# Synthesis of Metal Oxides Based Photocatalysts for the Removal of Organic Pollutants from Waste Water

**THESIS SUBMITTED IN PARTIAL FULFILLMENT FOR THE AWARD OF DEGREE**

DOCTOR OF PHILOSOPHY

By

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Year of Submission: 2022

# CHAPTER 6

## *Summary and Future Scope*

## 6.1 Major conclusions:

This chapter summarizes the important aspects of the thesis. As water is the most important component to sustain life on the earth surface. The fresh water may get contaminated by various types of organic pollutants such as dyes, phenolic derivatives, pharmaceuticals etc. These pollutants are highly stable in aqueous medium and are toxic to living beings. So, their complete and efficient removal from water is very crucial. Although various treatment techniques are discussed in this thesis but, heterogeneous photocatalysis has significant role toward the removal of organic pollutants from water because this process is economic, efficient and eco-friendly in nature. The major drawback of the photocatalytic process is the rapid recombination of the photogenerated electrons and holes which suppress the activity of the individual semiconductor photocatalysts, In order to overcome this drawback and improve the photocatalytic activity various efforts have been made. The modification in the surface of the semiconductor alters the direction of interfacial charge transfer and restrained the process of charge recombination.

This thesis presents the synthesis of g-$C_3N_4$/NiO, Ag/NiO and NiS/ZnO nanocomposites through hydrothermal and sol-gel routes. Different analytical and spectroscopic techniques such as XRD, FT-IR, TEM, FESEM, XPS, UV-DRS, fluorescence and B.E.T. were used to characterize the synthesized nanocomposites. The photocatalytic performances of the nanocomposites were evaluated by the degradation of MG, PNP and RhB dye on their surfaces under UV light irradiation.

The first study of the thesis is the synthesis of g-$C_3N_4$/NiO nanocomposite. The photocatalytic activity of this nanocomposite is evaluated by degradation of MG on its surface under UV light illumination. It was found that pure g-$C_3N_4$ shows very low activity

and the photocatalytic performance of pure NiO is also not very satisfactory. The reason of poor photocatalytic activity of pure g-$C_3N_4$ and NiO may be attributed due to the fast recombination of photogenerated exciton ($e^-/h^+$) and poor utilization of light while contrary to this, g-$C_3N_4$/NiO nanocomposite exhibits improved photocatalytic activity than pure g-$C_3N_4$ and NiO nanoparticles. This improvement in the photocatalytic activity of the nanocomposite (g-$C_3N_4$/NiO) is due to the formation of novel p-n heterojunction in between g-$C_3N_4$ and NiO which fascilitates the transfer of $e^-$ and $h^+$ and avoids the process of charge recombination. The larger specific surface area of the nanocomposite provides more number of reaction sites which also helps to achieve high activity, further the recyclability experiment confirms that there is no significant loss in the photocatalytic activity of the studied nanocomposite even after 3$^{rd}$ cycle; this means that it is photo-stable.

Chapter 4 reports the synthesis and photocatalytic activity of Ag/NiO nanocomposite, Ag/NiO nanocomposite was synthesized by sol-gel method and characterized by different spectroscopic techniques. RhB dye was used as a targeted pollutant to evaluate the efficiency of the synthesized catalyst under UV light. The band gaps of pure NiO and nanocomposite sample were determined from UV-DRS data, while photoluminescence (PL) spectrum was recorded to elucidate the recombination and charge transfer of photogenerated exciton ($e^-/h^+$). In this study Ag/NiO (6 wt% of $AgNO_3$) displayed the better photocatalytic activity than pure Ag and NiO nanoparticles. When the surface of the nanocomposite (Ag/NiO) was irradiated by UV light source the electrons jumped from the valence band (VB) of NiO to its conduction band (CB) which was further taken up by Ag to reduce the atmospheric oxygen in to anionic superoxide radical ($O_2^{\bullet-}$) that was the major reactive species in this case as also confirmed by the scavenger experiment. So, based on the band structure, PL studies and

scavenger experiments, it was concluded that delayed in charge recombination and plasmonic effect of silver enables the nanocomposites an efficient photocatalyst than pure nanoparticle (Ag and NiO). Moreover, creation of large number of oxygen vacancy in case of nanocomposite is also a reason of this enhanced photocatalytic activity.

The subsequent chapter 5 mainly explores the photocatalytic degradation of p-nitrophenol (PNP) and rhodamine (RhB) dye by NiS/ZnO nanocomposite photocatalysts. The nanocomposite samples were synthesized by hydrothermal method. The formation of the desired samples was validated by various analytical and spectroscopic techniques. From the XPS spectra it was confirmed that zinc and nickel is in +2 while oxygen and sulfur is in -2 oxidation states and from the B.E.T. study it was found that NiS/ZnO has larger surface area than pure ZnO nanoparticle. The PL study was carried out to understand the process of charge recombination in different samples. The activity of the photocatalysts were evaluated by applying them for the degradation of targeted pollutants (PNP and RhB) under UV light. The degradation followed pseudo $1^{st}$ order kinetics for all the samples. This chapter also presented that NiS/ZnO nanocomposites are more efficient than pure NiS and ZnO nanoparticles. The photocatalytic degradation of PNP and RhB by nanocomposite follows "S-scheme" (S-scheme is just a pathway of charge migration between two semiconductor photocatalyst in such a way that it looks like "S" letter of the English alphabet) mechanism. The reason of this improved photocatalytic activity is restrained recombination of electron and holes as well as band structures with strong redox potential of useful electron and holes.

**6.2 Future scopes of this work**

There are some possibilities pointed below on which the present work can be extended in future

1. pH dependent photocatalytic activity of the nanocomposites explored in current thesis can be performed.
2. The synthesized nanocomposites in this thesis can be applied in some more hazardous pollutants like pharmaceutical wastes.
3. Some other combinations of metals (Au, Pt) with NiO can be utilized to synthesized new nanocomposite and their photocatalytic activity would be compared with that of Ag/NiO nanocomposite.