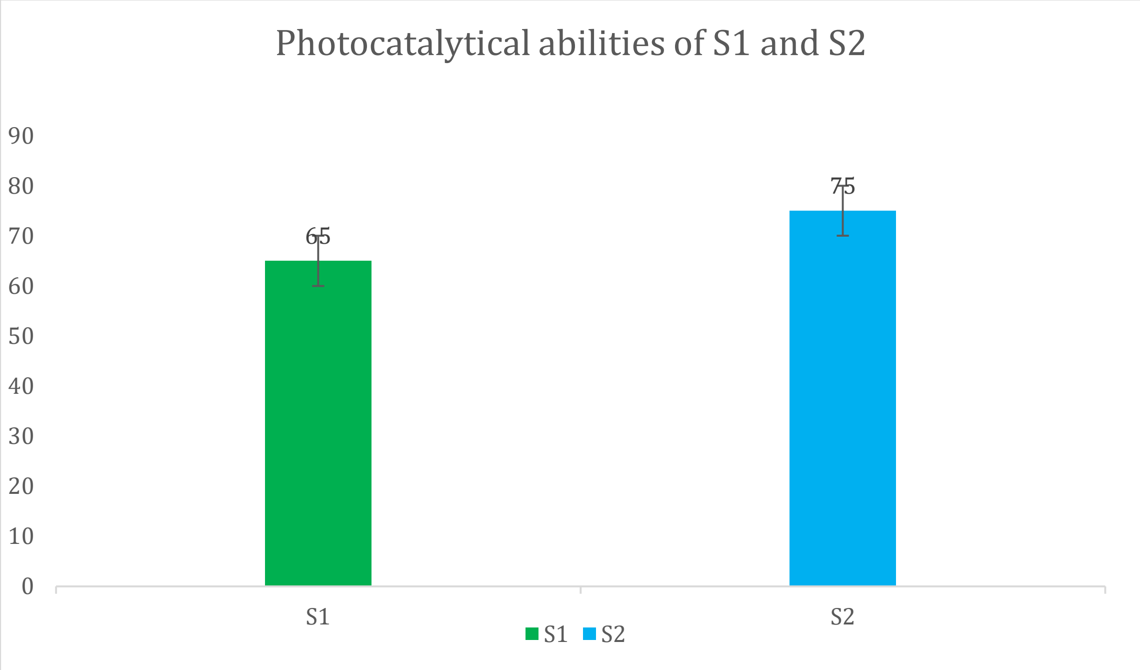

Conclusion

The article offers an effective and widespread way to produce Fe_2O_3 nanostructured thin films through pyrolysis of ferrocene. The method allows conformity to be deposited evenly, adhering efficiently as well as possessing good photocatalytic properties in the presence of sunlight. The concentration of the precursor, as well as morphological thickness, had a profound effect on the catalytic activity. The sample S2 illustrated the maximum methylene blue degradation efficiency which proved that the pyrolytic Fe_2O_3/FTO composites is suitable in remediation of the environment together with solar-driven catalysis.

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