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Fabrication of Hydrothermally Synthesized Molybdenum Disulfide Nanosheets Based Membrane

Adarsh Kumar Pandey[1], Sajal Rai[2], Ashish Borah[2], Ahsan Ali[2], Amit Kumar Patel[2], Vikram Singh[1], Anchal Srivastava[2]

[1]Department of Physics, Nehru Gram Bharti (Deemed to be University), Prayagraj, Uttar Pradesh

[2]Department of Physics, Banaras Hindu University, Varanasi, Uttar Pradesh

Abstract

The fabrication of hydrothermally synthesized molybdenum disulfide (MoS_2) nanosheet-based membranes represents a promising advancement in membrane technology for applications in water purification, gas separation, and energy storage. This study focuses on the hydrothermal synthesis of MoS_2 nanosheets, their characterization, and the subsequent fabrication of high-performance membranes. MoS_2 nanosheets were synthesized using a simple hydrothermal method, optimizing parameters such as temperature, reaction time, and precursor concentration to achieve uniform and defect-free nanosheets. The synthesized nanosheets were characterized using Raman spectroscopy, X-ray diffraction (XRD), transmission electron microscopy (TEM) and Fourier transform infrared spectroscopy (FTIR) to confirm their structural integrity, morphology, and chemical composition. The MoS_2 nanosheets were then incorporated on a Whatman filter paper using a simple vacuum-assisted filtration setup to fabricate membrane. This study highlights the potential of hydrothermally synthesized MoS_2 nanosheets in the fabrication of a simple & easy filtration membrane for sustainable and efficient separation processes.

Keywords: MoS_2 Nanosheets, Hydrothermal Synthesis, XRD, FT-IR, TEM, Membrane

1. Introduction

The development of filtration membrane has become an important area of research due to their applications in water purification, gas separation, and energy storage. Traditional polymeric membranes, while effective, often face limitations such as low permeability, poor selectivity, and susceptibility to fouling. To address these challenges, researchers have turned to nanomaterials, particularly two-dimensional (2D) materials, to enhance membrane performance. Among these, molybdenum disulfide (MoS_2) nanosheets have emerged as a promising candidate due to their unique structural, mechanical, and chemical properties [1]. MoS_2, a transition metal dichalcogenide, consists of layers of molybdenum atoms sandwiched between sulfur atoms, forming a hexagonal lattice structure. This layered structure allows for the exfoliation of MoS_2 into ultrathin nanosheets, which exhibit high surface area, excellent mechanical strength, and tunable electronic properties. These characteristics make MoS_2 nanosheets highly suitable for incorporation into filtration membrane, where they can improve permeability, selectivity, and fouling resistance [2]. Hydrothermal synthesis is a widely used method for producing MoS_2 nanosheets due to its simplicity, cost-effectiveness, and ability to control the size, morphology, and layer thickness of the nanosheets. This method involves the reaction of molybdenum and sulfur precursors in a sealed autoclave at elevated temperatures and pressures, resulting in the formation of high-quality MoS_2 nanosheets [3]. The fabrication of MoS_2 nanosheet-based filtration membrane typically involves the incorporation of the nanosheets into a Whatman filter paper using a simple vacuum-assisted filtration setup to fabricate membrane [4]. These membranes leverage the unique properties of MoS_2, such as its high surface area, mechanical strength, and chemical stability, to achieve superior performance in various separation processes. For instance, the nanochannels formed between MoS_2 nanosheets can facilitate the rapid transport of water molecules while rejecting ions and larger molecules, making them ideal for desalination and water purification applications [5]. This study focuses on the hydrothermal synthesis of MoS_2 nanosheets, their characterization, and the fabrication of filtration membrane. By optimizing the synthesis parameters and membrane fabrication process, we aim to develop membrane with enhanced permeability, selectivity, and durability. The successful integration of MoS_2 nanosheets into filtration membrane holds good for addressing the growing demand for efficient and sustainable separation processes in various industries [6].

2. Experimental Section

2.1 Reagents & Chemicals

Ammonium molybdate tetrahydrate ($(NH_4)_6Mo_7O_{24}4H_2O$, 99%) and thiourea (purified, $NH_2.CS.NH_2$) both chemicals were purchased from Molychem, located in Mumbai, Maharashtra 400002, India. Every chemical compound was of analytical quality and used exactly as is. Deionized (DI) water was utilized for the duration of the experiment.

2.2 Synthesis of MoS_2 Nanosheets

A schematic representation of the MoS_2 nanosheet production process is shown in Figure 1. As explained in [7], a hydrothermal process was used to create the nanosheets. In particular, 1.83 g of thiourea (CH_4N_2S) and 1.06 g of ammonium heptamolybdate ($(NH_4)_6Mo_7O_{24}\cdot 4H_2O$) were thoroughly dissolved in 60 mL of deionized water at ambient temperature while being constantly stirred. After that, the resultant solution was put in a 100 mL autoclave lined with Teflon and heated to 180°C for 16 hours. Following the reaction, the autoclave was allowed to cool to ambient temperature before the black substance that resulted was repeatedly cleaned with distilled water. The items were dried for 12 hours at 80°C.

Figure 1: Schematic diagram of the synthesis of MoS_2 nanosheets

2.3 Preparation of MoS_2 Nanosheets based Membrane

We dispersed 0.05 g of as synthesized MoS_2 nanosheets using a sonicator in 30 ml of DI water. Subsequently, a layer-stacked MoS_2 filtration membrane was assembled on a Whatman filter paper using a simple vacuum assisted filtration setup. The homogenous solution formed a membrane with a thickness of about 50μm Figure 2. The black film deposited onto the Whatman filter paper was then dried at 50 °C for 30 min fig shows a schematic of the whole method [8].

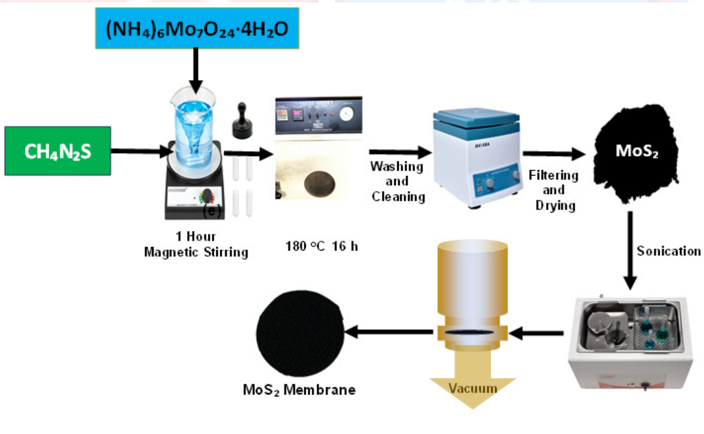


Figure 2: Schematic for preparation of MoS_2 nanosheets based filtration membrane

2.4 Optical Images of MoS_2 nanosheets based filtration membrane

MoS_2 nanosheets based filtration membrane images are shown in Figure 3. ((a), (b), (c), (d), (e) and (f))

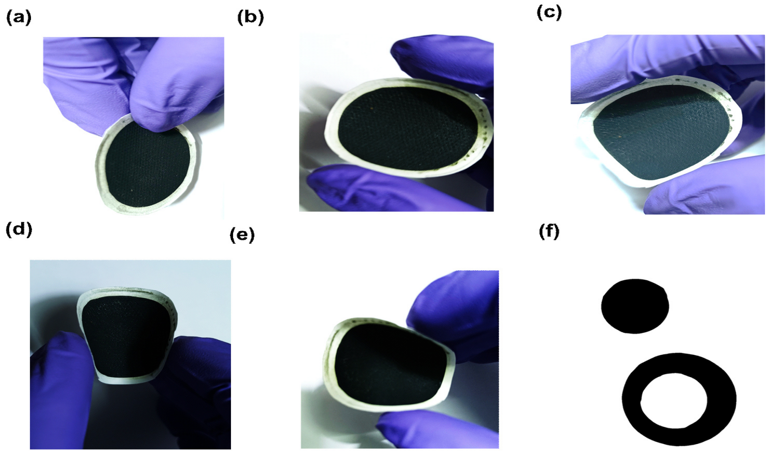
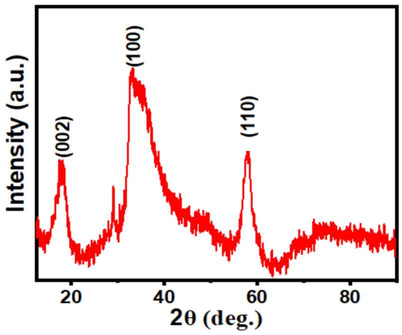
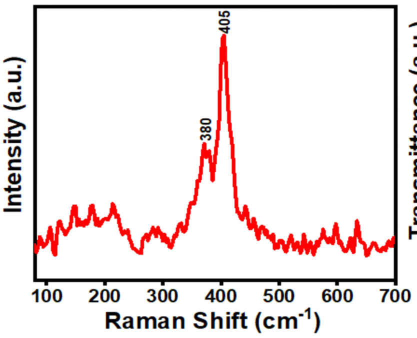


Figure 3: ((a), (b), (c), (d), (e) and (f)) Optical Images of MoS_2 nanosheets based filtration membrane.

3. Results and Discussion

We used Fourier-transform infrared (FT-IR), Raman, and X-ray diffraction (XRD) spectroscopy to examine the structural, vibrational, and chemical characteristics of MoS_2 nanosheets. The crystal structure of the MoS_2 nanosheets under various processing conditions was investigated using powder XRD analysis, as illustrated in Figure 4. The MoS_2 structure's (002), (100), and (110) crystal planes are represented by the XRD pattern, which has peaks indexed at 14°, 33°, and 58°. This is consistent with the standard reference (JCPDS card no. 37-1492) and validates the crystalline nature of the MoS_2 nanosheets. Interestingly, no secondary phases or impurity peaks were seen [9,10]. The peak broadens and the intensity of the (002) peak decreases, indicating effective exfoliation into thinner layers, indicating the presence of smaller crystallites in few-layer or monolayer MoS_2 [11,12]. As shown in Figure 5, Raman spectroscopy further confirms the nanosheet properties by identifying two unique vibrational modes: the out-of-plane Ag mode at about 405 cm^{-1} and the in-plane E_{12}g mode at approximately 380 cm^{-1}. The frequency difference (Ä) between these peaks is a crucial indicator of layer thickness. Because of stacking effects and interlayer interactions, Ä is typically between 18 and 20 cm^{-1} for monolayer MoS_2 and roughly 25 cm^{-1} for bulk MoS_2 [13,14]. Moreover, strain, flaws, and doping effects on MoS_2 nanosheets were studied using Raman analysis. FT-IR spectroscopy, as illustrated in Figure 6, further confirmed these results by revealing large absorption bands at roughly 480 cm^{-1}, 900 cm^{-1}, 1100 cm^{-1}, and 1650 cm^{-1}. Whereas the 480 cm^{-1} band is linked to MoS_2 vibrations, the 900 cm^{-1} band is related with S–S stretching [15,16]. A partial oxidation or interaction with adsorbed water molecules is indicated by the hydroxyl and Mo–O vibrations linked to the absorption band between 1100 cm^{-1} and 1650 cm^{-1} [17,18]. The notable lack of the distinctive hydroxyl stretching band at around 3500 cm^{-1} in the nanosheet sample indicates that either surface-bound water was removed during synthesis or processing, or there was less hydroxyl functionalization [19,20]. We can confirm the surface chemistry, layer thickness, phase purity, and crystallinity of MoS_2 nanosheets by combining various characterisation methods to provide a thorough understanding of their structure and chemistry.





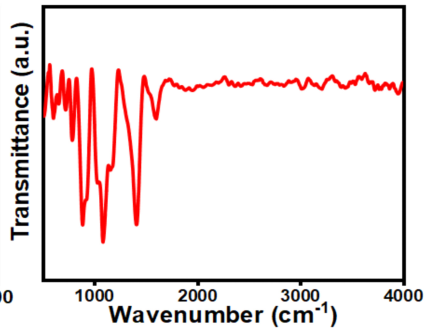


Figure 4: XRD patterns of MoS_2, Figure 5: Raman spectra of MoS_2, Figure 6: FT-IR spectra of MoS_2

The transmission electron microscopy (TEM) analysis of MoS_2 nanosheets synthesized using ammonium heptamolybdate and thiourea disclose about the multilayered structure with a uniform particle size distribution and well-defined morphology [21,22]. TEM images taken at various magnifications (Figures 7(a), 7(b), and 7(c)) shows the structural integrity of the nanosheets, highlighting their controlled size and shape [23,24]. Additionally, high-resolution TEM images (Figures 7(d), 7(e), and 7(f)) indicate that the nanosheets have a thickness ranging from 10 to 50 nm, with edge lengths adjustable within the nanometer scale, typically from 40 nm to several tens of nanometers [25,26]. These results align with the previous studies that retain chemical precursors and optimized reaction conditions to get the MoS_2 nanosheets with controlled synthesis and uniform morphology [27,28]. The precisely tunable dimensions of these nanosheets make them promising candidates for applications in energy storage, optoelectronic devices, and catalysis [29,30].

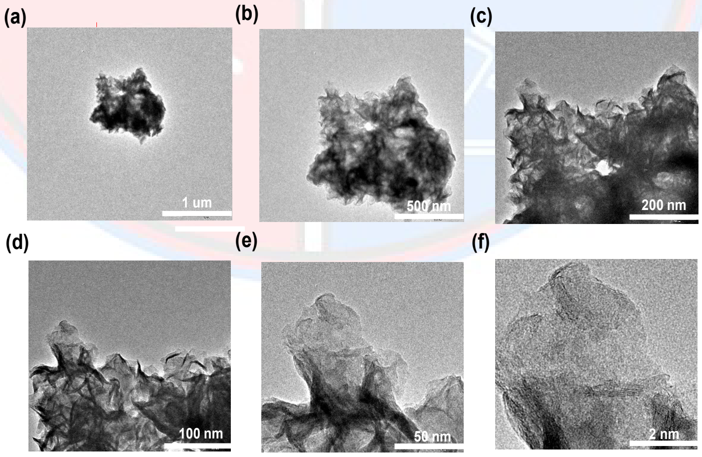


Figure 7: TEM images of MoS_2 nanosheets.: ((a), (b), (c), (d), (e), and (f))

4. Conclusion

In this work, we successfully synthesized MoS_2 nanosheets utilizing the hydrothermal technique. Together, the characterization methods of transmission electron microscopy (TEM), Raman spectroscopy, powder X-ray diffraction (XRD), and FT-IR verify the crystalline structure, layered morphology, and vibrational characteristics of the produced MoS_2 nanosheets. For the production of MoS_2 nanosheets for water contamination filtration applications, the hydrothermal process is the best option because of its ease of use and scalability. Using a straightforward vacuum-assisted filtration system, a basic MoS_2 filtration membrane was created using Whatman filter paper. For the filtration of water contamination, a prepared membrane can be created. For specific applications in water contamination filtering, further improvement of the synthesis and membrane fabrication conditions can improve the membranes' structural and functional characteristics.

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