

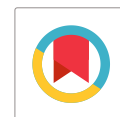


Synthesis, Electrochemical Characterization and Determination of Photocatalytic Activity of Zn-doped Iron Oxide / Graphene Oxide / Chitosan Nanocomposite

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ABSTRACT

A nanocomposite of Zn-doped Iron oxide/Graphene Oxide/Chitosan was prepared by simple solution mixing and evaporation using ultrasonication. Formation of composite in nano-dimensions was proved by XRD, TEM and AFM studies. Electrochemical characterization of the nanocomposite has shown that the composite had good electrochemical stability. Nyquist plot has shown a well-defined semicircle indicating its good capacitance behavior. Impedance studies through Bode phase angle plot have shown nearly 90° phase angle, indicating the nanocomposite as a very good candidate for supercapacitor applications. The synthesized nanocomposite has shown photocatalytic activity in sunlight against methylene blue dye with a highly efficient removal of 97.2% in 60 minutes.

Keywords: AFM; Chitosan; Graphene oxide; Nanocomposite; Photocatalytic activity; TEM.

1. INTRODUCTION

Graphene oxide (GO) and functionalized graphene oxide have attracted the attention of scientific community as they have a variety of novel applications due to their excellent chemical and physical properties, including low density, exceptional mechanical properties, large surface area, inherent impermeability, mechanical strength and excellent electrical conductivity. GO can be dispersed throughout a selected polymer matrix to make GO-based composite with excellent mechanical and thermal properties. To harness the benefits of photocatalytic activity of zinc, magnetic property of iron oxide (Fe_3O_4), biodegradability of natural polymer chitosan (CHI) and electrical conductivity of GO, a novel nanocomposite has been synthesized by simple solution mixing and evaporation method. Special emphasis was made to evaluate the electrochemical nature of the synthesized nanocomposite and its efficiency to degrade Methylene blue dye.

2. EXPERIMENTAL METHODS

Each of the components was synthesized separately (GO from graphite flakes, CHI - commercial sample, Zn-doped Iron oxide using ZnCl_2 , FeCl_3 and FeSO_4 with PEG as solvent) and then mixed in solution phase at room temperature. The solutions were degassed for 30 min. in a desiccator. The resultant Zn-

doped Fe_3O_4 -GO-CHI nanocomposite was separated by ultra-centrifuge and dried at 70 °C for 8 hours to remove the solvents. The dried samples were soaked in 2 wt.% aqueous sodium hydroxide for 1 hour to remove the acid, washed with water to neutrality and then dried at 70 °C for 6 hours. The homogenized and dried samples thus obtained, were used for characterization. Photocatalytic degradation property of the synthesized nanocomposites was also investigated using methylene blue (MB) dye as probe molecule, on exposure to sunlight.

3. RESULTS AND DISCUSSION

3.1 XRD and Microscopic studies

X-ray diffractogram has shown a shift in the GO-CHI peak from $2\theta = 20^\circ$ to $2\theta = 19.5^\circ$ and also a significant enhancement in the broadness of the peak, indicating a strong interaction between the components of the nanocomposite as well as the formation of the composites in nano-domain (Ding *et al.* 2015). The particle size calculated was found to be 23.96 nm. TEM image of nanocomposite has revealed the presence of exfoliated GO in the matrix of chitosan with the Zn-doped iron oxide incorporated into it with the particle size of 20 nm. AFM studies have shown the presence of

a larger specific surface area and more active sites (Christelle Pau Ping Wong *et al.* 2015).

3.2 Electrochemical studies

Cyclic Voltammetry (CV) was carried out using a three-electrode system by coating the nanocomposite onto glassy carbon electrode surface and scanning in the range from 50 to 500 mV/s (Fig. 1); it has shown that with increasing scan rate the peak current also increased, indicating good adherence and electrochemical stability of the composite. Interestingly, the CV taken at a higher scan rate of 500 mV/s exhibited a current of 2.724×10^{-5} A and well-defined redox peaks, showing the fast electro activity of the composite material. EIS studies carried out through Nyquist plot have shown the formation of well-defined semicircle (Sezai Sarac *et al.* 2008).

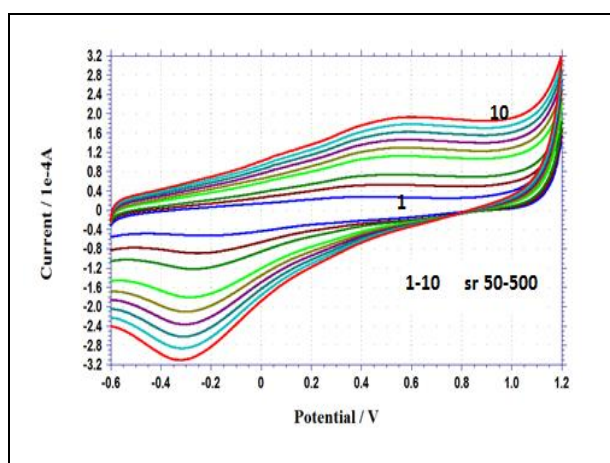


Fig. 1: CV of the nanocomposite with varying scan rate

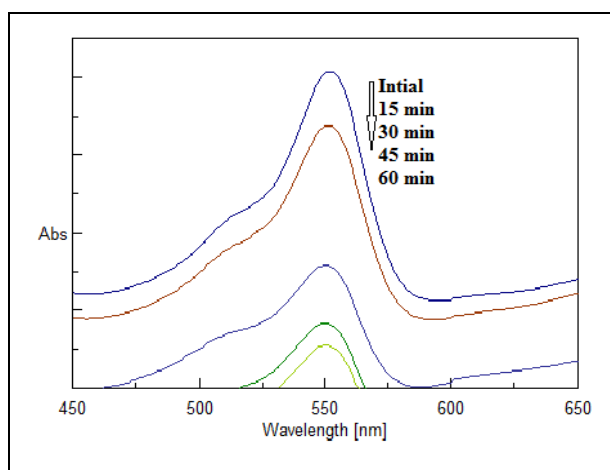


Fig. 2: Degradation of Methylene Blue dye with time

3.3 Photocatalytic activity

Photocatalytic degradation property of the synthesized nanocomposites was also investigated using

methylene blue dye as probe molecule on exposure to sunlight. It has shown 93.2% degradation efficiency after exposure for about 60 minutes (Fig. 2).

4. CONCLUSION

Novel zinc-doped Iron oxide/GO/CHI nanocomposite was synthesized by simple solution mixing-evaporation method from cheap and commercially available precursors. The XRD, TEM and AFM revealed the formation of nano sized composites with 20 nm dimensions. Electrochemical characterization of the nanocomposite showed good electrochemical stability as well as high capacitance behavior with the Bode phase angle of 87° . The nanocomposite exhibited enhanced photocatalytic activity in a short interval of time and hence can be efficiently used as photocatalyst in the process of removal of organic dyes for environmental cleaning and water purification.

FUNDING

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

CONFLICTS OF INTEREST

The authors declare that there is no conflict of interest.

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