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Hydrothermal Synthesis and Characterisation of Molybdenum Oxide/ Reduced Graphene Oxide Nanocomposite for Energy Application

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Abstract: **- Despite the importance of reduced graphene oxide/ molybdenum oxide (RGO/MoO_3) nanocomposite due to its wide range of properties and corresponding applications, it is a potential candidate for electrode materials in super capacitors. Though there are a number of methods to synthesize molybdenum oxide/graphene nanocomposites, a highly desirable, alternative green method involving eco-friendly precursors and reagents is most important for the current and future scenario as for as sustainability and scalable production concerned. We here in, have reported the synthesis of (RGO/MoO_3) nanocomposite using a mild and facile hydrothermal approach. The preparation method involved the mild reagents such as ammonium molybdate tetrahydrate, hydrazine hydrate Etc. The hydrothermal reaction was carried out at about 200 °C. Highly pristine graphene oxide was obtained from mild treatment of raw pyrolytic graphite in an appropriate ratio of ethanol and water. The structure and morphology of so obtained RGO/MoO_3 were studied by using X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. The composition of the material was analyzed using energy dispersive x-ray (EDX) technique.**

Index Terms: RGO, MoO_3 / RGO composite, hydrothermal reaction, super capacitor.

INTRODUCTION

Various morphologies of RGO/ MoO_3 nanostructure with excellent performance towards hydrogen evolution reaction (HER) have been developed through various methods and which have been reviewed recently [1-2] RGO/ $MoO_3$is also a promising material in gas sensors and catalysis for selective industrial oxidation processes. It is a potential candidate of electrode materials in super capacitor application, which is rarely reported. The numbers of methods have been reported in the synthesis of molybdenum oxide/ graphene nanocomposites among which hydrothermal, solvothermal and chemical reduction are well known. However, the use of concentrated acid/alkaline solutions and strong oxidizing/reducing agents during the synthesis processes is a serious environmental concern.Hence, it is very much needed to develop alternative green methods of nanocomposite preparation, using environmentally friendly precursors and less hazardous reagents. We herein, have reported a mild and facile solvothermal approach to synthesize graphene nanocomposites /molybdenum oxide. Various forms of carbons have been used as a common electrode material, indeed carbons such as glassy carbon,graphite, carbon black powder, carbonnanotube, graphene, etc. have been studied as electrode materials due to their abundance, wide electrochemical window, low cost, etc. Also quite often the carbons are hybridized with functional materials to improve their electrochemical performance.

MATERIALS AND METHOD

Commercially available raw flakes (99% carbon purity) were purchased from S d fine chemical. Ammonium molybdate tetra hydrate, Hydrazine hydrate and carbon black were obtained from S d Fine chemicals. Ethanol was obtained from Merck. Ethylene glycol were purchased from Sigma Aldrich. Deionized water (DI) from the Milipore system was used throughout the experiment. All chemicals were used as received without further purification.

Synthesis of graphene oxide

Graphene was synthesized via liquid phase exfoliation method as reported [3-7]. In a typical experiment about 60 mg graphite flakes was dispersed in 200 ml solution of ethanol and deionized water (2:3 ratio). Sonication treatment was carried out in a conventional ultrasonic bath with frequency of 50-60 Hz at room temperature for 3 h to form a darkish black suspension. The sonicated dispersions were then centrifuged at 1000 rpm for 30 min to remove unexfoliated graphite flakes. The supernatant solution was the filtered and dried at 80 °C overnight and graphene was collected and used.

Preparation of MoO_3 -RGO composites

The MoO_3-graphene composite was prepared by a solvothermal method. First, 60 mg of prepared graphene was dispersed in 40 ml of distilled water under sonication for 15 min while 1mmol of ammonium molybdatetetrahydrate separately dissolved in 20 ml of ethanol and D.I water in the

(2:3) molar ratio under vigorous stirring. Then, the two solutions were mixed together and sonicated for 15 min and then add 2 μL of hydrazine hydrate side by side to get saturated solution. The resultant mixture was transferred to a 50 ml Teflon stainless steel autoclave. The MoO_3 and RGO/MoO_3samples were prepared by hydrothermal method at 200 °C by heating for about 24 hours in separate experiments. The solid precipitations were isolated from the solution by centrifugation, washed repeatedly with excess water and ethanol respectively and dried in an oven overnight at 70 °C. Finally, the products were thermally heated at 500 °C under inert N_2 gas with a ramping rate of 10 °C/min for 4 h and finally we get MoO_3 on RGO.

Materials characterization

The product sample was analyzed by X-ray diffraction technique using Philip- X'pert X-ray diffractometer operated at 33 mA and 45 kV with Cu $K\alpha$ radiation (λ = 1.54056 Å) in the range of 10° - 70° with step size of 0.03°. The characteristic surface elemental compositions of the composites were analyzed using a scanning electron microscope (SEM, FEI Quanta-400 FESEM). The morphology of graphene and graphene-MoO_3 composites was studied by a transmission electron microscope (TEM, JEOL JEM-2100F) at an operating voltage of 200 KV.

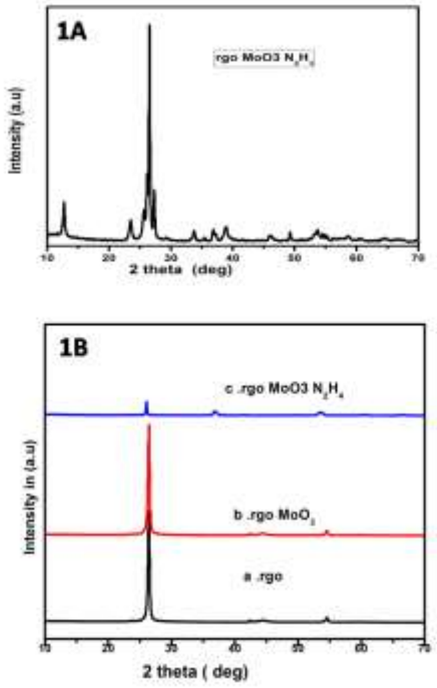


Fig.1(A) X-ray diffraction patterns of RGO/MoO_3 and in Fig 1 (B) pattern (a) for RGO, (b) for pure MoO_3 and (c) for RGO/MoO_3.

RESULT AND DISCUSSION

X-ray diffraction analysis has been performed to characterize MoO_3, RGO and RGO/MoO_3 samples. The patterns are as shown in Fig.1 and they show well-defined peaks that match the orthorhombic crystalline structure of α-MoO_3 (a = 3.962 Å, b = 13.858 Å and c = 3.697 Å, JCPDS 5-508). It can be observed that the strongest diffraction peaks for MoO_3 in the XRD pattern appear at 2θ = 26.7, The diffraction peaks for MoO_3 were appeared at 2θ = 12.72°, 23.56°, 25.64°, 27.50° and 38.89°, which correspond to the (020), (210), (170), (112) and (202) crystallographic planes respectively. The interlayer spacing (d) of MoO_3 was derived from the (201) plane using Bragg's law, which is approximately 3.49 nm.

The surface morphology of the samples was characterized using SEM technique and the micrographs are as shown in Fig 2. Fig.2 (A) and (B) are the SEM micrographs which show the surface morphology of RGO/MoO_3 sample which appear as attractive sheet like structures [8-9]. The molybdenum oxide nanoparticles would be formed by the reduction process in the presence of hydrazine which could be viewed as the dense agglomerations present in the image (B). This agglomeration is probably due to the solubility of the surfactant, hydrazine in the solvent under the given experimental conditions. Fig 2 (B) shows the MoO_3 particles of the size ranging from 204.84 nm to 492.4 nm.

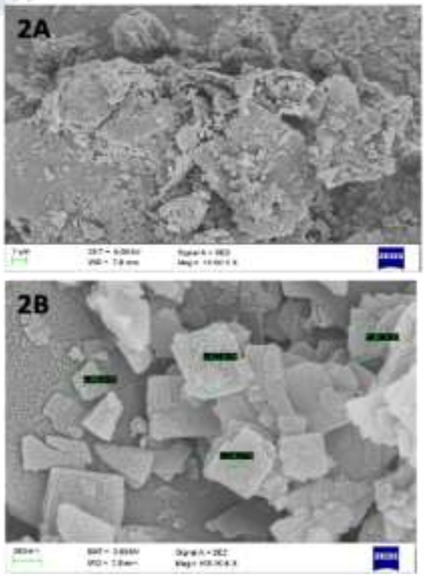
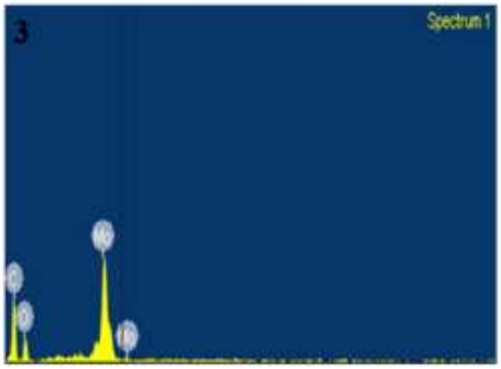


Fig.2 FE-SEM images, (A) and (B) of RGO/ MoO_3 samples at different magnifications.



Element	Weight%	Atomic%
C K	49.41	76.07
O K	14.72	17.01
Mo L	35.87	6.91
Totals	100.00	

Fig. 3 EDX pattern of the compound and Table 1 the elemental composition derived from EDX analysis.

Figure 3 shows the EDX pattern of MoO_3 deposited graphite powder. The elemental composition is as fallows. Element (weight %): carbon (76.07), oxygen(17.01) and Molybdenum(6.91%) respectively.

CONCLUSION

We have successfully prepared graphene-molybdenum oxide hybrid material using a facile one-pot hydrothermal synthesis. The method is a simple and environmentally being. This is an efficient and economical approach of preparation of RGO/ MoO_3. The structural characterization by XRD, SEM and EDX indicates the formation of RGO/ MoO_3 polycrystalline nanoparticles of molybdenum oxide on reduced graphene oxide surface. The will be tested for the energy application as there are several reports on RGO/ MoO_3 as electrode material for super and fuel cells.

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