Policy Research Journal
ISSN (E): 3006-7030 ISSN (P) : 3006-7022 Volume 3, Issue 10, 2025

FABRICATION, CHARACTERIZATION AND PHOTOCATALYTIC CHARACTERISTIC OF NANOSTRUCTURED IRON OXIDE THIN FILMS BY PYROLYSIS OF 4-FERROCENYLANILINE

Rajesh Kumar[*1], Bhajan Lal[2], Ajeeb Ali Mandan[3], Kavita Krishna Morti[4]

[*1,2,3]*Institute of Chemistry, Shah Abdul Latif University, Khairpur, Sindh, Pakistan*
[4]*Liaqat University of Medical and Health Sciences, Jamshoro*

[*1]rajeshkumar@salu.edu.pk, [2]bhajan.lal@salu.edu.pk, [3]ajeebalimandan@gmail.com,
[4]doctor.kavita85@gmail.com

[*1]Oric ID 0000-0002-0781-0992

DOI: https://doi.org/10.5281/zenodo.17461294

Keywords
4-ferrocenylaniline, Iron oxide thin films, Photocatalysis, FTO substrate, Pyrolysis, SEM, FTIR, EDX.

Article History
Received: 08 September 2025
Accepted: 15 October 2025
Published: 28 October 2025

Corresponding Author: * Rajesh Kumar

Abstract
The paper is concerned with the synthesis and the characterization of nanotrapped iron oxide thin films obtained by pyrolytic degradation of 4-ferrocenylaniline over fluorine-doped tin oxide (FTO) surfaces using 4-Ferrocenylaniline as iron source. The synthesized films have unique nanostructured morphologies and excellent photocatalytic action hence, promise in the applications of the environment and optoelectronics. The 0.1 g and 0.125 g precursor were subjected to pyrolysis at 500 °C to make homogeneous and adhesive layers. The presence of pure iron oxide nanofilms was confirmed by the use of Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX) and Fourier Transform Infrared Spectroscopy (FTIR). The degradation of the methylene blue in sunlight under the photocatalytic catalysis of the dissolved methylene blue showed that the 0.1 g sample S1 was more efficient in degradation the dye because it had a thinner, porous morphology. The paper emphasizes the prospects of 4-ferrocenylaniline as a new organometallic precursor to provide economical synthesis of catalytic nanofilms regarding an easy simple pyrolytic process.

1. INTRODUCTION

Nanotechnology has become indispensable for tailoring the structural and functional properties of materials down to the atomic and molecular scales, thereby enabling the emergence of novel physical, chemical, and electronic behaviours (Manjunatha et al., 2016; Madkour et al., 2019). Among the diverse classes of nanostructures, metal-oxide nanomaterials have garnered particular interest due to their distinctive optical, magnetic, and catalytic characteristics (Chavali & Nikolova, 2019). Iron oxides, in particular, stand out as inexpensive, non-toxic, and highly effective photocatalysts for environmental remediation (Ali et al., 2016;)

Iron-oxide nanomaterials can be produced in bulk via several physicochemical routes, including sol-gel synthesis, co-precipitation, and pyrolysis (Campos et al., 2015; Sun et al., 2014). Pyrolysis is especially attractive because it is cost-efficient, amenable to continuous operation and allows precise control over particle nucleation, growth and film deposition on substrates (Bang & Suslick, 2010). The resulting thin films can exhibit a wide variety of morphologies that depend on precursor composition and reaction parameters; these morphological traits exert a powerful influence on photocatalytic performance (Lee et al., 2011; Nanda, 2015).

Recently, there has been intense interest in the photocatalytic degradation of organic dyes, most notably methylene blue, as a green strategy for wastewater treatment (Fujishima & Honda, 1972; Khakpour et al., 2018). Semiconductor catalysts such as TiO_2, ZnO, and Fe_2O_3, which possess small band gaps and high chemical stability, are widely employed for this purpose (Sivula & Gratzel, 2013). Iron-oxide thin films, when deposited on conductive fluorine-doped tin oxide (FTO) substrates, exhibit pronounced sunlight absorption and efficient electron-hole separation, making them particularly attractive for solar-driven degradation of pollutants (Aroutiounian et al., 2007; Ramimoghadam et al., 2014).

The present study investigates the fabrication, structural characterization and photocatalytic behaviour of nanostructured iron-oxide thin films obtained by pyrolyzing 4-ferrocenylaniline. Two different sample loadings (0.1 g and 0.125 g) were employed to explore the influence of precursor concentration on film morphology and subsequent photocatalytic activity in MB degradation under natural sunlight. By correlating morphology with photocatalytic performance, this work aims to elucidate structure-activity relationships that can guide the design of efficient iron-oxide-based photocatalysts.

Materials

The reagents were of the analytical quality. The raw materials used were, 4-ferrocenylaniline ($C_{17}H_{16}FeN$), ethanol, ammonium hydroxide (NH_4OH), hydrochloric acid (HCl), and distilled water. Such conductive substrates were fluorine-doped tin oxide (FTO) coated glass slides. The slides were cut in 1 x 1 inch squares, ultrasonically cleaned with ethanol and dried at 95 °C prior to deposition.

Method

In each experiment, 0.1 g and 0.125 g of 4-ferrocenylaniline were sequentially weighed and put in to a clean small crucible. The conductive side of the FTO substrate was placed facing downwards over the crucible to be in direct contact with vapors generated in the process of pyrolysis. The mixture was fired in an electric furnace at 500 °C for a period of 30 minutes. Iron oxide vapors were formed during heating by thermal decomposition of 4-ferrocenylaniline and condensed on the FTO surface forming a reddish-brown thin film. The slides were annealed at 750 °C to improve surface morphologies and smooth crystalline growth.

Iron Oxide Thin film is prepared by fabrication.

Pyrolysis method enables the direct conversion of the organometallic precursor to oxide films to take place on the substrate. The amine group of 4-ferrocenylaniline enhances uniform nucleation by coordinating with centres of iron during decomposition. The 0.1 g precursor (sample S5) had produced a thin and porous film whereas the 0.125 g precursor (S6) had produced a thicker film with increased particle agglomeration. The films used in both the films were well-bounded and were very stable in the ambient conditions.

Work Scheme

Washing and preparations of FTO substrates. Weighing of 0.1 g and 0.125 g 4-ferrocenylaniline. Pyrolysis at 500 °C for 30 minutes in an electric furnace. Annealing at 760 °C for 10 minutes. FTIR, SEM and EDX characterization.

Policy Research Journal
ISSN (E): 3006-7030 ISSN (P) : 3006-7022 Volume 3, Issue 10, 2025

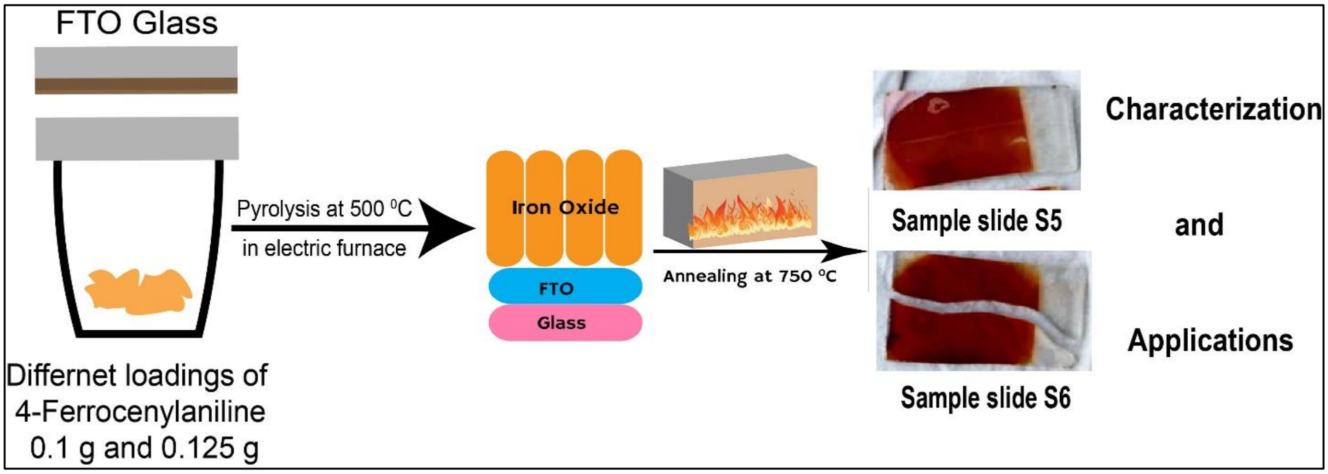
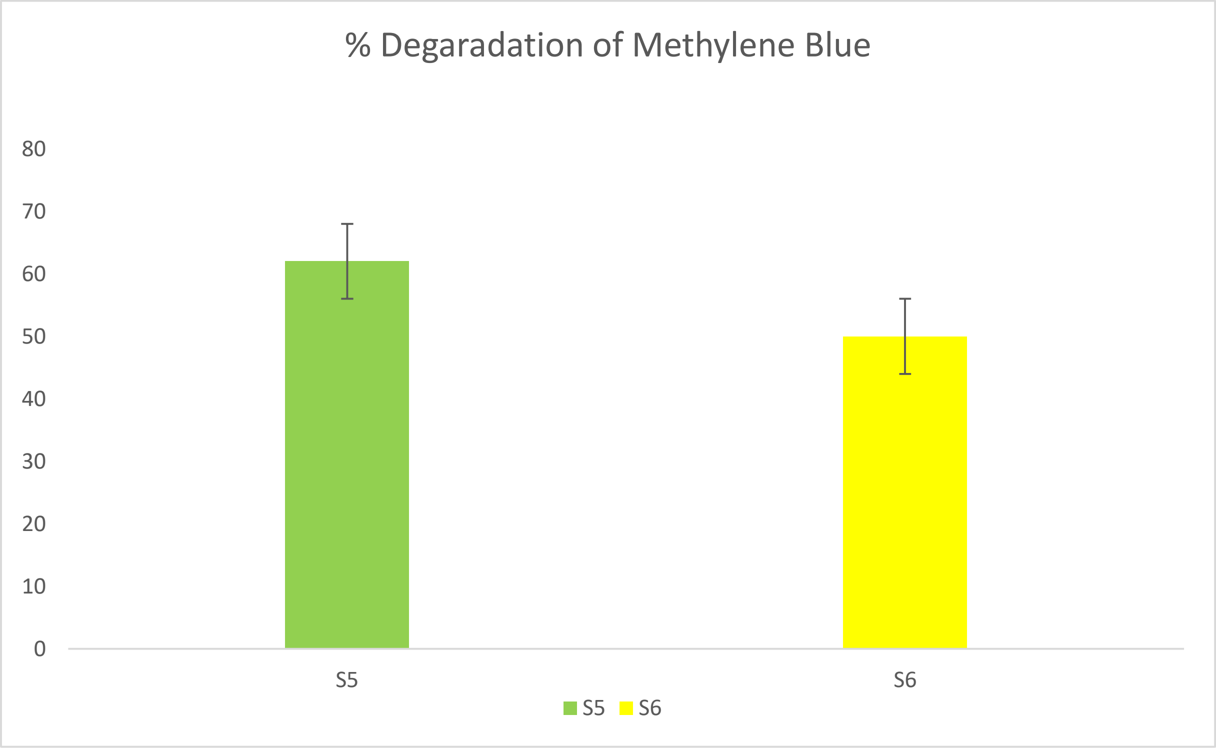


Figure 1; Working scheme of synthesis of iron oxide thin-film on FTO substrates

Testing of photocatalytic activity of methylene blue in the presence of sunlight.

Dye Degradation Catalytic Ability.

Methylene blue dye (10mg/L) was used to study photocatalytic performance. Every thin film sample was placed in a 50 mL of dye solution and subjected to sunlight being stirred continuous using magnetic stirrer. The degradation was monitored at UV/Vis spectroscope which recorded 665 nm absorption band initially. The absorbance intensity was decreasing progressively with time implying that the dye was decomposing. This is because the 0.1 g film was found to be more active since it had a higher surface area and more light penetrated through the porous structure.



Characterization

The FTIR spectra established the development of Fe-O stretching vibration around the 472-492 cm^{-1}. SEM micrographs indicated clear cluster of platelets on the nanoscale in heaps. The EDX spectra indicated that only peaks of iron (Fe) and oxygen (O) were obtained Figure 2 displays a scanning electron microscopy (SEM) image of the bare fluorine-doped tin oxide (FTO) glass substrate at a 1 µm magnification, taken before the deposition of the iron-oxide thin film. The and confirms that there has been a stoichiometric formation of Fe_2O_3 with limited impurities. The films were both optical and mechanically adherent on FTO surface.

Morphological Analysis

micrographs show that the FTO glass is composed of a network of interconnected crystalline grains, forming a uniformly distributed polycrystalline fluorine-doped tin oxide structure.

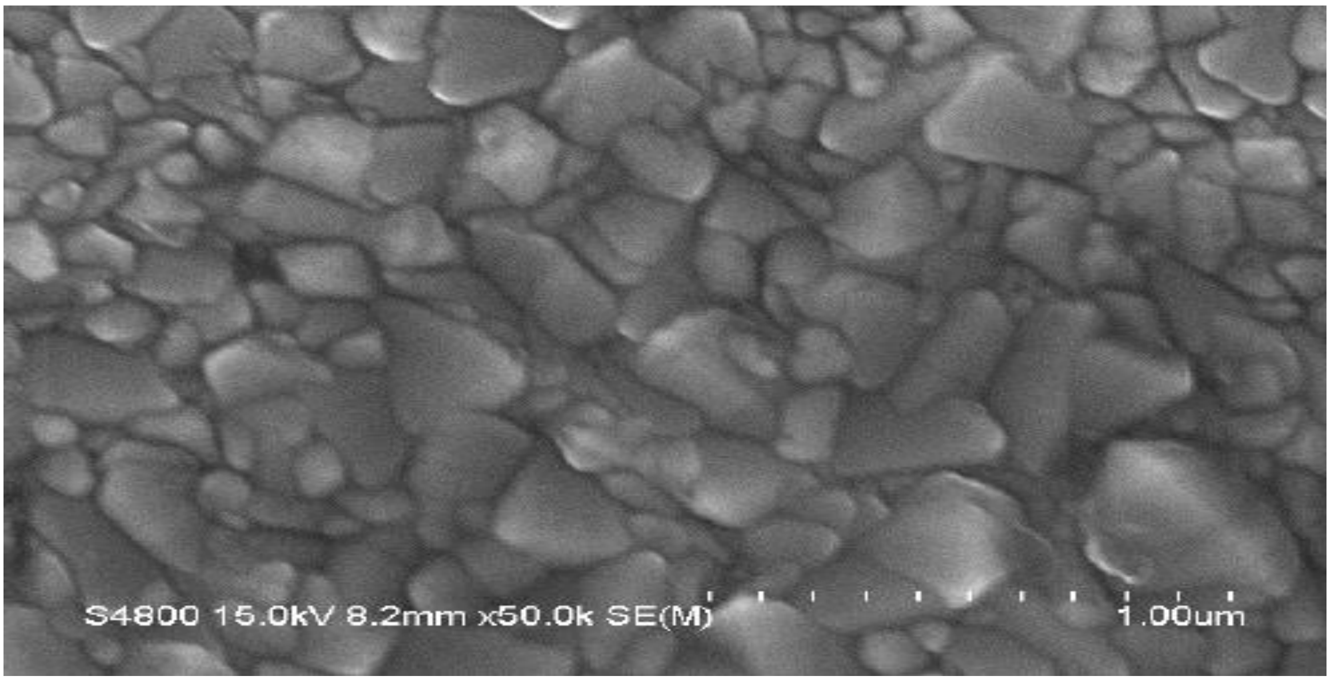


Figure 2: SEM of bare FTO

The morphology with Ferrocene 0.1 g and 0.125 g

SEM images captured on different zooming have shown that the examined fabricated with 0.1 g of 4-ferrocenylaniline (S5) exhibited a platelet structure of porous structure evenly-distributed on the FTO substrate. The nanoscale grains were assembled into loosely packed aggregates thereby increasing sites of reactive surfaces with which to provide photocatalysis. S6 (0.125 g) film had more compact stacks of platelet with a nanocrystal into the film. The high uniformity of the film was not associated with high catalytic performance due to the reduced penetration of light its thickness provided.

Policy Research Journal
ISSN (E): 3006-7030 ISSN (P) : 3006-7022 Volume 3, Issue 10, 2025

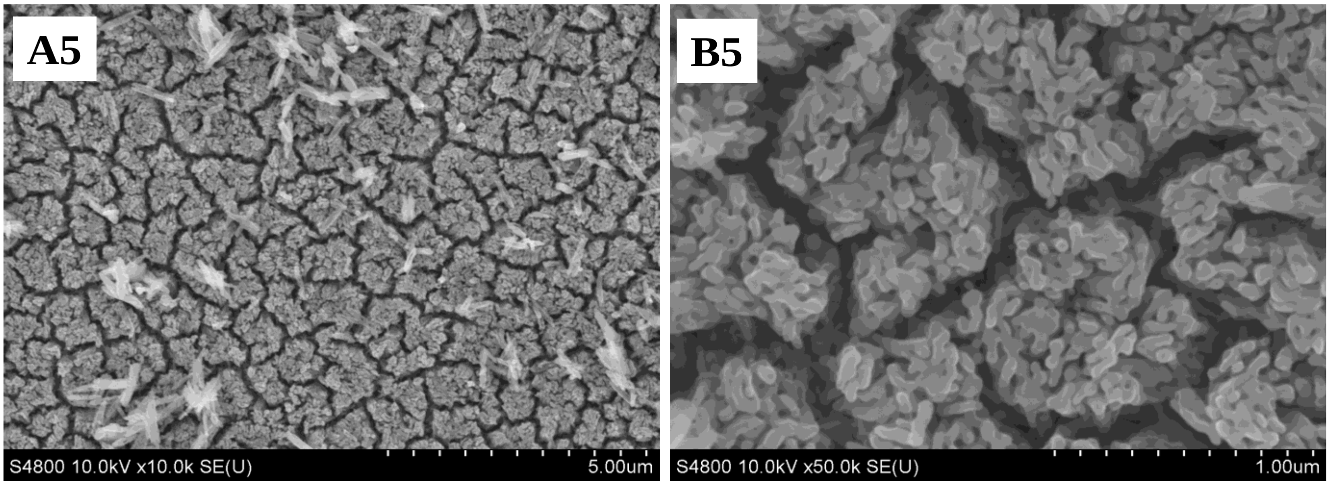


Figure 3: SEM images of 0.1 g 4-ferrocenylaniline loaded Iron Oxide thin film on FTO

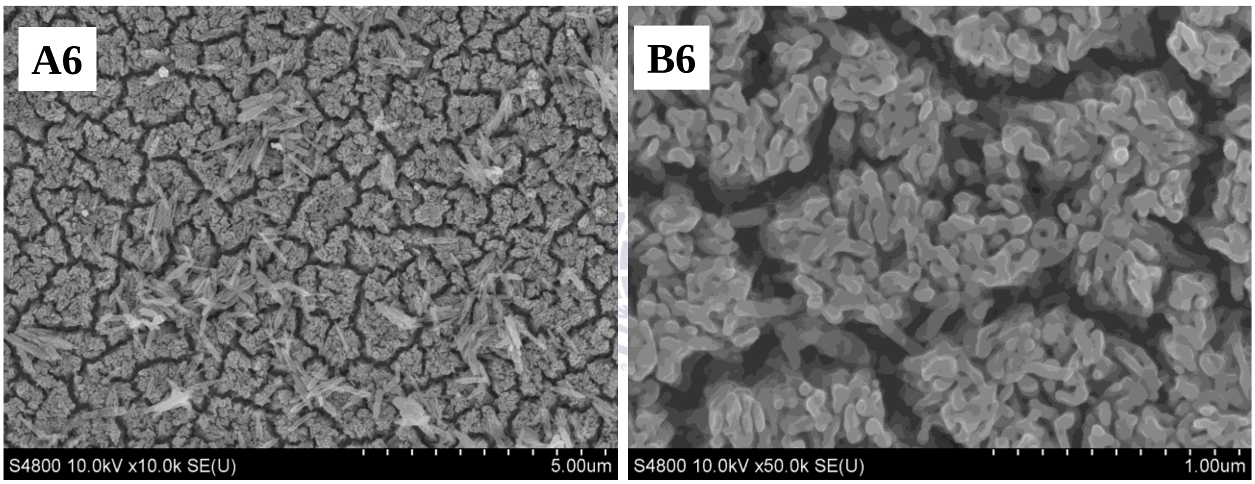
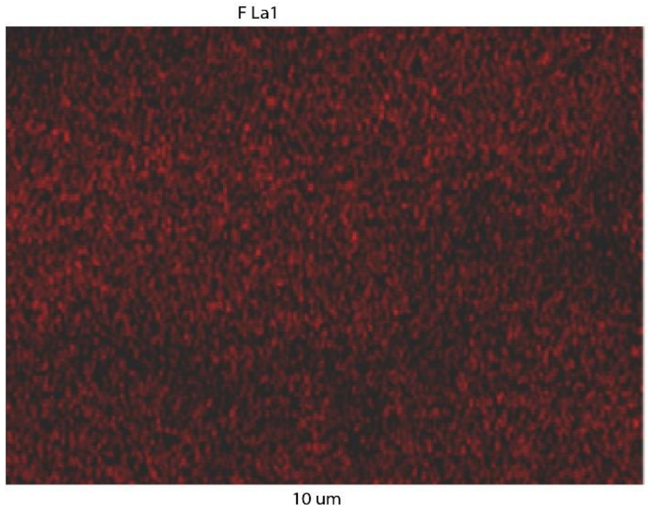
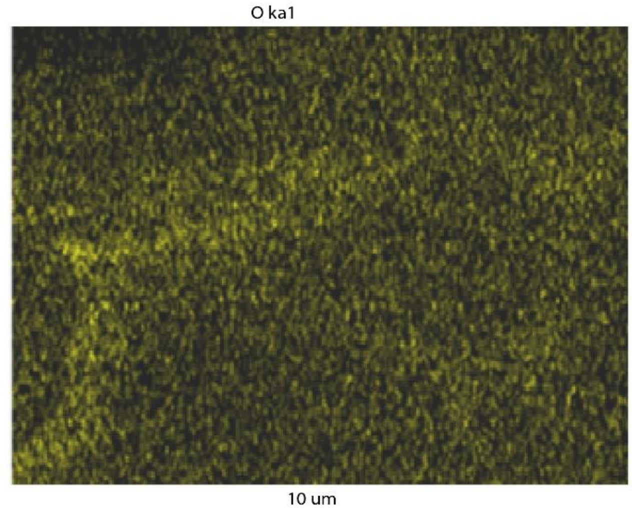


Figure 4: SEM images of 0.125 g 4-ferrocenylaniline loaded Iron Oxide thin film on FTO

Elemental Analysis

Figures 5 and 6 present the Fe (Lα1) and O (Kα1) EDS maps of the thin film. The red Fe signal reveals a smooth, homogeneous distribution of iron across the surface, while the green O map shows a similarly uniform oxygen coverage. Together, these data confirm the successful formation of iron oxide on the FTO substrate, with no detectable depletion or agglomeration zones.





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

Conclusion

The pyrolysis of 4-ferrocenylaniline is an easy, inexpensive and repeatable method of creating nanostructured iron oxide thin coatings. The amine group on the precursor improves the interaction and uniform nucleation, the films of the amine group have been observed to exhibit high morphological uniformity and have a high photocatalytic activity. The 0.1 g film showed the highest level of degradation of the dye because of its thin and porous structure that enabled a good deal with the photon absorption and the transfer of charge. The research supports the finding that 4-ferrocenylaniline is a good organometallic precursor that can be readily used to achieve effective photoactive coatings in remediation of the environment and solar applications.

REFERENCES

Ali, A., Zafar, H., Zia, M., ul Haq, I., Phull, A. R., Ali, J. S., & Hussain, A. (2016). Synthesis, characterization, applications, and challenges of iron oxide nanoparticles. *Nanotechnology, science and applications*, 49-67.

Ali, S., Badshah, A., Ataf, A. A., Lal, B., & Khan, K. M. (2013). Synthesis of 3-ferrocenylaniline: DNA interaction, antibacterial, and antifungal activity. *Medicinal Chemistry Research*, *22*, 3154-3159.

Assa, F., Jafarizadeh-Malmiri, H., Ajamein, H., Anarjan, N., Vaghari, H., Sayyar, Z., & Berenjian, A. (2016). A biotechnological perspective on the application of iron oxide nanoparticles. *Nano Research*, *9*, 2203-2225.

Boholm, M. (2016). The use and meaning of nano in American English: Towards a systematic description. *Ampersand*, *3*, 163-173.

Camacho-Flores, B., Martínez-Álvarez, O., Arenas-Arrocena, M., Garcia-Contreras, R., Argueta-Figueroa, L., De La Fuente-Hernández, J., & Acosta-Torres, L. (2015). Copper: synthesis techniques in nanoscale and powerful application as an antimicrobial agent. *Journal of Nanomaterials*, *16*(1), 423-423.

Campbell, Z. S., Bateni, F., Volk, A. A., Abdel-Latif, K., & Abolhasani, M. (2020). Microfluidic synthesis of semiconductor materials: Toward accelerated materials development in flow. *Particle & Particle Systems Characterization*, *37*(12), 2000256.

Campos, E. A., Pinto, D. V. B. S., Oliveira, J. I. S. d., Mattos, E. d. C., & Dutra, R. d. C. L. (2015). Synthesis, characterization and applications of iron oxide nanoparticles-a short review.

Journal of Aerospace Technology and Management, 7, 267-276.

Carenco, S., Portehault, D., Boissiere, C., Mezailles, N., & Sanchez, C. (2013). Nanoscaled metal borides and phosphides: recent developments and perspectives. *Chemical Reviews, 113*(10), 7981-8065.

Chavali, M. S., & Nikolova, M. P. (2019). Metal oxide nanoparticles and their applications in nanotechnology. *SN applied sciences, 1*(6), 607.

Clavero, C. (2014). Plasmon-induced hot-electron generation at nanoparticle/metal-oxide interfaces for photovoltaic and photocatalytic devices. *Nature Photonics, 8*(2), 95-103.

Dulińska-Litewka, J., Łazarczyk, A., Hałubiec, P., Szafrański, O., Karnas, K., & Karewicz, A. (2019). Superparamagnetic iron oxide nanoparticles—Current and prospective medical applications. *Materials, 12*(4), 617.

Falcaro, P., Ricco, R., Yazdi, A., Imaz, I., Furukawa, S., Maspoch, D., . . . Doonan, C. J. (2016). Application of metal and metal oxide nanoparticles@ MOFs. *Coordination Chemistry Reviews, 307*, 237-254.

Ishak, N. M., Kamarudin, S., & Timmiati, S. (2019). Green synthesis of metal and metal oxide nanoparticles via plant extracts: an overview. *Materials Research Express, 6*(11), 112004.

Khan, M., Tahir, M. N., Adil, S. F., Khan, H. U., Siddiqui, M. R. H., Al-Warthan, A. A., & Tremel, W. (2015). Graphene based metal and metal oxide nanocomposites: synthesis, properties and their applications. *Journal of Materials Chemistry A, 3*(37), 18753-18808.

Kumar, R., Lal, B., Korai, M.B., Korai, M.A., Kumar, S. (2025) IRON OXIDE THIN FILMS ON FTO to EFFICient PHotocatalytic Applications A Nano-enhanced Improved Approach (2025) *Annual Methodological Archive Research Review*, 3(10), pp. 11).

Kumar, R., Lal, B., Mandan, A.A., Korai, M.B., Kumar, S. (2025) "Fabrication and Catalytic use of Iron Oxide Thin Films on FTO with Ferrocene as source of Iron), *Physical Education, Health and Social Sciences*, 3(4), pp. 1723.

Kumar, R., Lal, B., and Mandan, AA., (2025) eco-friendly Photocatalytic Degradation of Methylene Blue Dye Using Iron Oxide Thin Film Produced by Pyrolytic Cleavage of 4-Nitrophenylferrocene, *Physical Education, Health and Social Sciences*, 3(4), pp. 2430.

Li, Y., Zhang, W., Niu, J., & Chen, Y. (2012). Mechanism of photogenerated reactive oxygen species and correlation with the antibacterial properties of engineered metal-oxide nanoparticles. *ACS nano, 6*(6), 5164-5173.

Madkour, L. H. (2019). *Nanoelectronic materials: fundamentals and applications* (Vol. 116): Springer.

Maduraiveeran, G., Sasidharan, M., & Jin, W. (2019). Earth-abundant transition metal and metal oxide nanomaterials: Synthesis and electrochemical applications. *Progress in Materials Science, 106*, 100574.

Mallakpour, S., & Madani, M. (2015). A review of current coupling agents for modification of metal oxide nanoparticles. *Progress in Organic Coatings, 86*, 194-207.

Manjunatha, S., Biradar, D., & Aladakatti, Y. R. (2016). Nanotechnology and its applications in agriculture: A review. *J farm Sci, 29*(1), 1-13.

Maynard, A. D. (2007). Nanotechnology: the next big thing, or much ado about nothing? *The Annals of occupational hygiene, 51*(1), 1-12.

Nunes, D., Pimentel, A., Gonçalves, A., Pereira, S., Branquinho, R., Barquinha, P., . . . Martins, R. (2019). Metal oxide nanostructures for sensor applications. *Semiconductor Science and Technology, 34*(4), 043001.

Sun, S.-N., Wei, C., Zhu, Z.-Z., Hou, Y.-L., Venkatraman, S. S., & Xu, Z.-C. (2014). Magnetic iron oxide nanoparticles: Synthesis and surface coating techniques for biomedical applications. *Chinese Physics B, 23*(3), 037503.

Xu, J., Liu, J. B., Liu, B. X., Li, S. N., Wei, S. H., & Huang, B. (2018). Design of n-type transparent conducting oxides: The case of transition metal doping in In2O3. *Advanced Electronic Materials, 4*(3), 1700553.

Xu, P., Zeng, G. M., Huang, D. L., Yan, M., Chen, M., Lai, C., . . . Wan, J. (2017). Fabrication of reduced glutathione functionalized iron oxide nanoparticles for magnetic removal of Pb (II) from wastewater. *Journal of the Taiwan Institute of Chemical Engineers, 71*, 165-173.

Zhang, G., Xiao, X., Li, B., Gu, P., Xue, H., & Pang, H. (2017). Transition metal oxides with one-dimensional/one-dimensional-analogue nanostructures for advanced supercapacitors. *Journal of Materials Chemistry A, 5*(18), 8155-8186.

Zhao, Y. S., Fu, H., Peng, A., Ma, Y., Liao, Q., & Yao, J. (2010). Construction and optoelectronic properties of organic one-dimensional nanostructures. *Accounts of chemical research, 43*(3), 409-418.

Statements & Declarations

Funding

The authors declare that no funds, grants, or other support were received during the preparation of this manuscript.

Competing Interests

The authors have no relevant financial or non-financial interests to disclose.

Availability of data and material: Corresponding authors will provide data on request.