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One Pot Synthesis, Characterization of Zn-GO Nanocomposite for the Electrochemical Decoloration of a Textile Dye

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ABSTRACT

A facile one pot efficient synthetic approach has been adopted to prepare Zn-GO nanocomposite material from the synthesized GO. The X-ray diffraction (XRD) analysis revealed the formation of Zn/GO structure from the zinc chloride precursor during the synthesis. The surface characteristics and elemental composition of the nanocomposites have been studied by means of scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS). The characterization of the Zn/GO composites suggested that the metal is uniformly dispersed in the graphene matrix and gives evidence indicates that the flake-like nanosheets are GO, which are formed and interface and we can see good compatibility between the graphite oxide and the zinc matrix. Electrochemical decoloration of an aqueous solution of a textile dye Acid Red 10B was investigated under different experimental conditions by using carbon electrodes. Our findings revealed that the electrodes evidenced great dye decoloration abilities to treat solutions containing this dye, in the presence of the synthesized nanocomposite. The influence of the synthesized nanocomposite was remarkable in the electrochemical dye decoloration process and ~100% color removal was found by the addition of 0.004g nano composite per 250 mL of the dye solution.

KEYWORDS: *carbon electrodes, electrochemical decoloration, nanocomposite, textile dye*

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I. INTRODUCTION

Nanocomposite (NC) materials have gained greater attention and interest of scientists in recent years because of their improved properties than the single metal nanoparticles. Current and growing interest in nanostructures results from their numerous potential applications such as in materials development, biomedical sciences, electronics, optics, magnetism, energy storage, and electrochemistry [1-6]. Nano-sized materials display properties that differ from their respective bulk counterparts [7].

Graphene is a one-atom thickness material with sp^2-hybridized carbon atoms packed into a hexagon structure. It has received a special attention due to its unique properties such as chemical, and thermal stability, high surface area, electron conductivity [8, 9]. The synthesis of graphene is mainly based on Hummers and Offeman method [10], which produces graphene oxide (GO). Graphene-based materials have textural and structural properties that enable the use in energy storage, photoreduction, supercapacitors, solar cells, drug delivery, adsorption [11–13].

Zn based nanomaterials has attracted significant attention for various optoelectronic and catalytic applications due to its non-toxicity, low-cost, excellent chemical stability, wide range of radiation absorption, good electrical properties, and high electrochemical coupling coefficient. [14]. The Zn based NPs are emerged as one of the highly promising metal NPs used in many applications, including gas sensor, protective coatings, electro photography, solar cells [15], healthcare, optics, ultraviolet, and blue light emitting diodes, and in the evolution of membrane area, due to their excellent properties of antimicrobial, anti-corrosive, thermal, and mechanical stability, high catalytic activity, strong adsorption capacity, low toxicity, and environmental friendly feature [16,17].

Large amounts of different dyes are being used in the textile industries for dyeing purpose. Since dyes are carcinogenic and toxic, they imbalance the chemical and biological nature when they are discharged into the water bodies [18]. A large amount of effluents

discharged from textile industries into water bodies, hence it is under considerable pressure to minimise the water consumption and to reduce the treatment cost. Conventionally textile wastewater is being treated through biological, physical and chemical methods [19]. Further, the main drawbacks of chemical coagulation are the addition of other chemicals. In this context, electrochemical technique is considered to be a powerful tool for the pollution control and in the treatment of textile wastewater. Electrochemical methods have been successfully tested [20] and it has certain significant advantages such as simple equipment, easy operation, lower operating temperature, etc. The process requires significantly less area and equipment than the conventional biological treatment processes [21, 22].

In the present study, the one pot synthesis of high-quality zinc - graphene oxide (Zinc-GO) nanocomposite was carried out using the simple and efficient method. The prepared nanocomposite material were characterized and reported in this study for better understanding the properties of nanohybrid material. The present work focuses on the electrochemical decolorization/degradation treatment of the textile dye, using carbon electrodes, with the addition of the prepared nanocomposite to the dye solution. The efficiencies of the electrochemical process is evaluated and various parameters are optimized for the electrochemical decolorization/degradation of the textile dye.

II. Materials and methods:

A. Chemicals and Reagents:

Natural graphite flakes, $ZnCl_2$, NaCl, NaOH, HCl, H_2O_2 (30%), $KMnO_4$ and tri sodium citrate are obtained for SD Fine chemicals, India. All the chemicals were of analytical reagent grades and used as received, without further purifications. The aqueous solutions were prepared in double distilled water. The textile dye, Acid Red 10B was obtained from a textile industry, Molakalmuru, Karnataka. The carbon electrodes of > 90% purity were obtained from commercial dry cell, tested for purity and proper surface treatment were given before electrochemical experiments.

B. Preparation of the Graphite oxide and metal – GO nanocomposites:

The GO was synthesized from natural graphite flakes, using the Hummers' method [10]. 0.1 g of obtained GO was dispersed in 100 mL of H_2O, to this 0.1g $ZnCl_2$ was added and ultrasonicated for 30 minutes. This mixture was stirred for 15 min to produce a uniform dispersion to have a metal leading oxide. The solution pH was adjusted to 10.0 using 0.1M Sodium hydroxide solution and stirred continuously for four hours. Then 50 ml of 0.1 M trisodium citrate was added and stirred continuously for two hours at 75°C. The mixtures were heated at 100 °C for 4 hours to obtain Zn/GO composite materials. The resulting material was then filtered and washed several times with double distilled water. It is then dried in oven at 80^0C. It was then calcined at 400°C for three hours.

C. Instrumentation:

Scanning Electron Microscopic studies:

The surface morphologies of the composite samples were examined by scanning electron microscopy, using a ZEISS Supra 40 scanning electron microscope (SEM).

EDAX analysis:

The weight percentage of elements in the metal nano-composite coatings were verified by using energy dispersive X-ray analysis using FEI ESEM Quanta (EDAX) machine.

UV-Visible Spectroscopic studies:

Ultraviolet-Visible (UV-Vis) spectra of synthesized graphene oxide samples were collected on a UV-Vis spectrophotometer (Systronics 119).

FTIR Spectroscopic studies:

Characterization techniques Fourier transform infrared (FTIR) spectra of the synthesized graphene oxide samples were recorded on a Shimadzu spectrophotometer using KBr as the mulling agent.

XRD studies:

XRD analyses of the powdered samples were performed using an X-ray power diffractometer with Cu anode (PAN Analytical Co., X'pert PRO, Almelo, The Netherlands), running at 40 kV and 30 mA, scanning from 4 to 80° at 3/min, for data including particle size or crystallinity measurement.

Electrochemical degradation of textile dyes:

Graphite carbon electrodes of 4.5 cm length and 0.8 cm diameter were used as anode and cathode for electrochemical degradation studies [23]. The Acid Red 10B dye solution having the concentration range from 10 to 50ppm was used for the experiment. The supporting electrolytes such as NaCl were added to the electrolytic solution, which increases the conductivity of the solution and reduces the electrolysis time. The solution was kept under agitation using magnetic stirrer.

III. Results and discussions:-

A. Characterization of GO and Zn-GO nano composite:

The formation of stable graphene dispersions enables the reaction process to be monitored using UV-Vis spectroscopy. The absorption peak of the GO

dispersion at 227 nm gradually confirmed the oxidation of graphite and formation of GO.

The presence of the different oxygen containing functional groups was confirmed by the FT-IR spectrum of graphite oxide. The most prominent features in the spectrum are the adsorption bands corresponding to 3430 - OH stretching vibration, 1733 - C=O Carbonyl stretching, 1621 - C=C Phenol ring stretching, 1422 - OH phenol deformation vibration, 1226 - C-OH Phenol stretching, 1040 - C-O stretching, 2465, 2329 - CH_2 symmetric and asymmetric vibrations.

The structural confirmation studies were carried out using X-ray diffraction (XRD) analysis. Figure 1 shows the XRD patterns of ZnO–GO composites. The 2 θ peaks were observed at 26.2, 33.3 and 46.4. The predominant peak orientation of the (101) lattice plane was observed for ZnO–GO composites, and the observed peaks were indexed with a standard hexagonal structure (JCPDS-36-1451). In addition, other diffraction lines related to the (100), (002), (102) and (112) planes of the lattice orientation of ZnO were observed for the ZnO–GO samples. Furthermore, we estimated the crystallite size of the nanocomposites using Debye-Scherer's formula to help deduce their microstructural characteristics [24, 25].

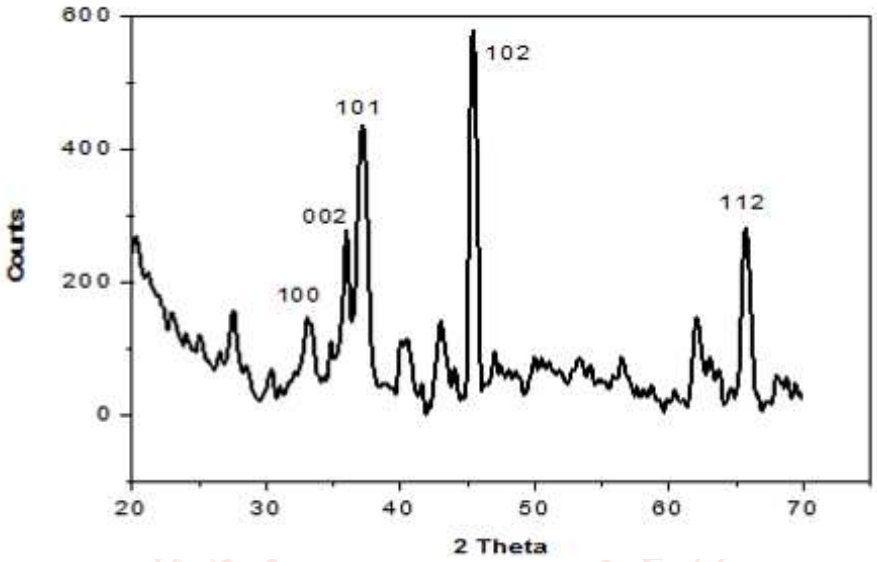

Figure 1 XRD patterns of Zn/GO composites

Figure 2 shows the SEM images, of Zn-GO composite. From the SEM images, GO and Zn enhanced the agglomeration process to form strongly bonded hybrid composites. The scanning electron microscope image (SEM) clearly shows the porous nature of the graphene matrix, forming coral and leave like structures. However, much number of zinc particles cannot be identified in the images.

The addition of graphite oxide leads to the surface is associated with crystal growth and occupies the grain boundaries. In case of the composite obtained is as seen in the figure, the SEM image clearly indicates the presence of GO flakes. Hence SEM study suggests proper bonding between matrix and reinforcement along their interface.

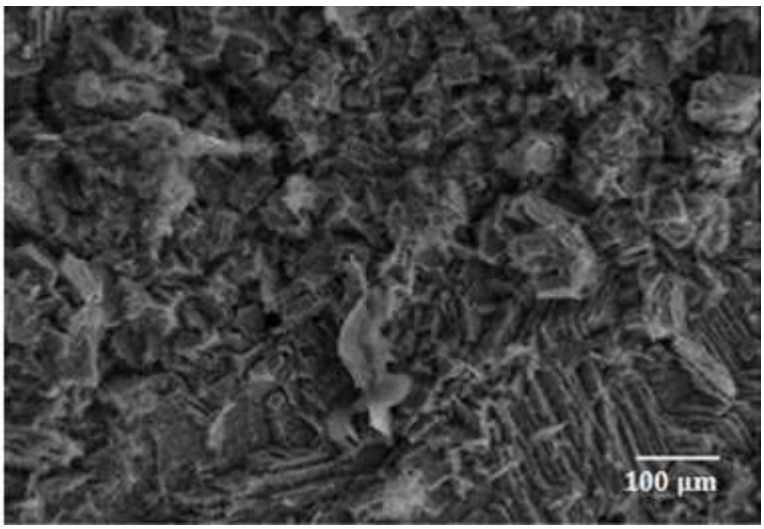

Figure 2 Scanning electron microscopy (SEM) image Zn– GO hybrid structures.

Despite the apparent absence of zinc in the SEM images, the energy dispersive X-ray spectroscopy clearly shows that the deposited films contain zinc (Figure 2). Mapping using ZnKa1 radiation does not show areas of increased zinc concentration. This suggests that the metal is uniformly dispersed in the graphene matrix beyond the degree of recognition under the resolution achievable at the chosen conditions.

In Figure 3, the EDS spectra of Zn-GO composite, with-45 % of Zinc sample are shown. Distinct Zinc and carbon and oxygen peaks can be seen from the EDS spectra. As seen in the insets of figure 3, the EDS spectrum also gives evidence indicates that the flake-like nanosheets are GO are formed and interface and we can see good compatibility between the graphite oxide and the zinc matrix on the stainless steel substrate. This is because of the presence of nearly 20% C and 35% O, and 45% Zn by atomic percentage as shown in the Figure 3 and indicated that the whole material is with the Zn-GO composite [26].

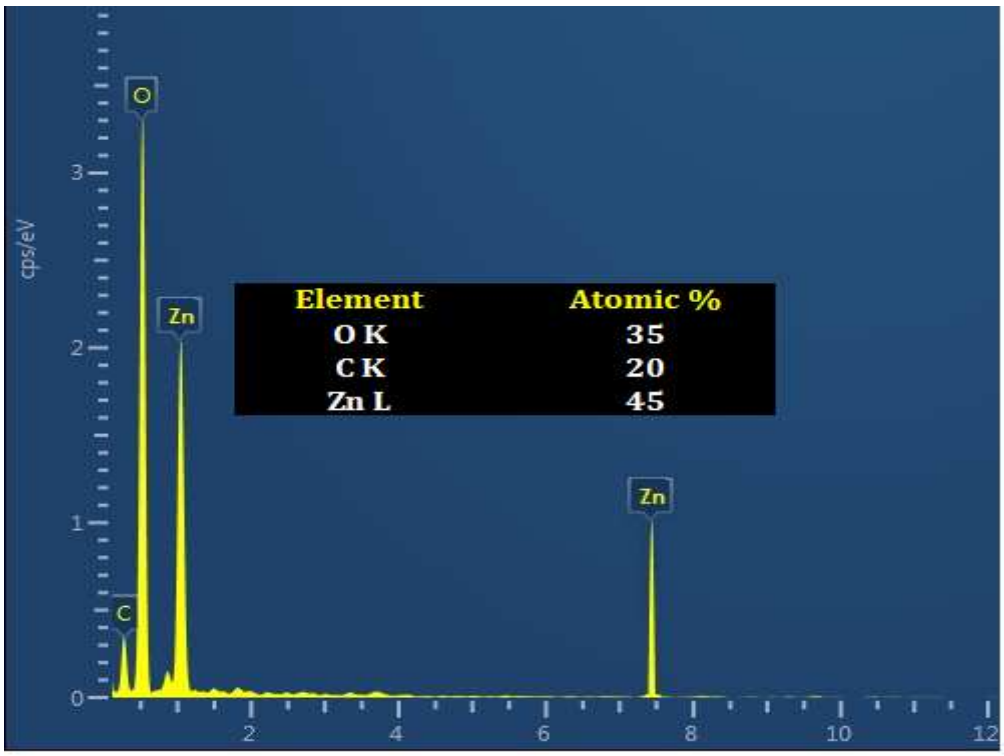

Figure 3 Energy dispersive X-ray spectroscopy of zinc –GO composite

B. Application of Zn-GO nano composite for the electrochemical degradation of Acid Red 10B: Influence of electrolytic conditions on dyes degradation

Effect of initial pH: Solution *p*H is one of the important factors that affect the performance of electrochemical process. Hence experiments were conducted to study the effect of *pH* on the degradation efficiency of textile effluent. A significant difference in the extent of decolourisation was noted when concentration of NaCl was at 0.5 g /250mL. The initial *p*H of the solution (3-11) was adjusted using 1N H_2SO_4 or NaOH. The electrolysis was carried out at the current density of 0.04 Acm^{-2} for 10 mins with a textile effluent and at room temperature (308K). After electrolysis the results indicate that final *pH* was slight varied from acidic condition and decreased from basic conditions. The decolouration efficiency of dye effluent was found percentage of 99.7 % in acidic *pH*-8 It indicated that the degradation of dye effluent in acidic solution is higher than that of in the basic media [27]. Therefore the optimum *pH* 8 was maintained in subsequent experiments.

Effect of current density: Current density is a very important variable in electrochemical engineering. As shown in the Figure 4. the colour removal efficiency was increased by increasing the applied current density (0.01to 0.06 A/cm^{-2}) the results may attributed to the increased oxidant such as: chlorine/hypochlorite, hydroxyl radicals at higher current densities. The hydroxyl radical adsorbed on the active sites at the anodic surface of the graphite carbon increases with current density, which in turn would enhance the rate of electro-oxidation of dye molecule present in the effluent. Further increase in the current density did not have much effect on decolourisation, which can be attributed to the fact that increasing current density increases over potential required for the generation of oxidants [28]. At the same time, more energy will be consumed at higher current density applied. Therefore, the optimal current density for the successive electrochemical degradation/decoloration of dye effluent was $0.04A/cm^2$.

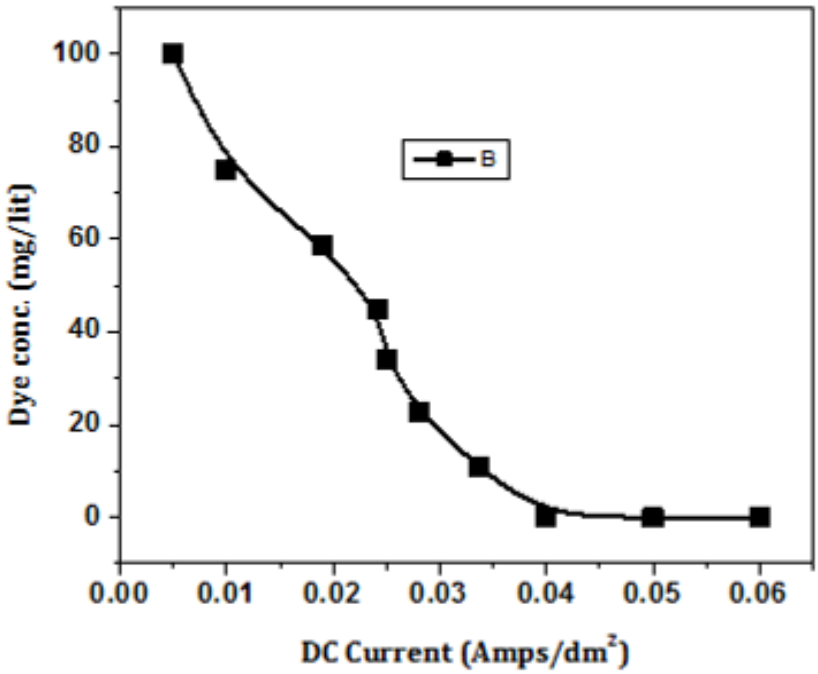

Figure 4 Effect of current density on the decoloration of Acid Red 10B

Effect of supporting electrolytes

When NaCl is added, the decolourisation efficiency increased with a subsequent decrease in the applied voltage. From this observation it concluded that the introduction of NaCl increases efficiency of decoloration and obtained maximum efficiency at 0.5g/250mL.

Electric energy consumption:

The electrical energy consumption during electrochemical degradation process is an important operating parameter. The electric energy consumption (E), required to decompose the dye solution of 50 ppm (w/v) concentration at various current densities, was calculated in terms of $kWhm^{-3}$ using the relation:

E=VItEVs10-3

where V is the applied voltage (V), I is the applied current (A), t_E is the electrolysis time (h) and V_s is the volume of dye solution (m^3). As per the results the minimum electrical energy consumption was 1.6 $kWhm^{-3}$ at 10 Am^{-2} current density. At higher current densities, the energy consumption was found to be little increased, which may be attributed to the increased hydrogen and oxygen evolution reaction (Table 1).

Table 1 The electric energy consumed during the degradation of 50 ppm (w/v) Acid Red 10B dye solution (pH 8) at various current densities

Current (A)	Current density (Am^{-2})	Electrolysis time (min)	Energy Consumption ($kWhm^{-3}$)
0.01	10	20	1.6
0.02	20	15	2.4
0.04	40	10	3.2
0.06	60	5	2.6

Effect of Zn-GO nanaocomposite:

Figure 5. shows the effect of Zn-GO nano particle concentration on the decoloration of Acid Red 10B. In these conditions, the removal of ions in dye from wastewater is mainly due to the combination of the electro-adsorption of particle electrodes and anodes and the enrichment of ions in the solution [29]. The concentration of Zn-GO nanoparticle is increased from 0 to 0.004g/250mL and maximum dye decoloration efficiency at 0.004g/250mL.

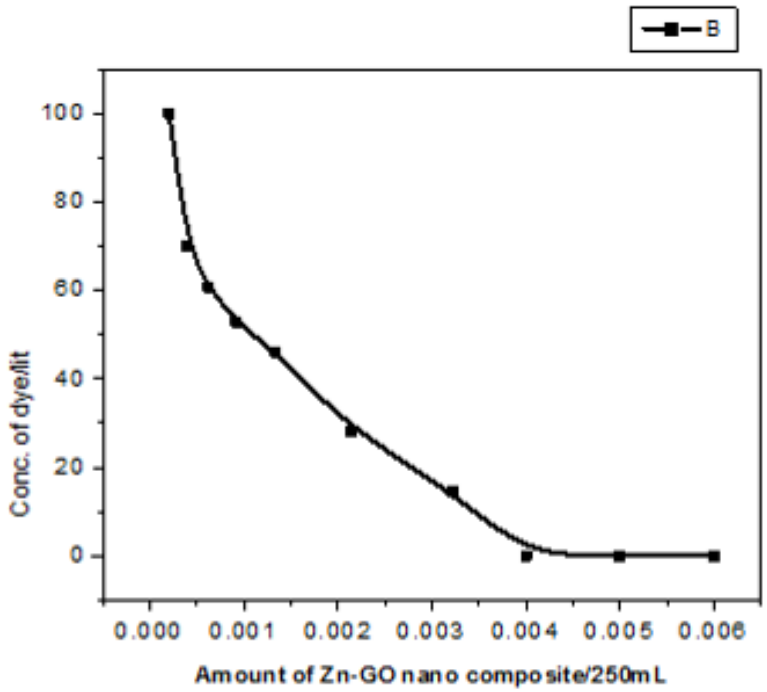

Figure 5 Effect of current density on the decoloration of Acid Red 10B

IV. CONCLUSION:

We successfully synthesized the metal Zn-GO nanocomposite by a facile one-pot preparation approach. The characterization of the Zn/GO composites suggested that the metal is uniformly dispersed in the graphene matrix and gives evidence indicates that the flake-like nanosheets are GO, which are formed and interface and we can see good compatibility between the graphite oxide and the zinc matrix. Electrochemical decoloration of an aqueous solution of Acid Red 10B dye was investigated under different experimental conditions by using carbon electrodes. Our findings revealed that the electrodes evidenced great dye decoloration abilities to treat solutions containing this dye, in the presence of the synthesized nanocomposite. The influence of the synthesized nanocomposite was remarkable in the electrochemical dye decoloration process and almost 100% color removal was found by the addition of 0.004g nano composite per 250 mL of the dye solution at a pH =8, under a current density of 0.04 A cm^{-2} on electrode and NaCl supporting electrolyte at 0.5 g/250mL . Our results showed the applicability of electrochemical technology with the application of the nanocomposite proposes, an alternative for dye removal from textile wastewater, eliminating their strong color by reducing their eco-toxicological consequences for the aquatic environment.

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