

ABSTRACT

The research takes both green and chemical approach for the synthesis of metal oxide nanoparticles. Several physicochemical and spectroscopic methods were employed during the investigation.

Due to their distinct characteristics and prospective uses in numerous industries, metal oxide nanoparticles are a class of materials that have attracted a lot of attention recently. These nanomaterials are made of oxide compounds, which are formed when metal atoms join forces with oxygen atoms. Numerous different elements, including titanium, zinc, iron, aluminium, and many others, can be found in the metal component. Compared to their bulk equivalents, these materials have unique physical, chemical, and optical properties. Due to their tiny size and high surface-to-volume ratio, they perform better in a variety of applications and exhibit greater reactivity and catalytic activity.

Zinc oxide (ZnO) based nanomaterials have special qualities like strong reactivity, semiconductivity, and superior optical qualities. Transparent electronics, UV sensors, and light-emitting diodes (LEDs) might all use ZnO nanoparticles. They are also investigated for their photocatalytic qualities, which make them valuable for solar energy conversion, air pollution control and water purification.

On the other hand, iron and oxygen atoms make up iron oxide (Fe_2O_3) based nanomaterials at the nanoscale. Since they have a high capacity and function better as electrode materials, they are being extensively researched for their use in lithium-ion batteries. In the sphere of medicine, targeted cancer therapy and magnetic resonance imaging (MRI) both use Fe_2O_3 nanoparticles.

Nanomaterials based on zinc oxide and iron oxide both have special qualities and a wide range of uses in numerous industries, advancing science and technology.

Environmentally friendly processes are referred to as "green synthesis" for creating nanoparticles. It involves using organic sources as reducing agents, including plant extracts or microbes. This strategy minimises the usage of dangerous chemicals and lessens the impact on the environment. Green synthesis methods can create nanoparticles with specific features while being economical and energy-efficient. They have uses in electronics, catalysis, medicine, and environmental cleanup. In general, green synthesis encourages environmentally beneficial and long-lasting nanotechnology. This study will try to

synthesize pure, manganese doped, molybdenum co-doped and copper co-doped iron oxide nanomaterials using peel extract of *Punica granatum*.

This study also includes the chemical synthesis of iron zinc oxide nanocomposites and their different bioactivities at different calcined temperatures. Here low-temperature calcined nanocomposite outperformed in anti-oxidant activities and wheat seed germination properties whereas high-temperature calcined nanocomposite performed best against bacterial strains.

Here the synthesized nanomaterials were characterized by different techniques like UV-Vis spectroscopy, FTIR spectroscopy, dynamic light scattering method, powder X-Ray diffractometer, scanning electron microscope, energy dispersive X-Ray spectroscopy, etc. Oxidase enzyme-mimicking activities were studied by UV-Vis spectroscopy for Mn-doped Iron oxide nanoparticles and Cu-co-doped Iron oxide nanomaterials.

In this study, UV-Visible spectroscopy was used to thoroughly evaluate the interaction of Mn doped Iron oxide nanoparticles with human serum albumin (HSA). The features of interaction studies that mattered the most were the form of binding and the strength of binding.

Investigations were done on the catalytic effectiveness of several nanoparticles against methylene blue dye. By utilising pure Iron oxide nanoparticles in photo-catalytic, Fenton, and photo-Fenton similar processes, the degradation of this dye was studied. Then, utilising $\text{Mo}_x\text{Fe}_{1-x}\text{O}$ nanocomposites and copper-assisted Iron oxide nanocomposites, respectively, elaborate studies of the degradation of methylene blue via Fenton and peroxodisulfate activation were conducted. For each scenario, studies on the effects of various reaction parameters, as well as kinetic and thermodynamic analyses, were conducted.

Iron-zinc oxide nanomaterials were synthesized by co-precipitation method and calcined at different temperature. Calcination effect were studied on structure, zone of inhibition for evaluating anti-bacterial activities, IC_{50} values for antioxidant activities, seed germination and seedling growth.

The research's conclusions ought to provide fresh light on the subject of metal oxide nanoparticles. According to the results, the generated nanomaterials can be applied to biological applications as well as catalysis.

SUMMARY OF THE WORKS:

CHAPTER I

An overview of earlier works on the creation and use of nanoparticles, idea about nanomaterials, reason of the choice of zinc and iron oxide based nanomaterials, different synthetic process and potential applications are all covered in this chapter.

CHAPTER II

This chapter includes the experimental part. This chapter provides a thorough description of the synthesis of the nanomaterials included in the thesis and various studies. Among the methods employed were Powder XRD, FTIR spectroscopy, Energy Dispersive X-Ray Spectroscopy, Scanning Electron Microscope, UV-Vis spectroscopy and Dynamic Light Scattering and also different chemical and biological activity studies in applied part.

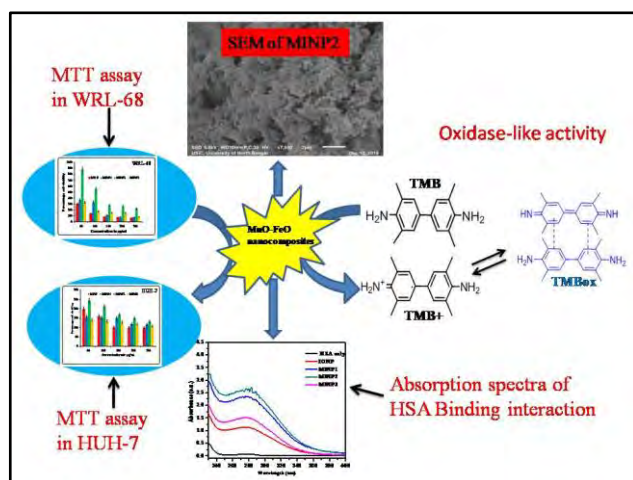
CHAPTER III

The investigation of the efficient anticancer use of magnetic iron oxide nanoparticles is currently a big topic of research that is growing daily in order to overcome the negative effects of traditional chemotherapy. In this chapter, peel extracts from *Punica granatum* were used to reduce ferrous sulphate in order to synthesise iron oxide nanoparticles, or IONPs. Flavonoids and phenolic substances with strong antioxidant content are abundant in *Punica granatum* peel extract and are effective reducing agents. For four hours, the nanopowders were calcined at 700 °C. Analytical approaches are employed to characterise the morphological assessment of cancer cells as well as their physicochemical, optical, photocatalytic, and cytotoxicity capabilities (in both normal and carcinoma cell types). The creation of IONPs with a spherical shape, trigonal (rhombohedral axis) phase structure, lower size (average size from XRD 43-47 d nm), and large surface area has been validated by XRD data, DLS, FTIR spectra, and scanning electron microscopy examination. Using the Tauc plot and UV-Vis absorption spectra, the optical band gap of 3.5 eV was determined. Under ideal conditions, IONPs demonstrate good photo-Fenton in Methylene blue (MB) dye degradation in 15 minutes with ~91% and 40 minutes with 99%. The MTT assay was also used to examine the cytotoxicity in human hepatic cell line (WRL-68) and human hepatocellular carcinoma (HUH-7) cell lines, and the results were confirmed by

morphological evaluation assay. These findings demonstrated the dose-dependent cytotoxicity of IONPs, opening up potential applications for safe usage by which is promising for the future applications.

CHAPTER IV

Here *Punica granatum* peel extract was proven to be a successful bioreducing species in heterostructure MnO-FeO nanocomposites. FTIR, UV-Vis spectroscopy, SEM, EDAX,



DLS, and X-ray diffraction (XRD) methods were used to characterise the synthesised nanomaterials. Compared to iron oxide nanoparticles (IONPs), these nanocomposites have a higher binding affinity for human serum albumin (HSA) and can function as superior nanozymes. We discover that these nanocomposites show dose-

dependent low cytotoxicity in human hepatic cell line (WRL-68) and significant toxicity to human hepatocellular carcinoma (HUH-7) cell line. The activities of IONPs are influenced by varying percentages of MnO present in their lattice.

CHAPTER V

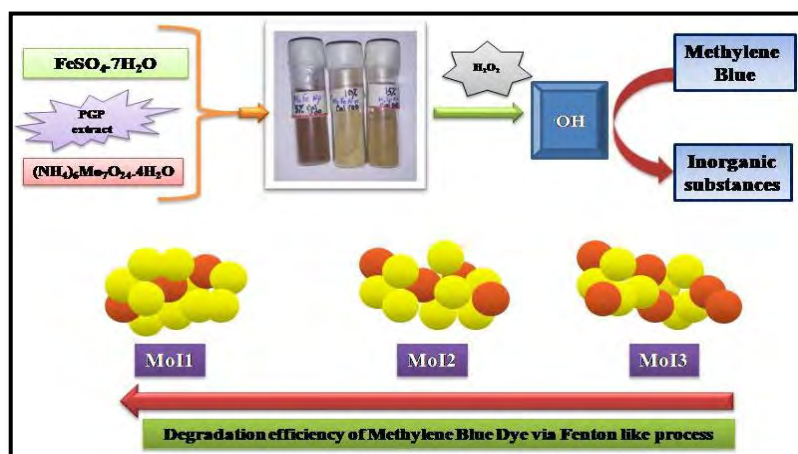
In the current work, a green technique was used to create a series of $\text{Mo}_x\text{Fe}_{1-x}\text{O}$ ($x=0.05, 0.10, 0.15$), nanocomposite catalysts. Scanning electron microscopy (SEM), UV-vis spectroscopy, X-ray diffraction (XRD), energy dispersive X-ray (EDX), Fourier transform infrared spectroscopy (FTIR), and dynamic light scattering (DLS) experiments were used to characterise the synthesised NPs. The Fenton-like activities of nanocomposites were tested by catalytic degradation of Methylene Blue (MB) dye solution, and in-depth kinetics and thermodynamics analyses were carried out. This work used *Punica granatum* peel (PGP) extract to create $\text{Mo}_x\text{Fe}_{1-x}\text{O}$ ($x=0.05, 0.10, 0.15$), nanocomposites that effectively degraded MB. This is an example of green synthesis. $\text{Mo}_x\text{Fe}_{1-x}\text{O}$ ($x=0.05, 0.10, 0.15$ nm) nanocomposites had an average crystalline size of 23–47 nm. It was discovered that the band gap values of the nanocomposites ranged from 3.31 to 3.34 eV. $\text{Mo}_x\text{Fe}_{1-x}\text{O}$ ($x=0.05,$

0.10, 0.15), an efficient catalyst, is employed in Fenton-like processes to remove MB dye.

The amount of catalyst utilised, variations in the dye's concentration, pH, H_2O_2 concentration, temperature, and other factors all had a major effect on the pace of degradation.

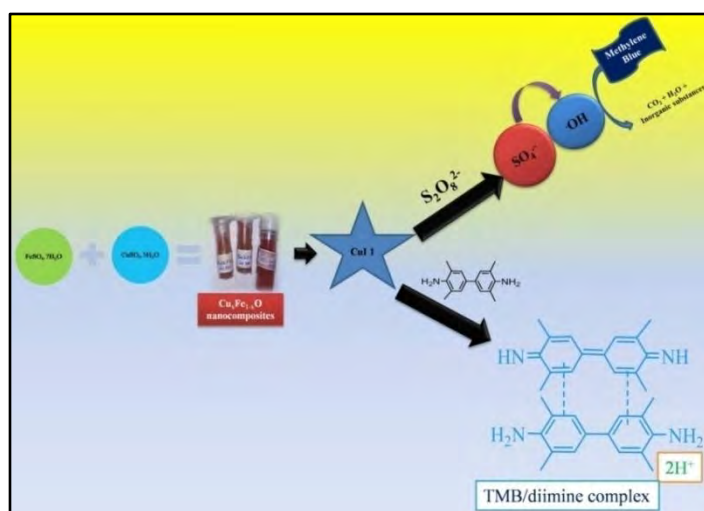
We investigated the rate of

degradation at 303.15 K temperature, with 10 mM to 30 mM of several nanocomposites and 20 mM $[H_2O_2]$. After 22 minutes of treatment, 99% of the product is destroyed under these conditions. Because of the low temperature dependency of MB degradation, the activation energy was 7.95 kJ/mol. This was shown by the scavenger quenching experiment. The breakdown of MB dye is controlled by the generation of OH radicals.



CHAPTER VI

Here, Punica Granatum peel (PGP) extract was used to create a series of nanocomposites called $Cu_xFe_{1-x}O$ (where $x = 0.05, 0.10$, and 0.15 , referred to as CuI 1, CuI 2, and CuI 3 accordingly). Several methods were used to characterise these composites. Fourier-



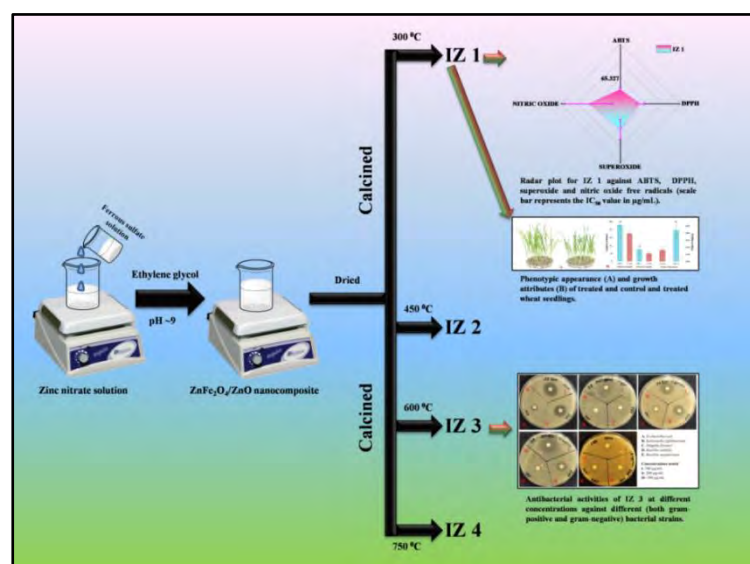
transformed infrared spectroscopy (FTIR), energy dispersive X-ray (EDX), and ultraviolet-visible spectroscopy (Uv-vis) were used to confirm the nanocomposites' formation. Using the X-ray diffraction (XRD), scanning electron microscopy (SEM), and dynamic light scattering spectroscopy (DLS) techniques, the

average sizes and shapes of the particles were examined. The nanocomposites eliminated the organic pollutant methylene blue dye (MB) by activating sodium peroxodisulfate (PDS) and imitating an enzyme. Since CuI 1 performed the best, various reaction kinetics and regulating factors were investigated for CuI 1 catalyst/nanozyme. The kinetic

parameters for the enzyme mimicking activities were $K_m = 0.04878072 \text{ mM}$ and $V_{max} = 2.221877 \text{ M min}^{-1}$. The computed rate constant for dye degradation by CuI 1 was determined to be 0.01868 min^{-1} . The activation energy, $E_a = 30.93 \text{ kJ/mol}$, which was determined from the thermodynamic analysis of the degradation process, strongly showed that the reaction rate is temperature-dependent.

CHAPTER VII

Because of their strong antibacterial and antioxidant characteristics, iron oxide and zinc oxide nanoparticles are particularly interesting. Here, we used the co-precipitation method to generate $\text{ZnFe}_2\text{O}_4/\text{ZnO}$ nanocomposites, calcined them at different temperatures (300-750 $^\circ\text{C}$), and examined a range of their properties. The average crystal size and



crystallinity both increase with rising calcined temperature, although dislocation density and microstrain decrease as temperature rises. XRD patterns and SEM images confirming the formation of the intended existing composites. EDAX spectra showed that the composites were distinctly pure. The

nanocomposite that was calcined at 600 $^\circ\text{C}$ exhibited the strongest anti-microbial activity against gram-positive bacterial strains such as *Bacillus subtilis* and *Bacillus megaterium*, as well as gram-negative pathogens including *Shigella flexneri*, *Salmonella typhimurium* and *Escherichia coli*. *Escherichia coli* showed the highest susceptibility to IZ 3 with a mean zone of inhibition of $22.333 \pm 0.577 \text{ mm}$. Overall, the synthesised nanocomposites, particularly the low-temperature product, demonstrated enhanced antioxidant activity against a variety of free radicals; these nanocomposites exhibited the greatest activity against ABTS+ (a radical with a lower IC 50 value) and the least activity against nitric oxide (a radical with a higher IC 50 value). The wheat seed germination and seedling growth were also shown to be effectively enhanced by the IZ 1, demonstrating their significance in the promotion of plant growth (increase in carbohydrate, protein, and chlorophyll content by 29.65%, 36.16%, and 26.66% over the control).

CHAPTER VIII

The concluding remarks regarding the research projects completed for this thesis are included in this chapter.

CHAPTER IX

This chapter includes the bibliography and references from all the earlier chapters.