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Synthesis and Characterization of Strontium Doped Graphene Oxide Nanocomposite for the Electrochemical Decoloration of a Textile Dye from Aqueous Medium

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Abstract: *Strontium-GO composites were synthesized by a facile one-pot approach. Microstructures of the constituent elements of the prepared composite were studied by scanning electron microscopy and X-ray diffraction (XRD) and energy dispersive spectroscopy (EDS)***.** *The characterization of the Sr/GO composites suggested that the disorderly, folded sheet-like accumulation in the graphene matrix. The stoichiometry of elements was estimated and presented in the inset from the area intensity of the peaks in the EDX spectrum. Electrochemical decoloration of an aqueous solution of a textile dye Violet 5BN 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% col*or 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

I. INTRODUCTION

Graphene oxide (GO) has been considered to be a promising adsorbent for the decontamination of many toxic substances, due to its high surface area, two-dimensional structure, chemical stability, environmental friendly, biocompatibility and having massive hydroxyl, carboxyl and epoxide polar functional groups [1-4]. It is found that removal efficiency of GO is decreased due to the GO-sheets aggregation and inter-layer and/or intra-layer self H-bonding. Hence the researchers are interested in development of novel GO-doped nanocomposites [5].

Strontium has physical and chemical properties similar to that of calcium. It occurs naturally and is the 15th most abundant element in the Earth's crust. Since the elements are chemically very similar to Calcium, stable strontium isotopes do not pose a significant health threat. It has good corrosion resistance property than that of stainless steel and titanium. Strontium produce strontium oxide when exposed to air which replicates the properties of strontium. Strontium oxide or Strontia is basic in nature and is in the form of white powder. It has cubic crystalline structure with a high melting point of 2,531°C. Strontia is soluble in potassium hydroxide, slightly soluble in alcohol but insoluble in acetone and ether [6].

Strontium based nanoparticles as an interesting nanoparticles have been exploited extensively for water decontamination due to their thermal and chemical stability, high surface area, photocatalytic activity and large carrier effective mass [7-9]. It has been reported that graphene oxide doped with $SrTiO_3$ nanoparticles provided an environmental friendly, low cost nanocomposite with a good thermal stability [10]. A survey of literatures showed that Sr based nanocomposite has been rarely considered as adsorption material.

Azo dyes are the most commonly used dye categories and account for more than 60% of total dyestuffs and their relative importance may even increase in the future. 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 [11]. It is estimated that nearly 10-15% of azo dyes are unutilized and discharged to the environment via wastewaters leading to serious pollution, 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,

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physical and chemical methods [12]. Further, the main drawbacks of chemical coagulation are the addition of other chemicals.

Azo dye elimination from textile discharge is usually linked first with decolorization followed by mineralization of colorless compounds. Nowadays, high efficiency, environmental-friendly, and cost-effective technologies for azo dye removal are urgently desired due to increasingly stringent environmental law requirements. Against this background, versatile methods have been proposed including physical, chemical, advanced oxidation, and electrochemical processes. However, disadvantages such as high operating cost, incomplete azo dye degradation, possible secondary pollution, and high energy consumption, impede the practical applications of these methods in full-scale wastewater treatment methods. Electrochemical methods have been successfully tested [13] 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 [14, 15].

In the present study, the one pot synthesis of high-quality Sr-Graphite oxide 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 Sr-GO nanocomposite could be successfully used electrochemical decoloration of toxic textile dye.

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, Violet 5BN. The existence of nanoparticles (MNPs) in the nanocomposite provide fast electrochemical decoloration of toxic textile dye on the carbon electrodes from aqueous media.

II. MATERIALS AND METHODS

2.1 Chemicals and Reagents

Natural graphite flakes, $Sr(NO_3)_2$, NaCl, NaOH, Na_2CO_3, HCl, H_2O_2 (30%), $KMnO_4$, CTAB, ethanol and tri sodium citrateare 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, Violet 5BN 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.

2.2 Preparation of the Graphite oxide and Sr –GO nanocomposites

The GO was synthesized from natural graphite flakes, using the Hummers' method [16]. The desired amounts of strontium nitrate ($Sr(NO_3)_2$) solution was added dropwise to the aqueous solution of 10 % excess of sodium hydroxide (NaOH) and sodium carbonate (Na_2CO_3) using vigorous stirring to obtain the precipitate, at a pH 12-13 and a temperature 40°C, respectively. Then, the precipitate was obtained by filtrating and washed with distilled water and ethanol. Then the precipitate was dried at 60 °C in vacuum for 24 h to obtain the precursor. The precursor Sr nanoparticles were calcined at 500 °C for 2h. Cetyltrimethyl ammonium bromide (CTAB) was added into the prepared GO solution with constant stirring to get a homogeneous dispersion. Then the prepared precursor Strontium nanoparticles were added into the above dispersion with vigorous stirring for 10 h. Then 50 ml of 0.1 M trisodium citrate was added into this suspension and stirred continuously for two hours at 80ºC. The suspension was centrifugally washed using distilled water and ethanol. The resulting product was dried at 60 °C in vacuum for 12 h to obtain GO/Srnanocomposites.

2.3 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.

D. 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 [17]. The Violet 5BN 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

3.1 Characterization of Sr-GO Composite

It was observed that the broadening and weakening of the (002) diffraction peak of graphene was obvious at a 2θ value of 22°, indicating the reduction process cannot restore graphene to the highly ordered crystal structure of graphite. The Sr/GO composites exhibited the very similar diffraction peaks, as shown in Fig.1. The diffraction peak of graphene was easy to be suppressed by Sr/GO due to the loading content of graphene in the composites. [18] labeled diffraction peaks in the range of 5–70° can be indexed to the hexagonal Sr/GO crystallites. [19, 20] The sharp diffraction peaks and high intensity indicated the good crystallinity of as-obtained composites.

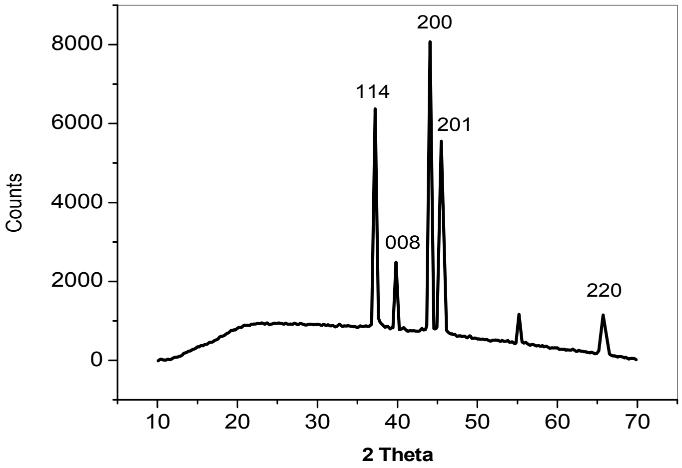


Figure 1: XRD patterns of Sr/GO composites

The series sharp diffraction peaks of Sr/GO at 2θ = 37.0°, 40.0°, 43.3° and 45.0° are assigned to the (114), (008), (200), and (201) crystal planes, respectively [21, 22]. Based on previous reports [23], GO is effectively reduced to under the effect of reducing agent and the lamellar dispersion GO/Sr composites. The characteristic diffraction peaks of R-GO/Srnanocomposites were observed, which means the presence of Sr/GO nanocomposites.

Fig.2 depicts the morphology of Sr/GO nanostructure, which displayed a specific flake-like structure. Fig.2 reveals the surface morphology of GO, clearly illustrating the disorderly, folded sheet-like accumulation. As evident from the micrographs, Sr nanoparticles have attained approximately uniform particle size. Figure exhibits Sr/ GO nanocomposite, which indicate the transformation in the surface morphology of GO upon addition of GO. As is obvious from the Fig., the folded and stacked GO sheets become segregated after being reacting with Sr[24].

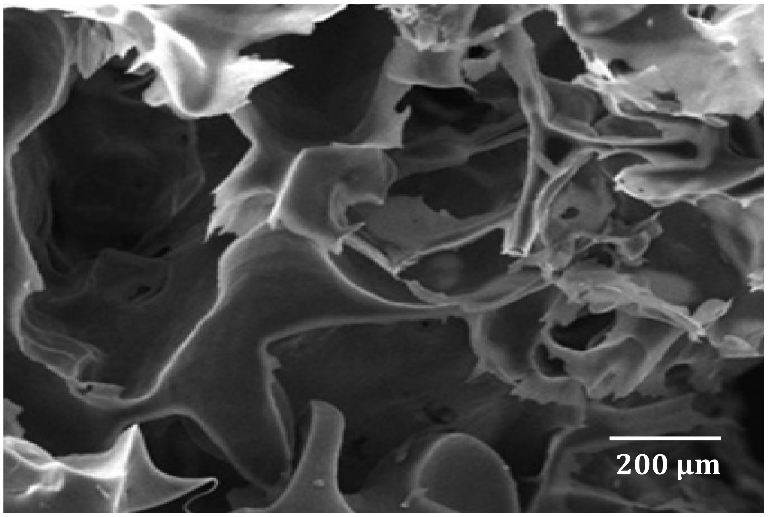
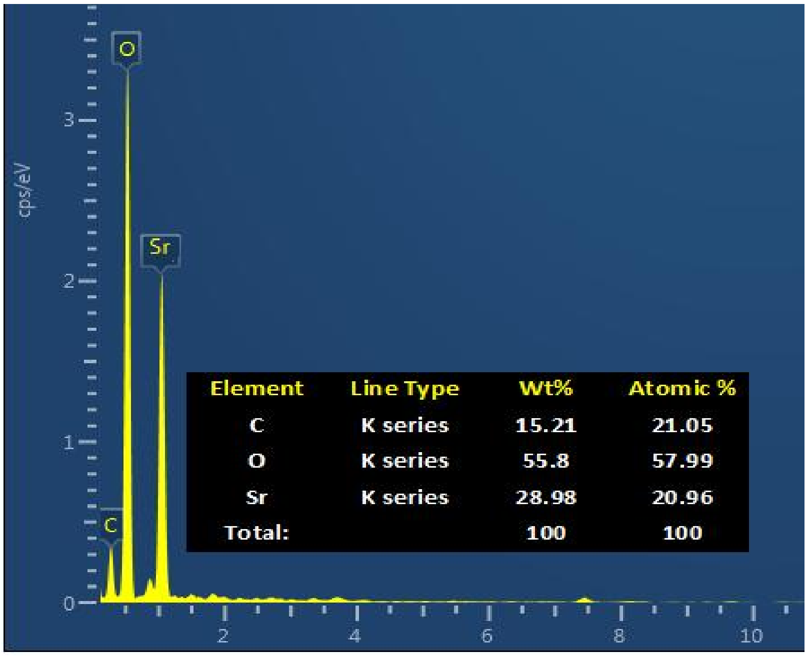


Figure 2: Scanning electron microscopy (SEM) image Sr– GO hybrid structures.

The EDX of Sr –GO composite is presented in Fig.3, and shows peaks due to Sr, C, O and C atoms. The presence of O atoms must be due to the presence of GO. Therefore, the EDX spectrum suggests that the chemical composition of the Sr/GO sample can be expressed as percentage atomic weight of elements could be confirmed by the EDX spectrum. The stoichiometry of elements was estimated and presented in the inset from the area intensity of the peaks in the EDX spectrum. Here we can point out that since each peak is substantially resolved in the EDX spectrum the area intensity is obtained with high accuracy [25].



Element	Line Type	Wt%	Atomic %
C	K series	15.21	21.05
O	K series	55.8	57.99
Sr	K series	28.98	20.96
Total:		100	100

Figure 3: Energy dispersive X-ray spectroscopy of Sr –GO composite.

3.2 Application of Sr-GO nano composite for the electrochemical degradation of the textile dye, Violet 5BN

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 [26]. 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 99.6 % in acidic *pH*-9. It indicated that the degradation of dye effluent in acidic solution is higher than that of in the basic media. Therefore the optimum *pH* 9 was maintained in subsequent experiments.

Effect of Current Density: Current density is a very important variable in electrochemical engineering. As shown in the Fig. 4, the colour and removal efficiency was increased by increasing the applied current density (0.01to 0.04 A/cm^{-2}) the results may attributed to the increased oxidant such as: chlorine/hypochlorite, hydroxyl radicals at higher current densities. Up to a current density of 0.03 A/cm^{-2}, the degradation efficiency of the dye was increased almost linearly [27]. At the same time, more energy will be consumed at higher current density applied. Therefore, the optimal current density for the successive electrochemical degradation of dye effluent was $0.03 A/cm^2$.

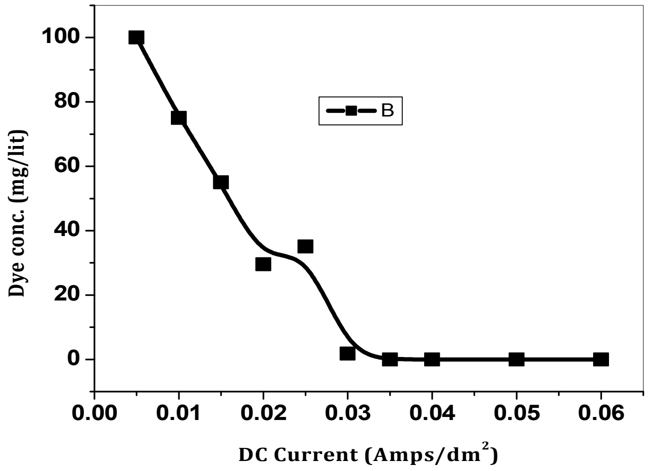


Figure 4: Effect of current density on the decoloration of Violet 5BN

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 0.25g/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:

$$E = \frac{VIt_E}{V_s} \times 10^{-3}$$

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 0.5 $kWhm^{-3}$ 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 I

The electric energy consumed during the degradation of 50 ppm (w/v) Violet 5BN 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	0.5
0.02	20	15	1.2
0.04	40	10	3.2
0.06	60	5	3.6

Effect of Sr-GO nanaocomposite:

Fig. 5 shows the effect of Sr-GO nano particle concentration on the decoloration of Violet 5BN. 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 [28]. The concentration of Sr-GO nanoparticle is increased from 0 to 0.004g/250mL.

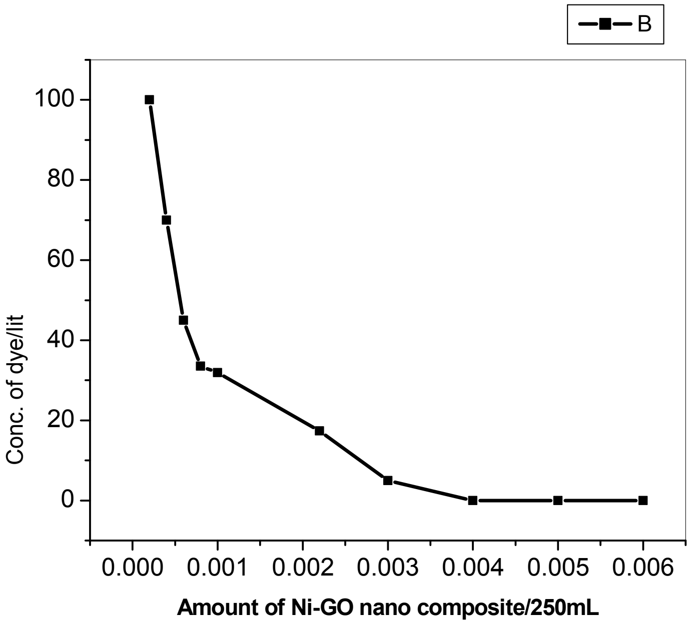


Figure 5: Effect of Sr-GO nano composite on the decoloration of Violet 5BN

IV. CONCLUSION

We successfully synthesized the metal Sr-GO nanocomposite by a facile one-pot preparation approach. The characterization of the Sr-GO composites suggested that the metal is uniformly dispersed in the graphene matrix and gives evidence indicates that the sheet-like nanostructures are GO, which are formed at the interface and we can see good compatibility between the graphite oxide and the Sr matrix. Electrochemical decoloration of an aqueous solution of Violet 5BN dye was investigated under different experimental conditions by using carbon electrodes.. 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 0.03 A cm^{-2} on electrode and NaCl supporting electrolyte at 0.25g/250mL. Our results explored the applicability of electrochemical method with the use of the nanocomposite, which proposes an alternative for dye decoloration from textile wastewater, eliminating their strong color by reducing their eco-toxicological consequences for the aquatic environment.

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