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Studies on MoS_2 Nanoflower by Hydrothermal Route

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Abstract

Synthesis and characterization of MoS_2 nanoflower by using conventional hydrothermal route. The reaction is carried out at 160°C for 12 hours by taking 1:2 proportion of citric acid and ammonium molybdate by adding 2.36 gm of thioacetamide. The obtained powder is characterized by using X-ray Diffractogram and Scanning Electron Microscopy. X-ray Diffractogram reveals that MoS_2 shows hexagonal crystal structure. Field Emission Scanning Electron Microscopy shows nanoflower type structure. Chemical analysis using Energy Dispersive Spectra (EDS) indicates the presence of Mo & S.

Keywords: Hydrothermal *route, Nanoflower, MoS_2.*

Introduction

Now days the two dimensional (2-D) and three dimensional (3-D) nanostructured materials have attracted great attention because of their potential applications in functional materials and nano devise [1]. The transition metal sulfides, MoS_2 have been studied in research for applications such as lithium batteries, wear resistance and catalytic hybrid sulfurization of petrol [1-2]. Recent research has suggested that MoS_2 crystal can used in lubrication properties [1]. MoS_2 is a transition metal sulfide with a layered structure where Mo layer in between two sulfur layers which are connected by weak van der Waals forces [3]. Various synthesis methods such as Sol gel, Co-precipitation, Spray Pyrolysis and Chemical vapour deposition [1, 4-7]. Hydrothermal synthesis is widely used because of a simple, low temperature, low cost and high purity [1, 3]. In this work, we report a simple hydrothermal synthesis and characterization of MoS_2 nanoflowers.

Methodology

Material:

Ammonium molybdate tetrahydrate $(NH_4)_6 Mo_7O_{24}.4H_2O$, thioacetamide ($CH_3CS.NH_2$) and citric acid monohydrate ($C_6H_8O_7$) H_2O. All starting materials were high purity AR grade.

Synthesis of MoS_2

MoS_2 nano flower were synthesized by using Ammonium Molybdate tetrahydrate, citric acid and Thioacetamide as a starting material. Citric acid was added to deionized water and Ammonium molybdate tetrahydrate with molar ratio 1:2, stirred at 120°C for 20 min on magnetic stirrer. Then thioacetamide dissolved in deionized water and slowly added to solution mixture. At the end solution brownish black precipitate was transferred in teflon lined stainless steel autoclave and autoclave kept in preheated muffle furnace 160°C for 12 hour. Autoclave cooled at room temperature and obtained product was washed 3 times with ethanol and DI water then filtered. The obtained powder was dry under the IR lamp for 8 hrs.

Material Characterization:

Powder X-ray diffractogram (XRD) was recorded on (Bruker AXS Model: D8 Advances, Germany) using cu kα radiations. The micro structural characterization with EDS was studied by using field emission Scanning electron microscopy JEOL. The FTIR spectrum was collected using FT/IR-4700 type A.

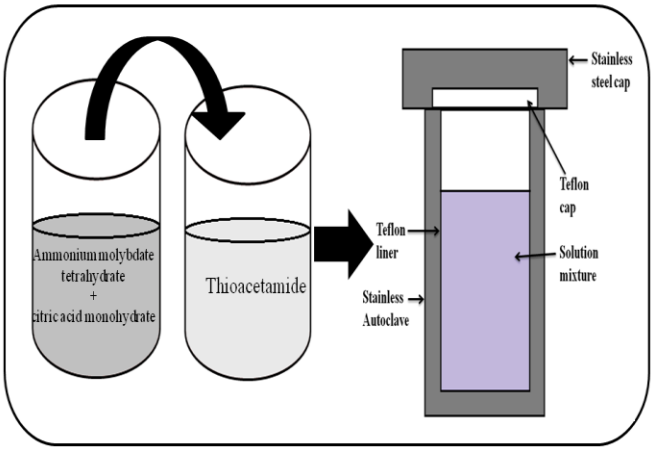

Fig. 1 Schematic of MoS_2 Synthesized by Hydrothermal route

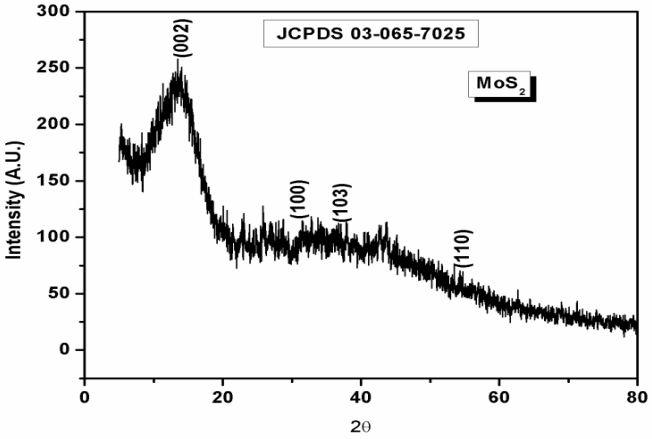

Fig 2: X-ray Diffractogram of MoS_2 hydrothermal synthesized powder

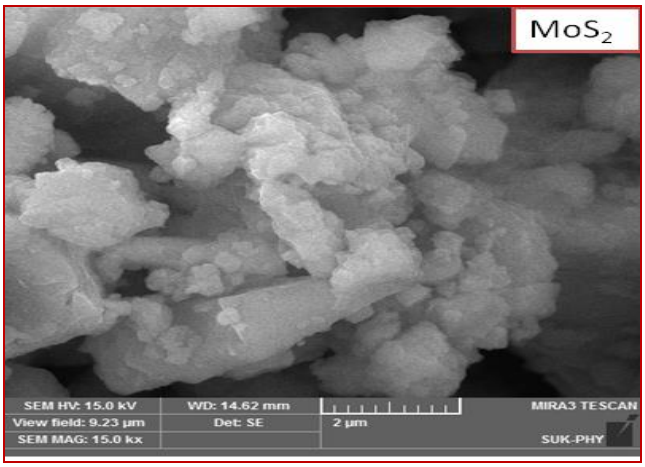

Fig.3 a) Field Emission Scanning Electron Microscopy of MoS_2

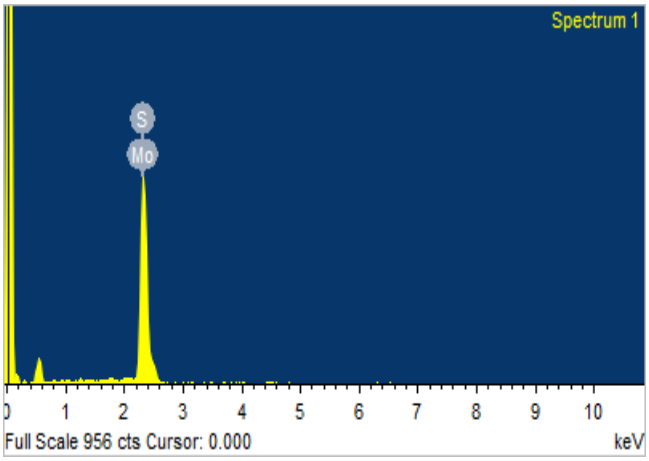

Fig.3 b) Energy Dispersive Spectra of MoS_2

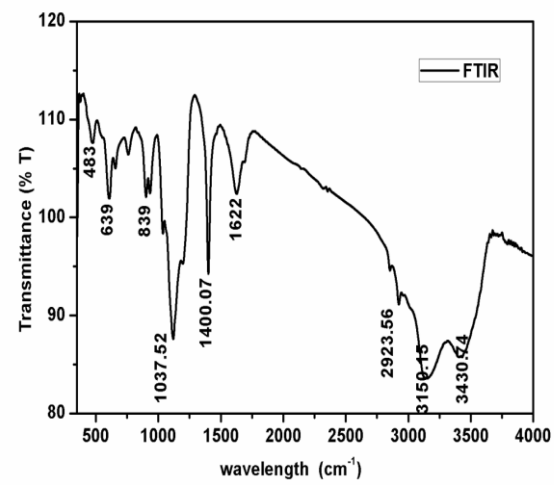

Fig. 4 FTIR of MoS_2

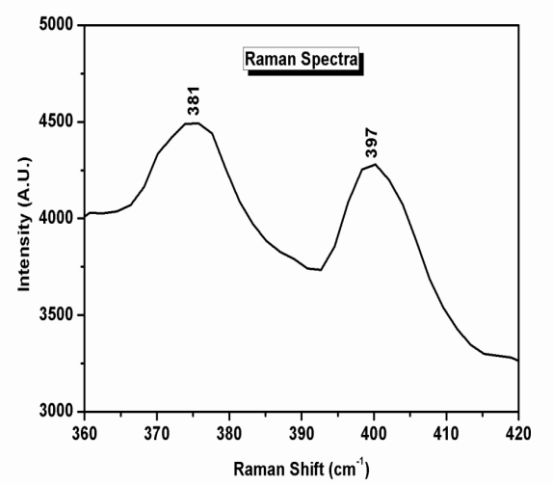

Fig. 5 Raman Spectra of MoS_2

Result and Discussion

The synthesized sample was characterized by using x-ray diffractogram (XRD) to study the crystal structure. From the X-ray diffractogram sample shows hexagonal (P63/mmc space group) crystal structure with a=3.1600 A^0, c= 12.2900 A^0. No any impurity peaks were found.

Fig.3 a) shows Field Emission Scanning Electron Microscopy (FESEM) image of synthesized powder of MoS_2. FESEM image show that the MoS_2 nanoflower type structure. The image is nanoflowers agglomeration type structure. Chemical analysis using Energy Dispersive Spectra (EDS) indicates the presence of Mo & S and no any other element excited. Fig. 3 b) shows the EDS pattern of MoS_2 sample.

Fig. 4 FTIR spectrum of hydrothermally synthesized MoS_2 shown in fig.4 There are broad absorption bands at 483 cm^{-1}, 639 cm^{-1},839 cm^{-1}, 1037.52 cm^{-1}, 1400.07 cm^{-1}, 1622 cm^{-1} for prepared sample. The band at 483 cm^{-1} ascribed to Mo-S bond and at 903 cm^{-1} is due to S-S bond, 1400.07 cm^{-1}, 1622 cm^{-1} is due to MoS_2, and broad band between 2600 cm^{-1} to 3500 cm^{-1} is attributed to the O-H stretching from intermolecular and intramolecular hydrogen bonds [8].

The Raman spectrum of MoS_2 shown in figure 5. There are two prominent peaks at 381 cm^{-1} and 397 cm^{-1} [9].

Conclusion

MoS_2 nanoflowers have been synthesized successfully synthesis of simple hydrothermal route using ammonium molybdate & thioacetamide. X-ray Diffractogram reveals that MoS_2 shows hexagonal crystal structure. Mo and S elements confirmed by EDX spectrum. Raman spectrum MoS_2 shows two peaks at 381 cm^{-1} and 397 cm^{-1}

Conflicts of interest: The authors stated that no conflicts of interest.

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