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Optimized Dye Degradation With Functionalized Iron Oxide Thin Films Using Organometallic Derivatives

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
During this research, it was kept in mind, to synthesize and functionalize iron oxide thin films with organometallic, iron containing derivatives and to improve their photocatalytic activity in the degradation of water waste pollutant dyes. The six sample slides from S1 to S6 formed as a result of pyrolysis of three iron containing precursors including ferrocene, 4-nitrophenylferrocene and 4-ferrocenylaniline on fluorine doped tin oxide (FTO) using different loadings. Fourier Transform Infrared Spectroscopy, The Scanning Electron Microscopy, The Energy Dispersive X-ray Spectroscopy and UV/Visible Spectroscopy were employed to characterize and to determine morphological behavior of thin films. Findings were valid to confirm the existence of high purity, nanostructured iron oxide thin films of various morphologies relying on the type of precursors and loading. The photocatalytic activity was analyzed using the degradation of methylene blue dye under sunlight. The Ferrocene derived films (especially S2) had a higher degradation rate (above 70%) after 30 minutes of exposure from solar irradiation. Such an increase of activity was explained by better homogeneity of the surfaces, control of nanograin formation and effective charge transfer upon photo-indication. The results indicate that precursor chemistry provides a significant impact on the physicochemical characteristics of iron oxide thin films and as a result, on their catalytic photocatalytic activity towards the environmental remediation.

Keywords: Iron Oxide Thin Films, Ferrocene Derivatives, Organometallic Precursors, Photocatalysis, Dye Degradation, Fto Substrate, Morphology Of Thin Films

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Introduction

The concept of nanotechnology is a paradigm shift in science and engineering because it enables the manipulation of materials at the molecular and atomic level to produce hitherto unprecedented properties (Manjunatha et al., 2016 and Madkour, 2019). Metal oxides are important building blocks in electronics, catalysis, sensing and environmental remediation among other nanostructured materials because they could have their moderate bandgaps and surface tailored properties (Ngo and Van de Voorde, 2014).

Iron oxide is a transition metal oxide that is quite popular as it possesses semiconducting behaviour, chemical stability, low toxicity and cost-effectiveness (Ali et al., 2016). It can exist as a-Fe_2O_3 (hematite), g-Fe_2O_3 (maghemite) and Fe_3O_4 (magnetite) and all exhibit strong magnetic and optical properties (Dulinska-Litewka et al., 2019). Hematite which is a material with a bandgap of about 2.0 eV has been in the spotlight of solar-driven photocatalytic and photoelectrochemical processes (Sivula and Gratzel, 2013).

Iron Oxide thin films provide a two dimensional platform that has nanoscale characteristics coupled with excellent mechanical and optical stability. Their geometry, crystalline structure and defectology are all open to controlled design using precursor chemistry, deposition pathways (Kirby et al., 2012 and Barranco et al., 2016). One technique, that is currently available, is the simple pyrolysis, which offers a low-cost and scaling method of forming films, which are applicable in large scale (Znaidi et al., 2010).

The organometallic precursor type used has a great influence on the stoichiometry and nanostructure of the final film. The addition of active site groups i.e. nitro (-NO_2) or amino (-NH_2) can tune the electron density and decomposition routes, have an effect on crystal growth and surface activity.

The current work investigates the effects of these organometallic precursors on the process of developing iron oxide thin films and its capacity of catalyzing the process of photocatalytic degradation of the methylene blue (MB) dye in the presence of sun rays. Precursor chemistry and film morphology are bridged with catalytic behaviour to develop cost effective photocatalytic materials to be used in wastewater treatment.

Materials

All reagents were of analysis grade and were not purified further. Chemicals used included, iron (III) chloride ($FeCl_3$), sodium hydroxide (NaOH), ammonium hydroxide (NH_4OH), ammonium chloride (NH_4Cl), sodium nitrate ($NaNO_3$), sodium chlorate ($NaClO_3$), ethyl alcohol (C_2H_5OH), hydrochloric acid (HCl) and ethylene glycol ($C_2H_6O_2$).

The precursors of organometallics were:

Ferrocene ($C_{10}H_{10}Fe$), 4-Nitrophenylferrocene ($C_{16}H_{13}FeNO_2$), 4-Ferrocenylaniline ($C_{17}H_{16}FeN$).

FTO coated glass plates (1 x 1 inch) were used as the source of conductive layer in deposition of the film.

Methodology

Synthesis of 4-Ferrocenylaniline was carried out as: isolation of 4-ferrocenylaniline was completed in two phases including the use of sulphate polymorph. The 4-Ferrocenylaniline was produced in two stages (Ali et al., 2013).

The first step involved the preparation of diazonium salt of 4-nitroaniline which was reacted with sodium nitrite under acidic condition and at 0-5 ^oC. Drop wise addition of the solution into a solution in diethyl ether containing hexadecyltrimethylammonium

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bromide was used in the second step as a phase-transfer catalyst. The product was a 4-nitrophenylferrocene which was isolated and purified.

The second step involved reduction of nitro derivative by hydrazine monohydrate and 10% Pd-C in refluxing ethanol after four hours to form 4-ferrocenylaniline.

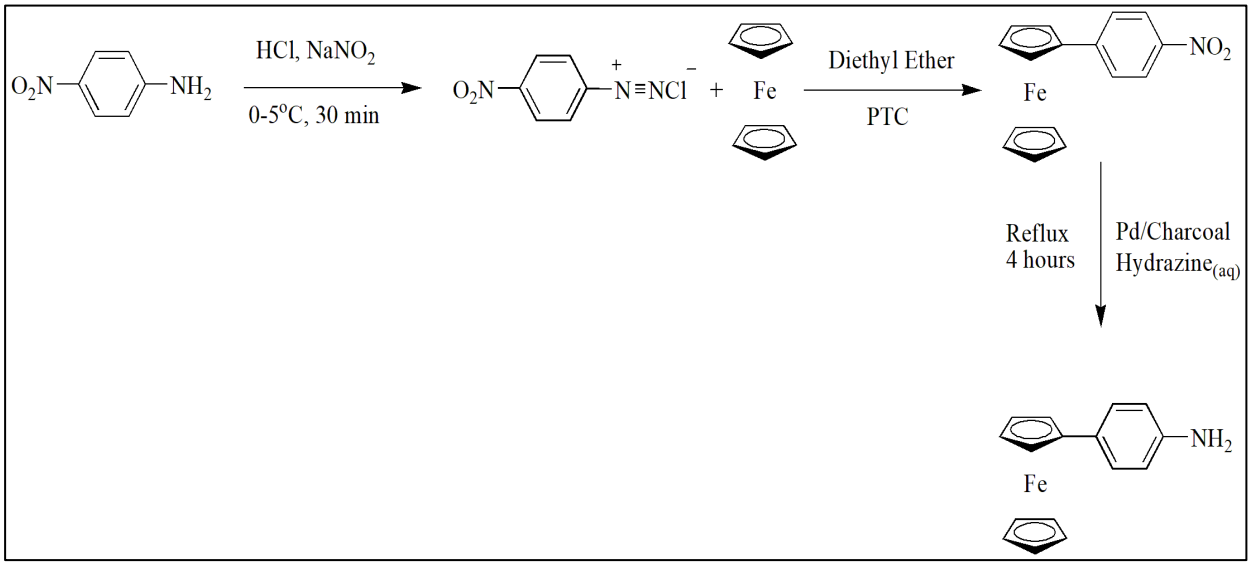

Figure 1; Synthesis of ferrocene derivatives

Preparation of functionalized Iron Oxide Thin Films

The FTO glass substrate was rinsed in ethanol using ultrasonic and dried at 95 ^{o}C. The precursors, ferrocene, 4-nitrophenylferrocene and 4-ferrocenylaniline (0.1 g and 0.125 g respectively) were put in small crucibles in varying amounts (0.1 g and 0.125 g respectively). The FTO slides were placed on top of the crucible with the conducting face in down direction and allowed to be heated in an electric furnace at and above 500 ^{o}C, for 30 minutes. In pyrolysis, the organometallic precursors decomposed to give iron oxide films of reddish -brown coloration equally over the substrate surface. The motion pictures were classified in the following way:

Sample	Precursor Type	Mass (g)
S1	Ferrocene	0.10
S2	Ferrocene	0.125
S3	4-Nitrophenylferrocene	0.10
S4	4-Nitrophenylferrocene	0.125
S5	4-Ferrocenylaniline	0.10
S6	4-Ferrocenylaniline	0.125

Work Scheme

Cleaning of FTO substrate, introduction of organometallic precursor by pyrolysis in an electric furnace at and above 500 ^{o}C, 30 minutes Annealing at 750 ^{o}C for 10 minutes Iron oxide Nanofilm formation on FTO Characterization by FTIR, SEM, EDX, UV/Vis. Photocatalytic analyses with degradation of methylene blue.

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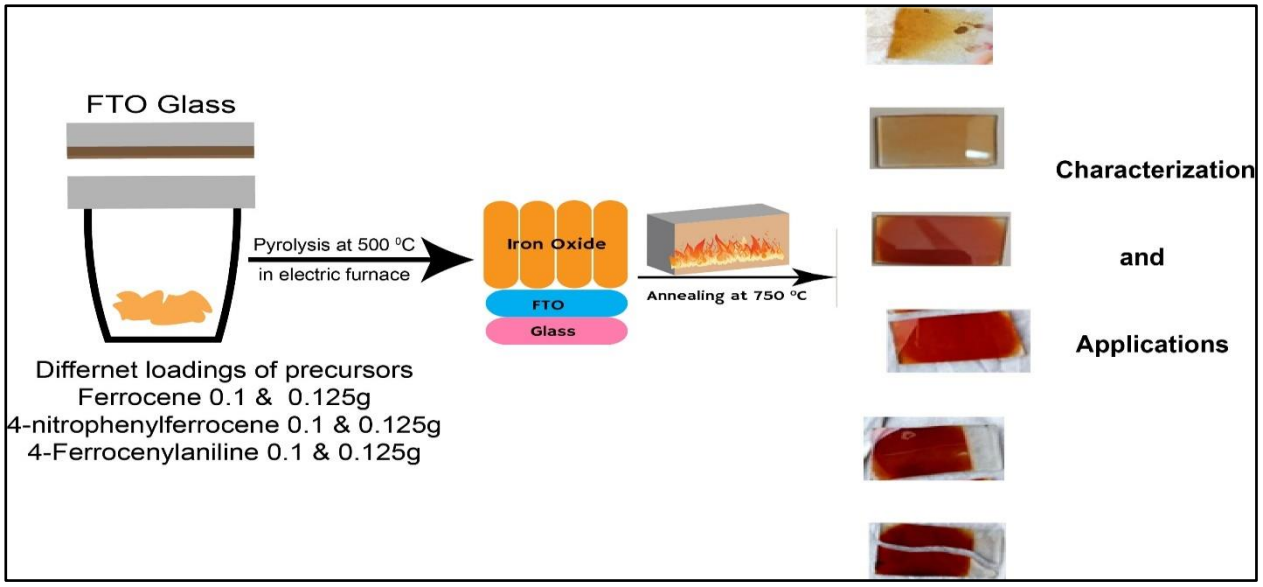

Figure 2; Work Scheme of pyrolysis and fabrication

Characterization

FTIR spectra revealed that all the organometallic compounds were fully decomposed and formed bonds with Fe-O. Distinctive Fe-O positioning vibrations were manifested at 472-492 cm^{-1}. Nitro groups had asymmetric and symmetrical peaks at 1549 and 1323 cm^{-1} respectively in the precursors and which vanished in the final films indicating that oxidation was achieved successfully.

Scanning Electron Microscopy (SEM)

Through SEM micrographs, it was found that films made of ferrocene (S1and S2) made attracted uniform spherical grains in contrast to 4-nitrophenylferrocene (S3 and S4) which formed denser and aggregated nanostructures. The 4-ferrocenylaniline (S5 and S6) films exhibited the platelet-like porous morphology indicating the increased surface area to provide catalytic services. 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 O and Fe to be the dominant elements.

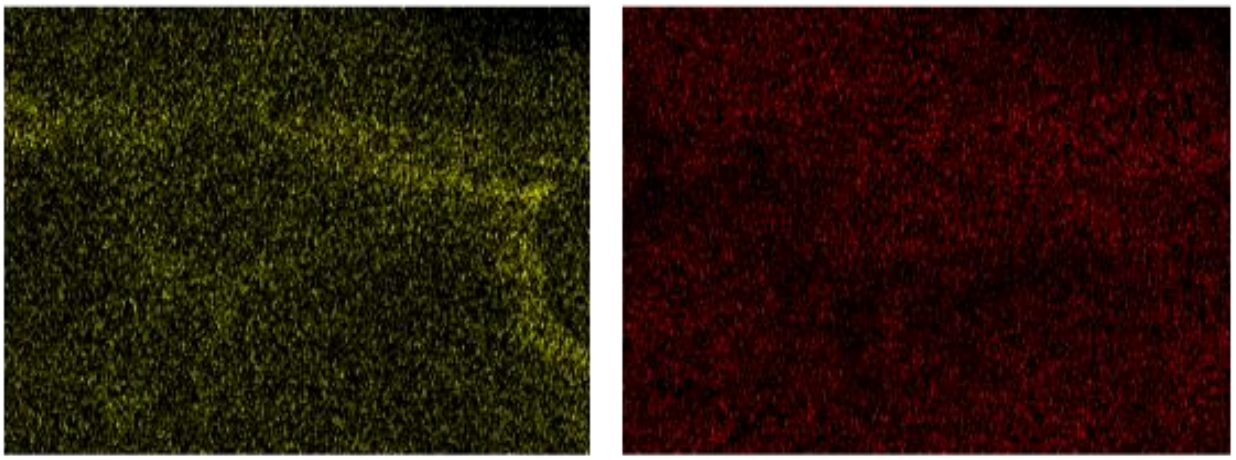

Figure 3 and 4; O and Fe mapping by EDX

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UV-Visible Spectroscopy

The absorption spectrum at 665 nm was attributed to the presence of methylene blue and spectral changes of photodegradation proved that iron oxide films were photocatalysts.

Morphology

Precursor chemistry and mass loading were very important to the morphological features. Ferrocene films exhibited homogeneous and small nanograins. Increasing the precursor mass (S2) gave the formation of thicker films and slightly larger aggregates, which ultimately increased light absorption and actively photocatalyzed.

It resulted in denser clusters with films of 4-nitrophenylferrocene because the electron-withdrawing character of nitro group decreases the rate of decomposition thus causing irregular formation of iron oxide. Conversely, 4-ferrocenylaniline produced more open, porous microstructures which are due to the electron donating amine group, which stabilizes intermediate species in the oxidation process. These morphological differences had a direct effect on the catalytic activity.

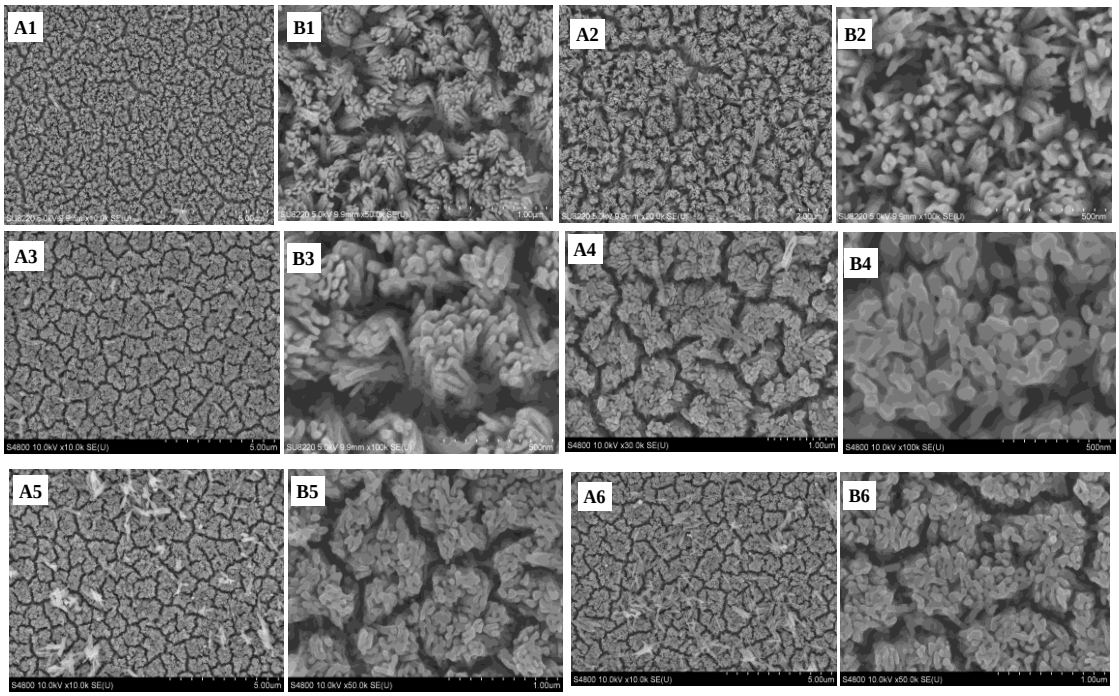

Figure 5; SEM of all sample slides from S1 to S6

Dye Degradation Catalytic Ability.

Experimental Procedure

Solar irradiation was used to degrade methylene blue (MB) dye by way of photocatalysis. The FTO films with iron oxide coating were put in 50 mL of 10^{-5} M MB solution. The mixture was stirred and the absorbance of the solution at 665 nm was recorded at 5 minute intervals in the span of 30 minutes using a UV/Vis spectrophotometer.

Results and Discussion

The sharp reduction in the absorption intensity of 665 nm was an indication of good degradation of MB. In the set of samples, the degradation efficiency was in the following

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order

S2 > S1 > S5 > S6 > S4 > S3

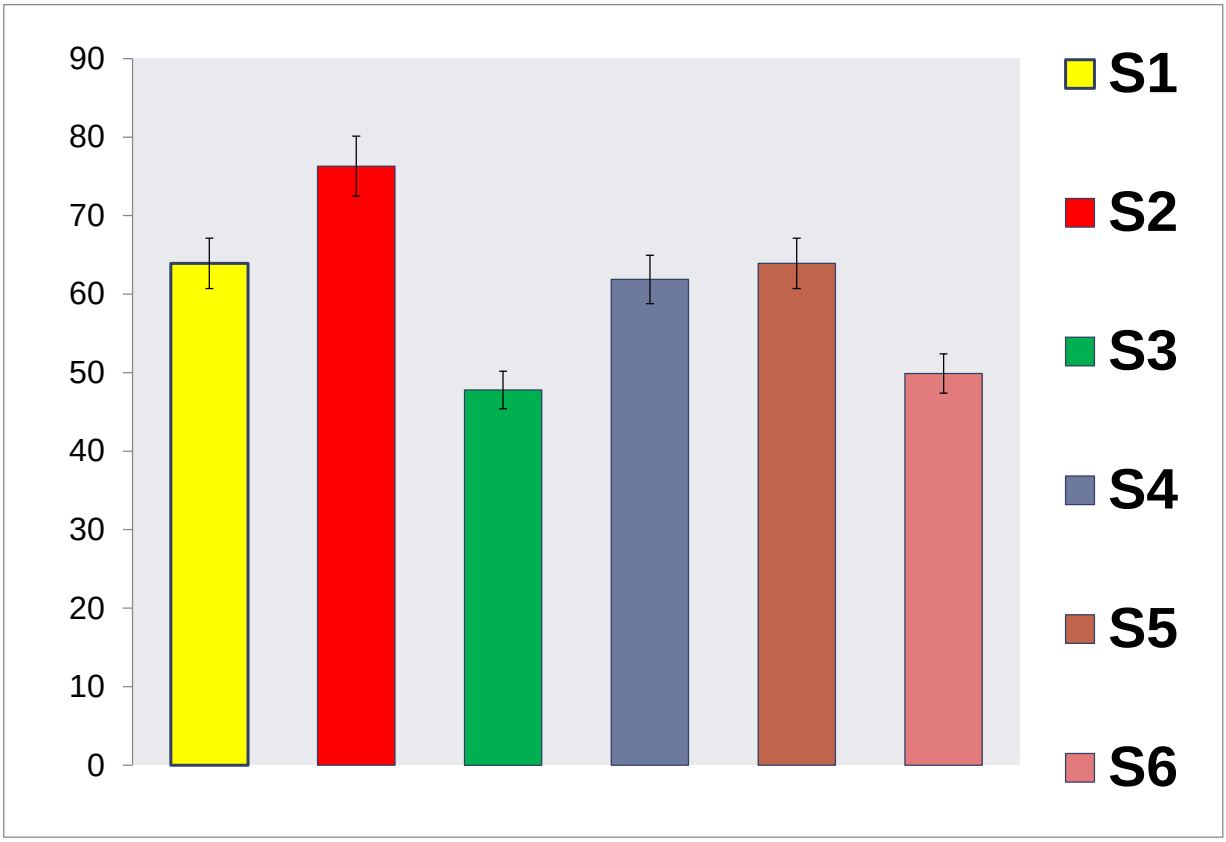
S 2 (ferrocene, 0.125 g) thin film almost degraded methylene blue dye almost by 80 percent in 30 minutes. The increased performance can be explained by:

Large surface smoothness and nano-porosity;

Extraction of photons and charge carriers;

Choosing optimum loading of iron oxide to eliminate loss through light scattering

The moderate efficiency of S5 (4-ferrocenylaniline, 0.1 g) (approximately 70 percent) can be attributed to the porous morphology, which permits active dye adsorption sites. On the other hand, S3 and S4 (4-nitrophenylferrocene) showed less activity because of the electron withdrawing influence of the nitro group, which hinders the oxidation of Fe and lowers the activity of the surface.



Conclusion

This work has shown that the choice of precursors in the manufacture of organometallic thin films is a decisive factor in the ways that iron oxide thin films are manufactured, in terms of morphology and photocatalytic performance. The fabrication of iron oxide thin films by pyrolysis technique on FTO substrates was more uniform and pure as attested by the FTIR, SEM and EDX. The highest photocatalytic activity of her precursors, which was photocatalytic degradation of methylene blue (approximately 80 percent in 30 minutes of sunlight), was obtained with ferrocene derived films, especially S2 (0.125 g). The amine-functionalized precursor (4-ferrocenylaniline) gave rise to porous films, which was generated with moderate efficiency and the nitro-functionalized precursors made denser, less active surfaces. These results support the significance of precursor design in producing effective iron oxide based photocatalysts in the treatment of the

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environment pollutants particularly when applied in the treatment of dye-contaminated wastewater. Future research can be done on incorporation of dopants, design of heterojunction and long-term stability with an aim of improving photocatalytic activity.

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