International Journal of Applied Nanotechnology
ISSN: 2455-8524
Vol. 5: Issue 2
www.journalspub.com

Two-dimensional MoS_2 Nanosheets: Preparation and Characterization

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ABSTRACT
The amazing properties and useful applications of graphene in the field of sensors, electronics and material science opens a new challenges to other two dimensional semiconductor materials. Specially due to extraordinary electronic, mechanical and optical properties, molybdenum disulfide (MoS_2) is attracting huge interest in several applications. Single layer MoS_2 with direct band gap shows better semiconductor behavior such as photoluminescence, makes them more acceptable in optoelectronic applications in comparison with graphene which is an indirect band gap material. In this paper MoS2 nanosheets has been prepared by simple one step hydrothermal and electrochemical exfoliation process. Hexaammonium heptamolybdate tetrahydrate and thiourea were used as principle material in hydrothermal method. Here hexaammonium heptamolybdate tetrahydrate and thiourea are acting as a source of molybdenum and sulfur respectively. Temperature and time plays a crucial role in hydrothermal synthesis of MoS_2 nanosheets. Here nanosheets were prepared with 20hrs time and temperature of 200^0C. Furthermore, nanosheets were characterized by scanning electron microscope, X-ray diffraction, fourier transformed infrared spectroscopy, UV-Vis and TGA-DTA analysis. In electrochemical exfoliation process, Bulk MoS2 crystal can been used as a cathode while a lithium foil was used as an anode material. Here only surface analysis were done by field emission scanning electron microscopy (FE-SEM) method. Transmission electron microscope (TEM) has also been used for clear understanding of MoS2 nanosheets. A little literature about the electrochemical process for synthesis of MoS2 nanosheets were also presented.

Keywords *Molybdenum disulfide, nanosheets, hydrothermal, field emission scanning electron microscope, electrochemical exfoliation, TEM.*

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INTRODUCTION

The discovery of graphene [1], a carbon based two-dimensional (2D) layered material opens a new area of research in the field of nanoscience and nanotechnology. It was discovered by Andre Geim and Konstantin Novoselov from mechanically exfoliating of graphite in 2004 . Since then it is widely used in transparent electrodes [2], energy storage [3], solar cells [4], wearable devices [5], catalysis [6], double-layer capacitors [7], lubricants [8], photoemitting devices [9], hydrogen storage [10], water purification [11] and even in introducing piezoelectricity [12] in some polymer material due to its unique optical, electronic, mechanical and thermal properties. Recently researchers are focusing on other 2D materials such as graphdiyne [13], phosphorine [14,15], borocarbonitrides [16], hexagonal boron nitride [17], transition metal di-chalcogenides (TMDCs) [18], borophene [19] which would make a possible substitution of graphene in many applications . However 2D transition metal di-chalcogenides (TMDCs) have been attracting a wide range for research interest due to their favorable mechanical, chemical, electrical and optical properties [20-33]. Among them molybdenum disulphide is suitable as a replacement of graphene in several applications like photodetectors, catalysis etc for its low cost, high chemical stability and amazing electrocatalytic performances. Normally MoS_2 is a leyered structure where a plane of molybdenum atom is sandwiched between two planes of sulphide ions. These three strata form a monolayer of MoS_2. Bulk MoS_2 is conists of such several monolayers connected each other through a weak van der Walls interactions. Bulk MoS2 is a indirect semiconductor material of band gap 1.23 eV similar to silicon. It is an inorganic compound, consists of molybdenum and sulfur which is relatively unreactive, unaffected by dilute acids and oxygen. But, two dimensional MoS_2 nanosheets or monolayer MoS_2 is an direct band gap material with a band gap of 1.8 eV. The structure and appearance of two dimensional MoS_2 is similar to graphene. For preparation of MoS_2 nanosheets, several methods such as liquid exfoliation [34], wet chemical reaction [35], hydrothermal process[36], chemical vapour deposition [37] have been discovered so far. Though it is a challenging issue in developing simple, reliable and economic methods for the preparation of MoS_2 nanosheets. In this article MoS_2 nanosheets [38-60] has been prepared by simple hydrothermal and electrochemical method. Hydrothermally prepared MoS2 nanosheets were characterized by scanning electron microscope, X-ray diffraction, fourier transformed infrared spectroscopy, UV-Vis and TGA-DTA analysis.

MATERIALS AND METHOD

Hydrothermal Method

Hexaammonium heptamolybdate tetrahydrate and thiourea were used as main material in this method. In this procedure, 1.24 g of hexaammonium heptamolybdate tetrahydrate and 2.28 g of thiourea were dissolved in 50ml deionized water under vigorous stirring for 50 min to form a homogeneous solutions. Then this solutions were transferred into a teflon-lined stainless steel autoclave and closed securely. Next solutions containing autoclave heated with a temperature 200^0C for a fixed 20h time. Furthermore the final solutions were cooled down normally and centrifuged for producing powder like sample. Samples were washed with distilled water and absolute ethanol for several time. Finally this samples were dried in vacuum at 60^0C for 18 h.

Electrochemical Exfoliation Method

Bulk MoS2 and lithium foil are used as main material in that process. Bulk MoS2 and lithium foil are used cathode and anode material respectively in an electrochemical cell. Lithium intercalation can be achieved by immersing 4 g of natural MoS2 crystals (Sigma-Aldrich) in 3 mL of 1.6 M butyllithium solution in hexane (Sigma-Aldrich) for 2 days in a flask filled with argon gas. After subsequent ultrasonication and exfoliation of LixMoS2 in ethanol, the samples are dried in an oven with 60^0C temperature for 6 hrs time. Finally a black product is produced.

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www.journalspub.com

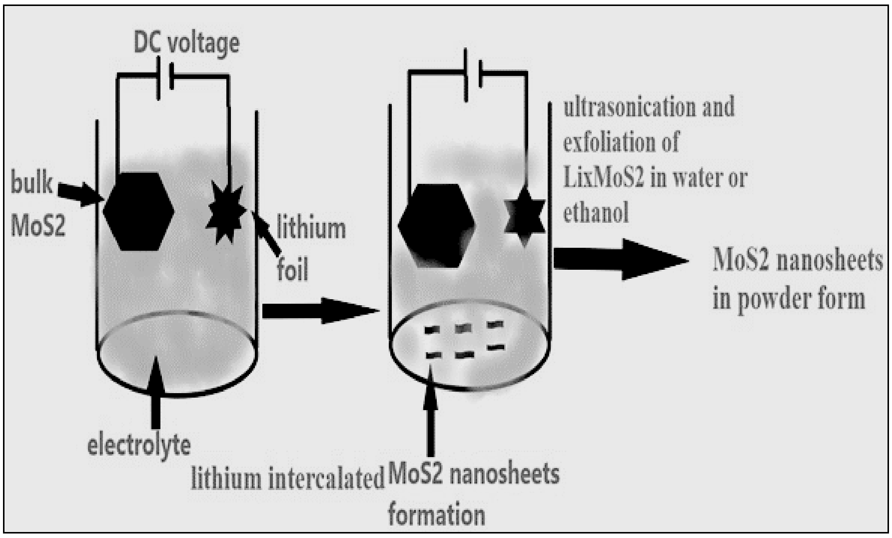


Fig. 1. *Electrochemical exfoliation method for synthesis of MoS2 nanosheets.*

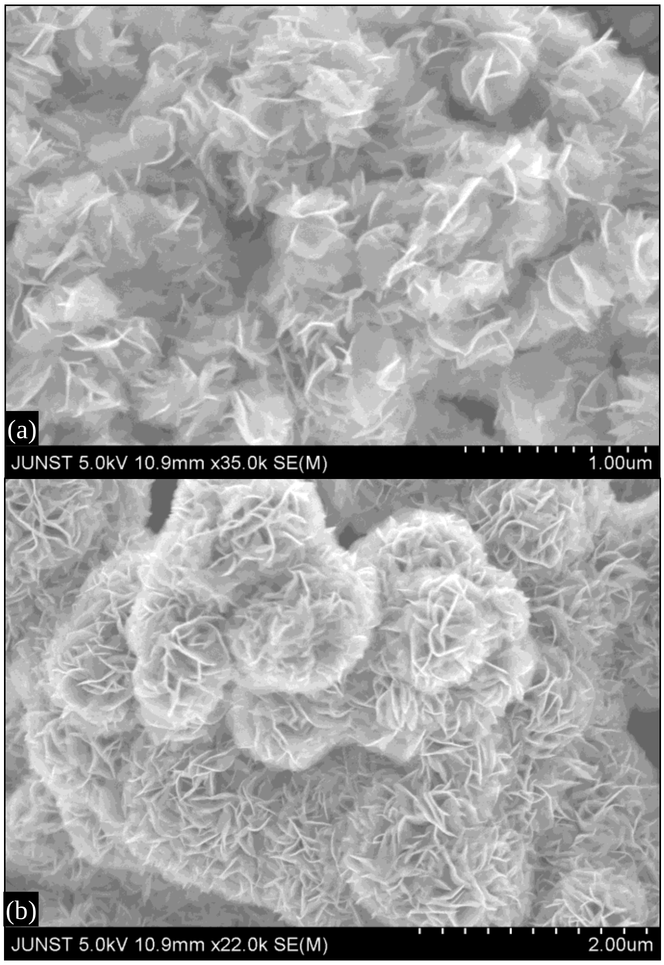


Fig. 2. *(a), (b) FE–SEM image of MoS2 nanosheets.*

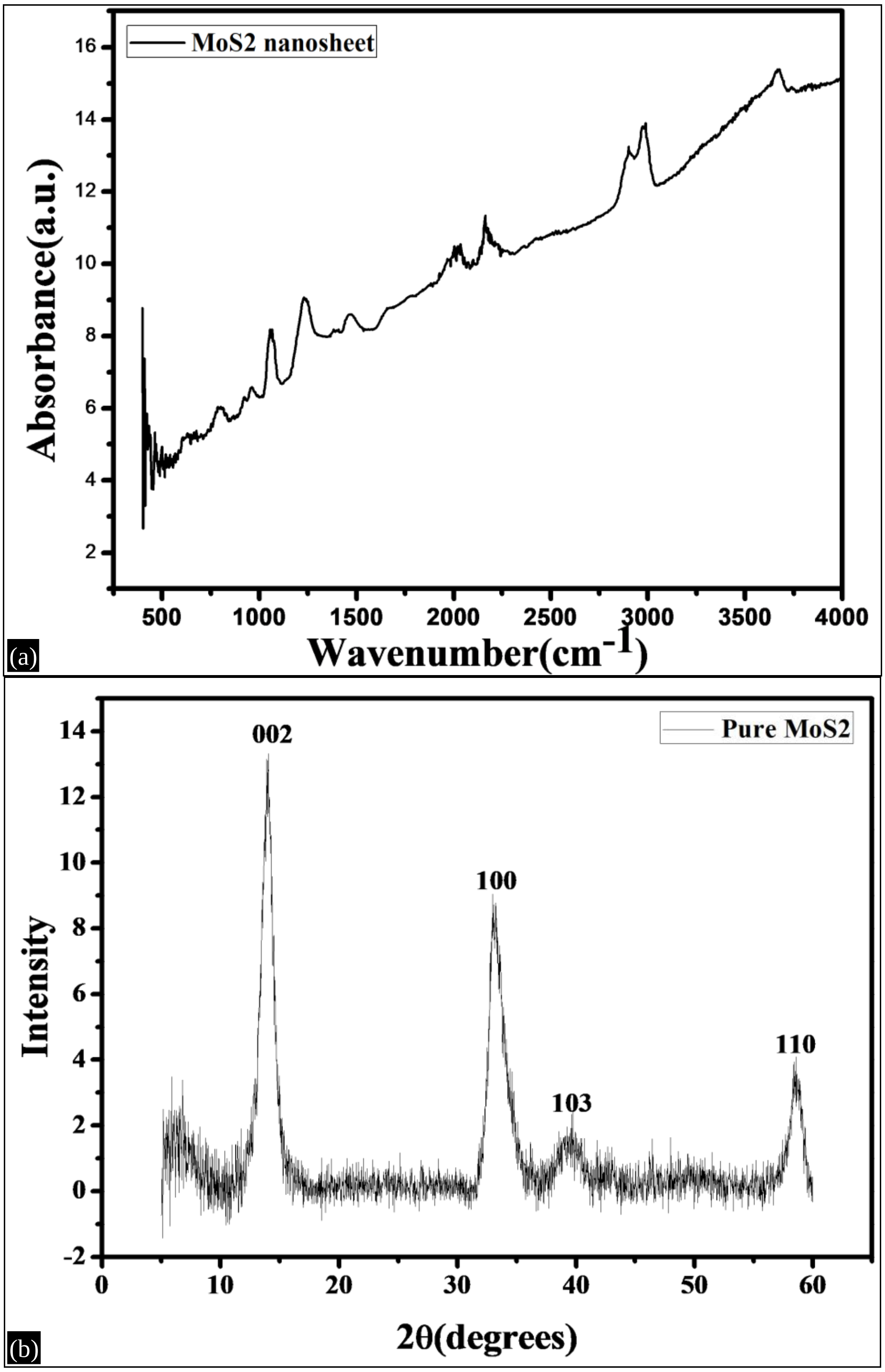
CHARACTERIZATIONS

Hydrothermally prepared MoS2 nanosheets has been characterized by field emission scanning electron microscope, X-ray diffraction (XRD, Bruker), fourier transformed infrared spectroscopy, UV-Vis and TGA-DTA analysis.

RESULTS AND DISCUSSIONS

Hydrothermal Method

Figure 2 and 3 (b) shows the FE-SEM image and XRD pattern of MoS2 nanosheets. From FE-SEM image, it is clearly observed the formation of MoS2 nanosheets. To reveal the formation of nanosheets, higher magnification image has also been done. From x-ray diffraction pattern, it is observed the high crystallinity of as produced MoS2 nanosheets.



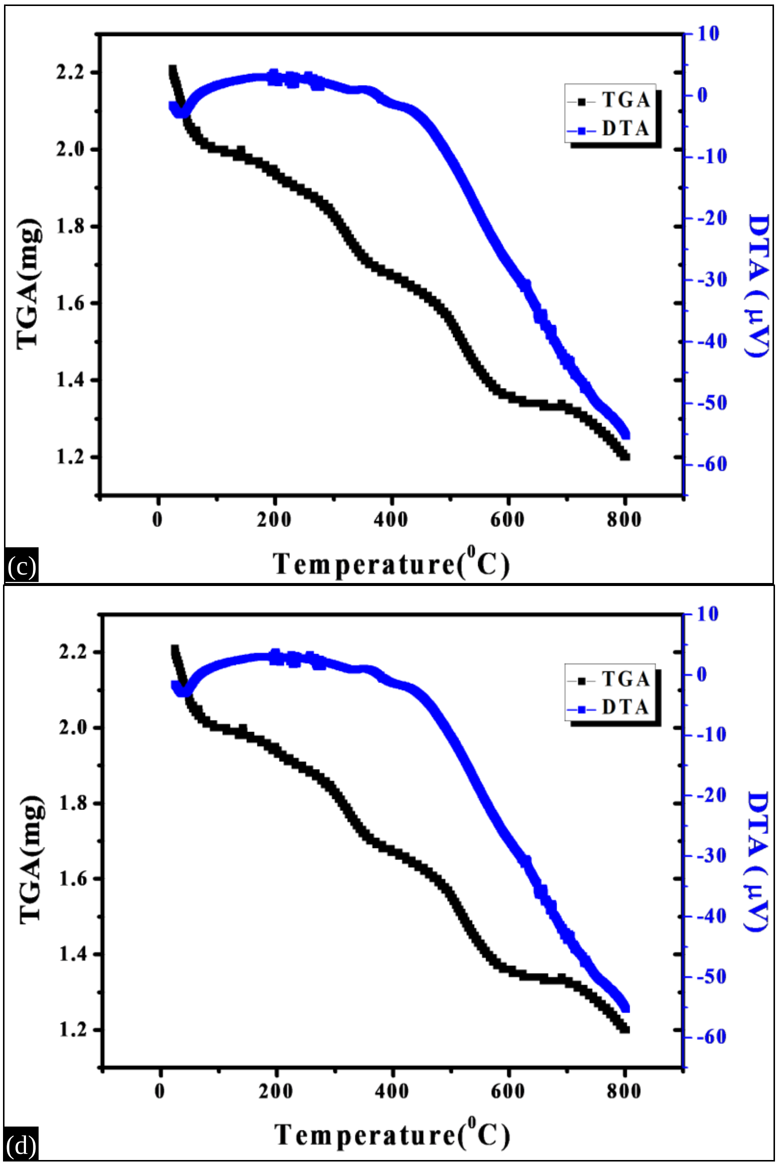


Fig. 4. *(a) FT-IR, (b) XRD, (c) TGA-DTA and (d) UV-Vis data of MoS2 nanosheets.*

Figure 4 shows the thermogravimetric – differential thermal analysis curve of MoS2 nanosheets prepared through hydrothermal method. Initial sample weight at room temperature was around 2.2 mg. Temperature increases upto 800^0C and the corresponding weight becomes 1.2 mg at 800^0C. An endothermic peak was observed at around 42^0C. Figure 3 represents the fourier transformed infrared spectroscopy and X-ray diffraction pattern of pure MoS2 nanosheets. XRD peak signifies the highly crystallinity of MoS2 nanosheets.

CONCLUSIONS

In this paper a simple one step hydrothermal process has been explored for producing MoS2 nanosheets. In hydrothermal method, the quality of nanosheets are dependent on the process temperature. Here nanosheets has been prepared at 200^0C with 20hrs time. The TEM image shows the beautiful formation of MoS2 nanosheets. For beautiful properties of MoS2 nanosheets, it is extensively used in several nanotechnology applications. MoS2 nanosheets is used to enhance the sensitivity of polymer based piezoelectric nanogenerator. It is also used as a nanomaterial in the formation of nanocomposite with several material which has not good physical, electronic and optical properties.

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Cite this Article: Binoy Bera, Diptonil Banerjee. Two-dimensional MoS2 Nanosheets: Preparation and Characterization. *International Journal of Applied Nanotechnology*. 2019; 5(2): 8–16p.

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