Synthesis And Characterization of Polyaniline Zirconium Oxide (PANI/Zro₂) Composite as A Catalytic Material

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***Abstract*—A Polyaniline–Zirconium oxide (PANI/ZrO_2) composite was successfully synthesized using a chemical oxidative polymerization method followed by in situ precipitation. The composite material was characterized using X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), scanning electron microscopy (SEM), and energy dispersive X-ray spectroscopy (EDS). XRD analysis confirmed the amorphous nature of polyaniline and the monoclinic crystalline phase of ZrO_2 with an average crystallite size of 21.63 nm. SEM images revealed uniform deposition of fibrous polyaniline on the zirconium oxide surface, while EDS confirmed the presence of Zr, O, C, and N elements in appropriate stoichiometric ratios. FT-IR spectra indicated strong interactions between polyaniline and zirconium oxide through characteristic functional groups. The structural and morphological features suggest enhanced catalytic activity of the PANI/ZrO_2 composite material.**

***Index Terms*—Polyaniline, Zirconium oxide, Composite material, XRD, SEM-EDS, FT-IR**

I. INTRODUCTION

Conducting polymer–metal oxide composites have gained significant attention due to their improved catalytic, electrical, and thermal properties compared to their individual components. The combination of organic conducting polymers with inorganic metal oxides often results in synergistic effects such as enhanced surface area, improved charge transport, and better structural stability. These materials have found wide applications in catalysis, sensors, energy storage devices, and environmental remediation[1-2].

Polyaniline (PANI) is one of the most extensively studied conducting polymers because of its simple synthesis, low cost, environmental stability, and tunable electrical conductivity. Its reversible redox behavior and multiple oxidation states make it particularly suitable for catalytic and electrochemical applications. Additionally, the polymeric structure of PANI enables strong interactions with inorganic substrates, leading to improved dispersion and composite formation[3-5].

Zirconium oxide (ZrO_2) is an important metal oxide known for its high thermal stability, chemical inertness, and excellent catalytic performance. However, the tendency of ZrO_2 particles to agglomerate often limits its surface activity and overall efficiency. Incorporation of polyaniline onto ZrO_2 surfaces provides an effective approach to overcome these limitations by reducing particle agglomeration and increasing surface-active sites[6-8].

In the present study, a polyaniline–zirconium oxide (PANI/ZrO_2) composite was synthesized using a chemical oxidative polymerization method. The prepared composite was systematically characterized using XRD, FT-IR, SEM, and EDS techniques to evaluate its structural, morphological, and functional properties[9-10].

II. EXPERIMENTAL SECTION

2.1 Materials

All chemicals were purchased from Spectrochem and used without further purification. Distilled water was used throughout the synthesis.

2.2 Preparation of Polyaniline Zirconium Oxide Composite

The PANI/ZrO_2 composite was synthesized via a chemical oxidative polymerization method. A solution containing 0.35 mL of aniline in 30.8 mL of distilled water was slowly added to 0.61 mL of concentrated sulfuric acid while maintaining the temperature between 5–10 °C. The mixture was oxidized by the gradual addition of 17 mL of aqueous potassium persulfate under continuous stirring.

The synthesized polyaniline was then mixed with 7.1 g of zirconium oxychloride octahydrate ($ZrOCl_2 \cdot 8H_2O$). Ammonium hydroxide was added dropwise until precipitation occurred with constant stirring. The resulting precipitate was filtered and dried in a hot air oven at 100 °C for 6 hours to obtain the final PANI/ZrO_2 composite material.

III. CHARACTERIZATION TECHNIQUES

Melting points were determined in open capillaries and are uncorrected.

^1H and ^{13}C NMR spectra were recorded on a 300 MHz FT-NMR spectrometer using $CDCl_3$ as solvent.

FT-IR spectra were recorded using a JASCO FT-IR-4100 spectrometer with KBr pellets.

XRD patterns were obtained on a Bruker D8 Advance diffractometer using Cu-Kα radiation ($\lambda = 1.5405$ Å).

SEM and EDS analyses were performed using a JEOL JSM-6330 LA instrument operated at 20 kV.

IV. RESULTS AND DISCUSSION

4.1 X-Ray Diffraction (XRD) Analysis

The XRD pattern of polyaniline showed two broad peaks at $2\theta = 20.27°$ and $28.40°$, indicating its amorphous nature and low crystallinity. The ZrO_2 phase exhibited characteristic peaks at $2\theta = 23.86°$, $32.72°$, $46.81°$, $52.85°$, and $58.39°$, corresponding to the (110), (111), (021), (112), (300), and (212) planes of monoclinic zirconium oxide, consistent with JCPDS card no. 37-1485.

No peaks corresponding to cubic or tetragonal phases or other impurities were observed, confirming the high purity of monoclinic ZrO_2. The composite XRD pattern indicated complete deposition of polyaniline on the ZrO_2 surface, which is beneficial for catalytic performance.

The average crystallite size was calculated using the Debye–Scherrer equation and found to be 21.65 nm. The reduced crystallite size is attributed to the presence of polyaniline, which restricts particle growth and prevents agglomeration.

4.2 SEM and EDS Analysis

SEM images revealed fibrous and globular polyaniline structures uniformly distributed over the zirconium oxide surface. The images indicate strong interaction between the organic polyaniline and inorganic zirconium oxide framework. A decrease in grain size and an increase in crystallinity were observed, which are favourable for catalytic applications.

EDS analysis confirmed the presence of zirconium (13.62%), oxygen (50.37%), carbon (28.71%), and nitrogen (7.35%). The elemental composition verified that the required stoichiometric ratio was maintained in the composite material.

4.3 FT-IR Analysis

The FT-IR spectrum showed characteristic absorption bands confirming the formation of the PANI/ZrO_2 composite. The band at 3143.33 cm^{-1} corresponds to N–H and Zr–OH stretching vibrations, while the peak at 1632.3 cm^{-1} is attributed to Zr–OH bending vibrations. Peaks at 1404.23 cm^{-1} and 1230 cm^{-1} correspond to C=C stretching of the benzenoid ring and C–N stretching vibrations, respectively. The band at 1131.4 cm^{-1} indicates quinoid ring vibrations (N=Q=N), and the peak at 832.23 cm^{-1} is assigned to aromatic C–H bending of a 1,4-disubstituted benzene ring. These results confirm strong interactions between polyaniline and zirconium oxide.

V. CONCLUSION

The PANI/ZrO_2 composite was successfully synthesized using a chemical oxidative method and characterized by XRD, SEM-EDS, and FT-IR techniques. The composite exhibited a monoclinic ZrO_2 phase with reduced crystallite size and uniform polyaniline coating. Structural, morphological, and spectroscopic analyses confirmed strong interactions between polyaniline and zirconium oxide, leading to improved surface properties. These features indicate that the synthesized PANI/ZrO_2 composite is a promising catalytic material.

REFERENCES

[1] MacDiarmid, A. G. (2001). Synthetic metals: A novel role for organic polymers. *Synthetic Metals*, 125(1), 11–22.

[2] Stejskal, J., & Gilbert, R. G. (2002). Polyaniline. Preparation of a conducting polymer. *Pure and Applied Chemistry*, 74(5), 857–867.

[3] Bhadra, S., Khastgir, D., Singha, N. K., & Lee, J. H. (2009). Progress in preparation, processing and applications of polyaniline. *Progress in Polymer Science*, 34(8), 783–810.

[4] Rahman, M. M., & Kim, J. J. (2011). Synthesis, characterization and electrochemical properties of polyaniline–metal oxide nanocomposites. *Electrochimica Acta*, 56(4), 1436–1442.

[5] Livage, J., Henry, M., & Sanchez, C. (1988). Sol–gel chemistry of transition metal oxides. *Progress in Solid State Chemistry*, 18(4), 259–341.

[6] Gupta, V. K., Agarwal, S., & Saleh, T. A. (2011). Chromium removal by combining the magnetic properties of iron oxide with adsorption properties of carbon nanotubes. *Water Research*, 45(6), 2207–2212.

[7] Kooti, M., & Afshari, M. (2012). Preparation and antibacterial activity of NiO nanoparticles. *Journal of Nanoscience and Nanotechnology*, 12(2), 1501–1508.

[8] Cullity, B. D., & Stock, S. R. (2001). *Elements of X-ray Diffraction* (3rd ed.). Prentice Hall, New Jersey.

[9] Stuart, B. (2004). *Infrared Spectroscopy: Fundamentals and Applications*. John Wiley & Sons, Chichester.

[10] Goldstein, J. I., Newbury, D. E., Joy, D. C., Lyman, C. E., Echlin, P., Lifshin, E., Sawyer, L., & Michael, J. R. (2003). *Scanning Electron Microscopy and X-ray Microanalysis* (3rd ed.). Springer, New York.